Quality Assurance Project Plan
Freeway Landfill, Dump and Transfer Station Investigation

Freeway Landfill, Dump and Transfer Station

Burnsville, Minnesota

Revision 0.1

December 2018

Prepared for Minnesota Pollution Control Agency

By Barr Engineering Co.

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By Barr Engineering Co.

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Acronym List

%R – Percent recovery

CFR – Code of Federal Regulations

COC – Chain of Custody

COI – Contaminant of interest

DQO – Data quality objective

EDD – Electronic data deliverable

GC/MS – Gas chromatograph / mass spectrometer

LCS – Laboratory control sample

MDH – Minnesota Department of Health

MDL – Method detection limit

MPCA – Minnesota Pollution Control Agency

MS – Matrix spike

MSD – Matrix spike duplicate

NELAP – National Environmental Laboratory Accreditation Program

OSHA – Occupational Safety and Health Administration

PHASP – Project Health and Safety Plan

PID – Photoionization detector

PT – Proficiency testing

QA – Quality assurance

QAM – Quality Assurance Manual

QAPP – Quality Assurance Project Plan

QC – Quality control

RCRA – Resource Conservation and Recovery Act

RPD – Relative percent difference

SLV – Soil Leaching Value

SOP – Standard operating procedure

SRV – Soil Reference Value

USEPA – United States Environmental Protection Agency

A3 Distribution List

Pat Hanson, Minnesota Pollution Control Agency, Project Manager

William Scruton, Minnesota Pollution Control Agency Quality Assurance Coordinator

Dan Fetter, Barr Engineering Co. Principal in Charge

Sheryl Filby Williams, Barr Engineering Co. Project Manager

Michael Dupay, Barr Engineering Co. Quality Assurance Manager

Erin Evans, Pace Analytical Services Laboratory Senior Quality Manager

Shane Olund, Minnesota Department of Health Quality Assurance Manager

Brad Jacobson, Pace Analytical Services Project Manager

Paul Moyer, Minnesota Department of Health, Environmental Laboratory Manager

A4 Project and Task Organization

The project organization is shown on Figure 3. Qualifications of the main project team members are included in Appendix A.

A4.1 MPCA Project Manager

Mr. Pat Hanson is the MPCA project manager. He is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The MPCA project manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The MPCA project manager will provide the major point of contact and control for matters concerning the project. The responsibilities of the MPCA project manager include:

- Acquiring and applying resources as needed to ensure performance within budget and schedule constraints
- Directing and approving project activities
- Reviewing project deliverables and overseeing project strategies
- Representing the project team at meetings and public hearings

The MPCA project manager may delegate some of these responsibilities to competent individuals representing MPCA.

A4.2 Barr Engineering Co.

At the direction of MPCA, Barr has responsibility for project oversight, management, and implementation of site monitoring activities required for compliance with the Site Investigation. The various quality assurance and management responsibilities of key project personnel are defined below.

A4.2.1 Barr Principal in Charge

Dan Fetter is the Barr principal in charge. His resume is included in Appendix A. The principal in charge has overall responsibility for verifying that the project meets the established objectives and quality standards.

Specific responsibilities of the principal in charge include:

- Leading and overseeing, management, contracting, administration, and technical quality of the project
- Providing independent quality review and validation for technical issues
- Monitoring client satisfaction for contract work
- Verifying project meets the established objectives and resolving quality issues

A4.2.2 Barr Project Manager

Sheryl Filby Williams is the Barr project manager. Her resume is included in Appendix A. Barr's project manager is MPCA's primary contact for technical issues and day-to-day communication of scope, schedule, and budget progress and has the day-to-day and overall responsibility for managing implementation of the project. The Barr project manager's primary function is to see that technical, financial, and scheduling objectives are achieved successfully. Specific responsibilities of the Barr project manager include:

- Preparing project reports
- Managing day-to-day administration, budgeting, coordination, scheduling, and other managerial tasks
- Matching project needs with staff abilities and informing the team members of the project requirements and objectives
- Directing technical aspects of the project, including defining project objectives, developing a detailed work plan and schedule, reviewing reports, and directing field and other technical staff
- Ensuring project quality, including technical correctness and completeness, contract compliance, and budget and schedule compliance
- Communicating directly with the MPCA project manager on technical recommendations, project updates, and scope, schedule, or budget modifications

The Barr project manager may delegate some of these responsibilities to competent individuals.

A4.2.3 Barr QA Manager

Michael Dupay is the Barr QA manager. His resume is included in Appendix A. The role of the QA manager is to provide an independent review of the data and the process to see that the work meets quality standards. He is responsible for reviewing the implementation of the QA program in conformance with the requirements of this quality assurance plan, and the demands of specific project tasks. Specific responsibilities of the QA manager include:

- Providing QA technical assistance to the project team
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the Barr project manager
- Evaluating laboratory data
- Conducting or coordinating field audits
- Initiating, tracking, and reviewing QA/QC corrective actions
- Maintaining and distributing the approved QAPP and subsequent revisions (if applicable)

A4.3 Pace Analytical Services, Inc.

Pace Analytical Services, located in Minneapolis, MN (Pace), will conduct the physical and chemical analyses of the analytical samples, with the exception of the PFAS and VOC analyses in water which will be analyzed by the Minnesota Department of Health Environmental Services Laboratory located in Saint Paul, Minnesota (MDH). The analytical work required for this project is listed in Table 1, which includes Pace's address. Copies of their certificates and scopes of accreditation are provided in Appendix C. Specific roles of laboratory personnel are outlined below.

A4.3.1 Pace Laboratory Project Manager

Brad Jacobson is the Pace project manager. His resume is included in Appendix A. The Pace project manager is responsible for verifying that the assessment data meets the established objectives and quality standards. The Pace project manager is responsible for technical quality control and project oversight. The Pace project manager's primary function is to see that technical, financial, and scheduling objectives are achieved successfully. The Pace project manager will be the primary laboratory contact for administrative, financial, and scheduling considerations. Specific responsibilities include:

- Developing and meeting ongoing project requirements
- Reviewing work performed by Pace to verify its quality, responsiveness, and timeliness

A4.3.2 Pace Laboratory QA Manager

Erin Evans is the Pace QA manager. Her resume is included in Appendix A. The laboratory QA manager will remain separate and distinct from analytical testing-related duties. The QA manager is responsible for maintaining conformance to project QA requirements, the laboratory's Quality Assurance Manual, USEPA, and related methodologies. The following lists several specific duties of the laboratory QA manager:

- Tracking validation data and ensuring adherence to published guidelines
- Determining if the levels of QA/QC are being achieved
- Maintaining QA/QC procedures
- Initiating and overseeing internal audits
- Initiating and implementing corrective actions

A4.3.3 Pace Field Staff

The role of Pace field staff is to collect analytical samples following the procedures outlined in this QAPP and associated work plans. Additional field staff responsibilities include:

- Obtaining and calibrating the necessary field equipment prior to beginning an assessment
- Overseeing investigation contractors to ensure proper techniques are being followed and the desired information is being collected
- Collecting field measurements and samples during sampling events, and submitting samples to the laboratory for analysis
- Meeting quality objectives during sample collection, packaging, documentation, and shipping
- Documenting field activities to assist subsequent data analysis interpretation and reporting

A4.4 Minnesota Department of Health Environmental Services Laboratory

The Minnesota Department of Health Environmental Services Laboratory, located in Saint Paul, MN (MDH), will conduct the PFAS and VOC analyses for water samples. The analytical work required for

this project is listed in Table 1, which includes MDH's address. Copies of their certificates and scopes of accreditation are provided in Appendix C. Specific roles of laboratory personnel are outlined below.

A4.4.1 MDH Laboratory Project Manager

Paul Moyer is the MDH project manager. His resume is included in Appendix A. The MDH project manager's responsibilities are the same as those identified in A4.3.1 for Pace with respect to analyses of PFAS and VOCs in water.

A4.4.2 MDH Laboratory QA Manager

Shane Olund is the MDH QA manager. His resume is included in Appendix A. The laboratory QA manager will remain separate and distinct from analytical testing-related duties. The MDH laboratory QA manager's responsibilities are the same as those identified in A4.3.2 for Pace with respect to analyses of PFAS and VOCs in water.

A4.5 Minnesota Pollution Control Agency (MPCA)

The MPCA is the lead regulatory agency for the project. The MPCA project manager and quality assurance coordinator must approve quality documents prior to beginning any field work. Specific responsibilities for the MPCA project manager and the MPCA quality assurance coordinator are addressed in the following sections.

A4.5.1 MPCA Project Manager

Pat Hanson is the MPCA project manager. Specific responsibilities include;

- Directing review and approval of the QAPP and work plans
- Consulting with the Barr project manager
- Consulting regarding decision making process to evaluate alternative corrective measures and/or final decisions that have an effect on project outcomes and data quality
- Reviewing progress reports detailing completed work
- Reviewing and approving final reports

A4.5.2 MPCA Quality Assurance Coordinator

William Scruton is the MPCA QA Coordinator. Specific responsibilities include:

- Reviewing and approving the QAPP
- Assisting in review of the sampling protocols
- Conducting external performance and system audits of laboratory and field activities, as needed
- Reviewing and evaluating analytical field and laboratory procedures

A5 Problem Definition and Background

This QAPP was developed in preparation for a Phase B investigation planned for the Site, but with enough flexibility to address potential future investigation or remediation at the Site. Barr Engineering Co. (Barr) prepared this QAPP in accordance with the guidance and requirements for Quality Assurance Project Plans set out by the *EPA Requirements for Quality Assurance Project Plans* EPA QA/R-5 (March 2001), *Guidance for Quality Assurance Project Plans* EPA QA/G-5 December 2002, and on the Minnesota Pollution Control Agency Quality Assurance Project Plan Guidance (February 2012).

A5.1 Problem Definition

As detailed in the following paragraphs, both Freeway Landfill and Freeway Dump are unlined facilities, which were constructed inconsistent with modern landfill design. The presence of waste in unlined facilities presents potential risk to adjacent receptors, which include both groundwater and surface water bodies (the Minnesota River, a wetland complex, and a potential future lake associated with the current Kraemer Quarry).

A5.2 Site Background

The Site comprises three primary project areas (Figure 1): the Freeway Dump (Dump), the Freeway Landfill (Landfill), and the Freeway Transfer Station (Transfer Station). All three areas are located in Burnsville, Dakota County, Minnesota and are owned by either a McGowan family entity, including the R.B. McGowan Company, Inc., the Michael B McGowan Trustee, or Freeway Transfer Company, Inc. This section provides a descriptive and historical summary of each project area. In addition to the three primary project areas, investigation activities are planned on the adjacent properties, which are described in the following paragraphs. Dewatering associated with mining operations at the adjacent Kraemer Quarry, which is present to the south of the Landfill, plays a significant role in the site conceptual model. Dewatering has lowered the groundwater table significantly and has reversed the groundwater flow direction

Freeway Dump

Freeway Dump is an unlined, inactive dump located at 11937 Highway 35 W (Parcel ID: 02-03410-38-010), Burnsville, at the north end of the east service road for Highway 35W, north of the Cliff Road interchange (Figure 2a). The Dump encompasses approximately 28 acres and has recently been used as a golf driving range. Two trailers and one small building are located on the Property. The

surrounding properties include the wetlands of the Minnesota Valley Wildlife Refuge to the north and east, the Edward Kraemer and Sons quarry (Kraemer Quarry) to the west (west of Highway 35W), and commercial properties to the south, including storage facilities and a car dealership.

The Dump is a mostly flat-top mound that sits above the surrounding wetland to the north and east. The general topographic gradient of the Dump and the land near the Dump trends to the north towards Black Dog Lake and the Minnesota River. The surrounding wetland is located at an elevation ranging from approximately 700 feet above mean sea level (MSL) along the north perimeter to about 710 feet MSL to the southeast of the Dump. The elevation of the Dump ranges from approximately 720 feet MSL along the north boundary to 730 feet MSL in the south. The raised elevation of the Dump extends beyond the north and east boundaries of the Dump property.

The Dump property was purchased by Richard McGowan and his business partner Jim Vallez sometime around 1960. Although it is not certain exactly when the dump became active and started receiving waste, some reports indicate that dumping began as early as 1960. A review of historical aerial photographs indicate that the Dump was active between 1960 and 1969. The Dump initially accepted ash from a nearby power plant and later accepted other refuse including municipal solid waste and construction waste (MPCA, 2017). After the Dump ceased operating in 1969, the property remained unused until 1993, when the driving range operations began.

Previous investigations of the Dump have been conducted, starting with the 1987 Preliminary Assessment (PA) conducted by the MPCA (MPCA, 1987). The PA was prompted by concerns from the U.S. Fish and Wildlife Service (USFWS), whose property abuts the Dump to the north and east. USFWS had observed stressed vegetation, erosion, and waste materials at the eastern edge of the Dump. MPCA identified dichlorodiphenyltrichloroethane (DDT) and PAHs in soil samples collected from the perimeter of the Dump and concluded there were exposure risks from the Dump, including the groundwater and surface water migration pathways. Following the Preliminary Assessment, the Dump was placed on the CERCLA inventory of potentially hazardous waste sites.

A subsequent investigation was conducted in the early 1990's, as documented in the Screening Site Inspection Report (MPCA, 1992). The investigation included soil and groundwater testing. Organic compounds and metals contamination were detected in soil and groundwater, and additional investigation was recommended. An additional investigation was conducted in 1997/1998 by the MPCA during which nine monitoring wells were installed around the perimeter of the Dump. Groundwater sample results indicated the presence of arsenic, boron, manganese and low levels of

VOCs and PCBs. In the fall of 2003, response actions including grading and drainage improvements were completed as noted in a correspondence between the MPCA and McGowan (MPCA, 2004).

Freeway Landfill

Freeway Landfill is an unlined, inactive landfill located just south of the Minnesota River (Figure 2b). The surrounding properties include the U.S. Salt Company to the north and Highway 35W to the east. Kraemer Quarry is located to the south. The vacant land to the west is also owned by Kraemer Quarry.

The Landfill consists of several parcels, totaling approximately 189 acres, 131 of which were used for placement of waste during landfill operation and approximately 58 of which include a quarry and undeveloped land (Liesch, 1993). The Landfill property comprised multiple parcels that were purchased from several different owners sometime in 1968 by Richard McGowan. Prior to the Landfill operating, the area was mostly wetland and undeveloped, with the exception of farming activities visible in the 1937 aerial photo and a few small structures that were located north of the frontage road on the south bank of the Minnesota River, visible in the 1966 aerial photo.

Prior to landfill operations commencing, the topography of the Landfill area likely varied from 696 to 705 feet MSL (Liesch, 1991). According to current Lidar survey data (Fugro and MDNR, 2011), the maximum elevation of the Landfill is approximately 750 feet MSL at its peak near the center of the property. The ground surface slopes downward in all directions to an elevation of approximately 700 feet MSL at the property limits. The ridge on the east side of the Landfill is part of an intermittent surface water channel that runs north to the river, between the Landfill and Highway 35W.

The Landfill began accepting waste in July of 1969 under a conditional use permit issued by the City of Burnsville. In October of 1971, the MPCA issued the Landfill a permit (No. SW 57). From a review of historical aerial photos, it appears that Landfill operations began in the northeast corner of the property and then expanded to the south. In the late 1970s and 1980s, environmental regulations were significantly updated in response to evolving knowledge about environmental contaminants and associated risks to human health and the environmental. Landfill regulations were updated to require engineered liners and caps for new landfills. Based on concerns at the Site, the Landfill was added to the Superfund National Priorities List in 1986 (MPCA, 2015). Under the new regulations, landfill owners were requested to either make necessary upgrades to their facilities or to stop accepting waste. In 1990, Freeway Landfill stopped accepting waste. It is estimated that approximately 5 million cubic yards of waste were deposited in the 131-acre area of the Landfill.

Previous investigations have been conducted at the Landfill, including remedial investigations conducted on behalf of the Landfill owner (CRA, 1988 and Liesch, 1991) and environmental assessments conducted on behalf of the MPCA. More recently, in 2005, a subsurface investigation was conducted on behalf of the MPCA throughout the Landfill site that included nearly 70 soil borings and detailed surveying to assess the topography and subsurface conditions (FES, 2005).

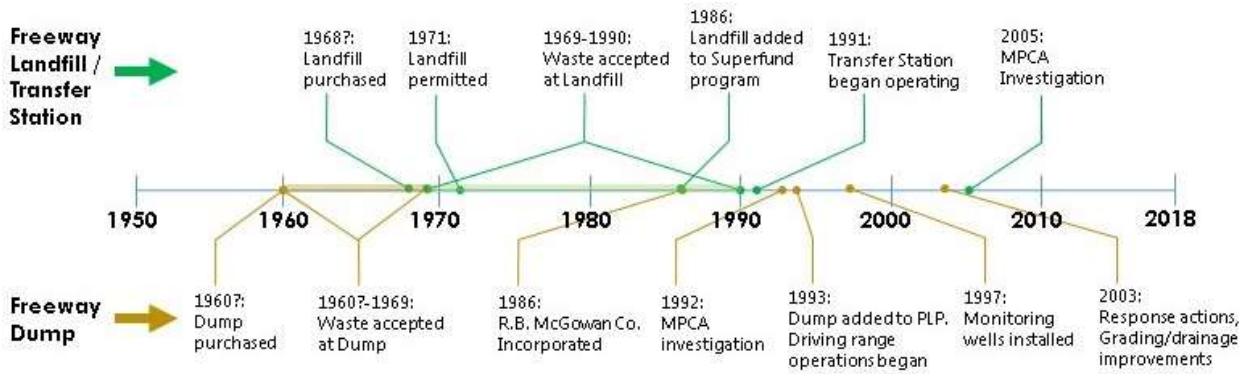
Freeway Transfer Station

The Transfer Station is located at 11501 Embassy Road (Parcel ID: 02-15600-01010), Burnsville. The Transfer Station is located on the east side of the Landfill property, approximately 1,500 feet south of the Minnesota River, and currently operates as a waste processing, recycling, and hauling facility.

The Transfer Station was constructed sometime in the late 1980s and operates on a 12-acre parcel bounded by the Freeway Landfill to the north, south, and west. The Transfer Station is currently in operation and has been since 1991 (Liesch, 1993).

Topographically, the Transfer Station is located in a depressed area at approximately 710 feet MSL. Surrounding the Transfer Station to the north, south, and east is a berm feature that rises to approximately 745 feet MSL, and to the west is the access road that rises out from the station to Landfill grade of approximately 735 feet MSL.

An approximate chronology of significant milestones is provided below.



A6 Project/Task Description

The purpose of this QAPP is to document the steps that will be taken to investigate water (including groundwater and surface water), solid media, and soil gas at the Site. The objective of the analytical sampling is to characterize existing conditions in support of remedial design.

It is anticipated that samples will be analyzed for the parameters listed in Tables 3 and 4. Also, where the analysis includes individual parameters and the criteria is based on a total, as in the polychlorinated biphenyls (PCBs) and B(a)P-equivalents, the detected target parameters will be added together and then compared to the criteria. Specifically, in the case of the B(a)P-equivalents, the current potency equivalency factor is applied to each parameter and then summed before being compared to the criteria.

Tables 3 and 4 list each analyte, the method reference, laboratory RL, and current criteria (if applicable).

The estimated schedule for the work described by this QAPP will follow the following general timeline. The schedule may vary from that described as warranted by Site conditions, weather or other unforeseen impacts.

QAPP Preparation and Finalization – Early January

Phase B Work – Begins in late January or early February

Data Reported by Laboratory – approximately 10-21 business days after submission

Draft Report Submission – Early May

A7 Quality Objectives/Criteria for Measurement Data

A7.1 Project Data Quality Objectives

Project data quality objectives (DQOs) were designed to ensure that the type, quality, and quantity of data used in decision-making were appropriate for their intended application. The seven-step DQO process (MPCA, 2012b) was used to develop the overall approach to each study element, and ultimately to design the various field and laboratory investigations. The seven steps include state of the problem (define the problem), identify the decision, identify the inputs to the decision, define the boundaries to the study, develop a decision rule, specify tolerable limits on decision errors, and optimize the design for obtaining data.

The DQOs for this project are described in Table 5.

A7.2 Data Quality Indicators

The data quality objectives for the project are to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide the level of data required for determining the characteristics of the various environmental media. Specific procedures for sampling, chain of custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field instrument, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the objectives for the six data quality indicators (precision, accuracy, representativeness, comparability, completeness, and sensitivity), along with the means by which they are measured to monitor the compliance to the project needs.

A7.2.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Precision in the laboratory is assessed through the calculation of RPDs for laboratory control samples/laboratory control sample duplicates (LCS/LCSD), matrix spike/matrix spike duplicates (MS/MSD), and laboratory duplicates. Laboratory duplicates or MS/MSD samples will be analyzed at the frequency presented in Table 6. Laboratories' precision criteria are included in the laboratories' reports and/or analytical SOPs and in Tables 3 and 4. The laboratories' SOPs are included in Appendix B.

A7.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value and measures bias in a measurement system.

A7.2.2.1 Field Accuracy Objectives

Accuracy in the field is assessed through field equipment calibration and maintenance, use of field blank samples, and through the adherence to sample handling, preservation, and holding time requirements. Field equipment is tested and maintained when needed using manufacturers' recommendations. Pace's procedure manuals (Water, Soil, and Soil Gas) outline the field equipment's precision, accuracy limits, and preventive maintenance procedures. Field equipment SOPs used for this project are provided in Appendix E.

Field quality control samples will be collected and sent to the laboratory at the frequency presented in Table 6.

A7.2.2.2 Laboratory Accuracy Objectives

Accuracy of laboratory results may be assessed using the analytical results of laboratory control samples/laboratory control sample duplicates, matrix spike/matrix spike duplicate samples, surrogate standards, and method blanks. The percent recovery (%R) for matrix spikes will be calculated using the following equation (for LCS and other laboratory-prepared samples, B is zero):

$$\%R = \frac{A - B}{C} \, x \, 100$$

Where: A = The analyte concentration determined experimentally from the spiked sample

B = The background level determined by a separate analysis of the unspiked sample

C = The amount of the spike added

LCS, MS, and method blank samples will be analyzed at the frequency presented in Table 6. Laboratories' accuracy criteria are included in the laboratories' reports and/or analytical SOPs and in Tables 3 and 4. The laboratories' SOPs are included in Appendix B. The results of method blanks should not have a reportable concentration of any target analyte above its RL (exceptions may be made for the common laboratory contaminants). The data validation accuracy limits are listed in

Tables 3 and 4. An exceedance of these limits will result in corrective actions by the Barr QA manager. These corrective actions are discussed in more detail in Section D1.4 of this QAPP for laboratory content.

A7.2.3 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Rejected data, or sampling points that do not yield a usable sample, count against the percent completeness. Field completeness goals for each project will be greater than 90 percent. It is expected that the laboratories will provide data meeting QC acceptance criteria for 90 percent or more of all samples tested. However, other factors may affect the decision to resample for lost or otherwise invalid data, such as if the sample was collected for confirmation of an earlier detection, or if the same parameter at the same well was somehow invalidated during consecutive sampling events. Following completion of analytical testing, completeness will be calculated as a percent using the following equation:

$$Completeness\ (\%) = \frac{Number\ of\ valid\ data}{Number\ of\ targeted\ data}\ x\ 100$$

Data = # of samples X # of parameters per sample

A7.2.4 Representativeness

Representativeness is defined as a measure of the degree to which data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. As described in the work plans, the sampling network has been designed to provide samples representative of site conditions. During development of this network, consideration has been given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the monitoring program. The rationale of the sampling network is based on the data needed to develop a remedial design and the presence of receptors that may potentially be affected by the presence of waste materials.

The representativeness criteria will be satisfied by the use of proper sampling techniques and appropriate analytical procedures. This will be measured on this project through the use of matrix spikes, matrix spike duplicates, and field blanks.

A7.2.5 Comparability

Comparability is defined as the confidence with which one set of data can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling methods, sample preparative procedures, analytical methods, and holding times. Comparability will be satisfied by ensuring that the sample plan is followed and proper and consistent sampling techniques are used. This will be accomplished by the project team with the use of matrix spikes, matrix spike duplicates, and field duplicates as described in Section B5.

A7.2.6 Sensitivity

Sensitivity expresses the methodology's and laboratory's ability to meet or exceed the applicable criteria. Laboratory sensitivity will be assessed by comparing the analytical RL to the applicable site criteria. Current laboratory RLs are less than site criteria or are deemed acceptable for the purposes of this project.

A8 Special Training Requirements/Certification

A8.1 Field Personnel

Barr field personnel will be under the supervision of the Barr project manager. They will be trained to follow the health and safety procedures as outlined in the project health and safety plan (PHASP). The PHASP provides guidelines, requirements, and procedures intended to help protect the health and safety of all employees who will participate in the field work. The MPCA's sampling subcontractor (Pace) field personnel will be under the supervision of the Pace project manager. Pace will develop their own PHASP and will provide training in relation to proper field equipment operation, sampling and preservation techniques, sample handling and custody, and quality control to field personnel. Training records are kept in Pace's human resources personnel training files located in the human resources department.

A8.2 Laboratories

The laboratories utilized for this project will have the appropriate certifications necessary to perform analysis in the state of Minnesota, where applicable. A summary of the laboratories' certification documentation is included in Appendix C. The laboratories' personnel training will be conducted and monitored by the laboratories' QA managers as outlined in Section 1.9 of Pace's Field Services Division QA manual, Section 1.9 of Pace's Analytical Laboratory QA manual, and in Section 18 of MDH's QA manual (Appendix B).

A9 Documentation and Records

The following is a list of information that must be documented and records that must be reported or available for review. The list is not intended to be a complete list of every item, rather general guidance on required information. Project files will be archived in accordance with Barr's project records retention guidelines (Appendix F).

A9.1 Field Records

Field records should include:

- Sample collection records
- Chain of custody
- QC sample records, if applicable
- Field measurement results
- Equipment calibration documentation
- Corrective action reports
- Observation notes
- Names of the personnel on site

A9.2 Laboratory Records

Laboratory records should include:

- Date of sample analysis
- Sample management information (e.g., receipt, numbering, handling)
- Analytical procedures
- Notes of deviations from procedures
- Instrument standardization
- Sample preparation and analysis information

- Results of analytical testing
- Reporting limits
- QC criteria and results
- Data handling information
- Electronic data deliverable (EDD) – Barr and MPCA defined EQuIS formats (as required for the project)

Laboratory records will be managed in accordance with Section 6.5 of Pace's QA manual and Section 14 of MDH's QA manual (Appendix B). In the event there are concerns in the future, Barr will contact the laboratory before the records are destroyed in accordance with the policy.

A9.3 Quality Assurance Project Plan

The Barr QA manager is responsible for ensuring that a copy of the QAPP, any subsequent QAPP revisions, and/or QAPP addenda are provided to everyone listed on the distribution list of this QAPP to ensure the most current approved version of the QAPP is available for use.

B Data Generation and Criteria

B1 Sampling Process Design

The sampling procedures to be used in this site investigation will be consistent for the purpose of this project. The sampling locations are shown on Figures 1 and 2 and the rationale behind the samples is included in Table 2.

The estimated number of samples that will be collected is listed in Table 2.

B2 Sampling Method Requirements

B2.1 Field Sampling Equipment and Procedures

Analytical samples will be collected in the field in accordance with standard operating procedures included in Appendix E. A direct-push sampling unit, drilling rig, or hand-sampling equipment are expected to be used to collect the soil samples using coring, split-spoon sampling, and hand-sampling gear. Bailers (stainless steel or disposable), direct sampling, or pumps (peristaltic, submersible or bladder) will be used to collect water samples, as appropriate for the sample location. Soil gas samples will be collected with summa canisters under vacuum.

Investigation derived waste (IDW) will be disposed of or managed in accordance to Pace's SOPs, SOT-ALL-W-002 *Waste Handling and Management* and SOT-ALL-C-001 *Sample Management*.

Additional information on the Pace field equipment (including decontamination procedures) and sampling techniques is provided in the Pace's Field SOPs located in Appendix E.

B2.2 Field Sampling Documentation

Field data sheets will provide the means of recording data collecting activities. Entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory. Field data sheets will be loose sheets which will be generated into a report after the event. Each field sheet will contain the following minimum information:

- Project name
- Field work date
- Field staff name

Field data sheets will include a variety of information depending on the purpose of the site visit. Measurements made, type of sampling equipment used, and samples collected will be recorded. Entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, dated, and initialed. Whenever a sample is collected or a measurement is made, a description, name, or number of the location shall be recorded. Additional information on field documentation is included in the SOPs in Appendix E.

B2.3 Sample Containers, Preservation Techniques, and Holding Times

The sample containers, preservation, and holding times associated with the anticipated analytical tests are provided in Table 7.

B2.4 Field Corrective Action

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or quality control performance which can affect data quality. Corrective action in the field can be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP, etc.), sampling procedures and/or field analytical procedures require modification due to unexpected conditions, deficiencies are identified in field audits, etc.

Technical staff and project personnel will be responsible for reporting suspected technical or QA nonconformance or suspected deficiencies of any activity or issued document by reporting the situation to the Barr project manager or designee. Field staff may take corrective actions to correct deficiencies or nonconformances with field equipment and report actions to the Barr project manager upon completion, as discussed further below. The Barr project manager will be responsible for assessing the suspected problems and may consult with the Barr QA manager when deciding if the situation has the potential to impact the quality of the data. If the situation is expected to impact the quality of the data, corrective action will be initiated by the Barr project manager. The Barr project manager will be responsible for ensuring that corrective actions are initiated by:

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a record of nonconformances
- Reviewing corrective actions taken
- Ensuring nonconforming situations requiring corrective action are reported in the final reports to the MPCA

If appropriate, the Barr project manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

When it becomes necessary to modify a program, the Barr project manager or designee will notify the MPCA project manager of the anticipated change and implement the necessary changes after obtaining the approval of the MPCA project manager.

The Barr project manager is responsible for controlling, tracking, and implementing the identified changes. Reports on all changes will be distributed to all affected parties, which include the MPCA. The MPCA project manager, or designee, will be notified whenever program changes in the field are made.

For field equipment issues, implementation of the corrective actions may be performed by the Pace field team at the time of occurrence. Corrective action for field measurements may include:

- Repeating the measurement to check the error
- Checking for all proper adjustments for ambient conditions such as temperature
- Checking the batteries
- Verifying the calibration and recalibrating the equipment, if needed
- Replacing the instrument or measurement devices
- Stopping work (if necessary)
- Informing the Barr project manager

Corrective actions are verified as effective when the actions were implemented as intended, the root cause was addressed, and recurrences of similar issues in the future have been prevented. In cases where removal of the root cause may not be possible, there should be a reduction or less severe appearance of the issue. The scale of verification should match the scale of the actions taken. Corrective actions that are not effective will require additional corrective action.

B3 Sample Handling and Custody

Proper sample handling and custody procedures are crucial to ensuring the quality and validity of data obtained through field and laboratory analyses. This custody is in three parts: field sample collection, sample custody within the laboratory, and final evidence files. An item is considered in custody if it is:

- In a person's possession.
- In view of the person after being in their possession.
- Sealed in a manner that it cannot be tampered with after having been in physical possession.
- In a secure area restricted to authorized personnel.

B3.1 Field Chain-of-Custody Procedures

Samples will be collected following the sampling procedures documented in Appendix E and approved work plans. In order for the laboratory to generate a compliant EDD, the MPCA COC will be used for sample submission to capture all of the required information for the EDD. This COC is included in Appendix H and is available on the web page: https://earthsoft.com/products/edp/edp-format-for-mnpca/. Sample site-specific identification numbers corresponding to the sample location name as shown in Table 2, sample collection date and time, and number of containers will be noted on the chain of custody. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. The QA/QC samples will be identified by the following codes, followed by a sequential number.

M = Field (masked) Duplicate Sample – Example: M-1, M-2

FB = Field Blank Sample – Example: FB-1, FB-2

Samples will be packaged and shipped according to Pace's SOPs for sample collection and transport to the laboratory (Appendix B). The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain of custody intact.

- The Pace field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

- The sample containers will be identified by use of sampling labels with location numbers, and date and time of collection.
- Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a field data sheet notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
- Samples will be properly packaged for shipment with a completed and signed chain-of-custody (COC) record enclosed in a plastic bag.
- Shipping containers will be sealed and secured with tape for shipment to Legend via an overnight delivery service for receipt within two days of sample collection. Samples may be held for shipment, under appropriate storage conditions, up to four days (e.g., due to holidays or special circumstances) unless sample holding times dictate shorter delivery.

When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This record documents transfer of custody of samples from the sampler to another person or to the laboratory. This will not include transport people such as messengers or overnight delivery service employees (e.g., FedEx).

Shipments will be accompanied by the COC record identifying the contents. The original record will accompany the shipment, and the pink and gold copies will be retained by the sampler for returning to the sampling office.

B3.2 Sample Custody within the Laboratory

Sample handling and custody are described in Section 2.0 of Pace's Quality Assurance Manual and in Section 24 of MDH's Quality Assurance Manual (Appendix B).

B3.3 Custody of Evidence File

Until completion of the project, correspondence, laboratory reports, and data will be maintained in Barr project files. Laboratory reports and field data are maintained and stored in their original format. The Barr project manager will direct maintenance of the project file. Following completion of the project, the evidence file will be stored in the Barr project file storage area or transferred to a secure document storage facility. The files will be maintained as required by Barr's project records retention guidelines (Appendix F).

B4 Analytical Methods Requirements

B4.1 Field Analysis

Trained Pace personnel will perform the field analytical methods. Pace personnel will perform analytical screening in the field. Soil field screening will include inspection for visual evidence of contamination (i.e., incidental odor, discoloration, and sheen) and tested for headspace volatile organic vapor concentrations, in accordance with SOPs included in Appendix E. Field screening during water sampling will include the use of a Photo-Ionization Detector (PID) to measure the aromatic content at the wellhead and elevated PID reading(s) recorded on the COC as well as in field notes. Field-screening methods will be selected to allow for real-time data, while meeting data quality objectives. Samples analyzed by Barr personnel will be either returned to the sample site or properly disposed of in accordance with local, state, and federal disposal regulations.

B4.2 Laboratory Analysis

The laboratories performing the analytical testing are detailed in Table 1. A list of anticipated laboratory methods, their corresponding RLs, and applicable criteria is provided in Tables 3 and 4. Analytical methods were selected to provide adequate RLs for compounds of interest based on the final intended data usage. SOPs have been prepared for the methods used for analysis of samples for this project. The laboratories' SOPs and analytical method number are included in Appendix B. Each of these SOPs is based on an analytical method published by the USEPA, Standard Methods, or other recognized source as available.

The laboratory data deliverable will be generated by the laboratory within 10-15 working days from sample submittal. Once the laboratory deliverable has been evaluated, Barr may compare the data with project acceptance criteria and/or background concentrations.

B4.3 Laboratory Nonconformance and Corrective Action

The laboratory takes appropriate steps necessary to ensure sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconformances are often handled at the bench level by the analyst who reviews the preparation or extraction procedure for possible errors, verifies spike and calibration mixes, checks the instrument

calibration, checks the analytical data to determine if other samples in the batch were affected or if it was isolated to a single sample, verifies variables being used in the final result calculation, etc. If the problem persists or cannot be identified, the matter is referred to the supervisor, manager, and/or QA department for further investigation. Reanalysis of the sample may be performed. If the nonconformance has not been corrected and the validity of the data is in question, the laboratory director, Barr QA manager, or Barr project manager will contact the client. The client contact should be documented and included in the job file.

The corrective actions are performed prior to release of the data from the laboratory and will be documented in a corrective action report (signed by analyst, section leader, and QA manager), and the narrative data report sent from the laboratory to the Barr QA manager. As part of their report, the laboratory may qualify (flag) their data for such items as concentration below RL, estimated concentration due to poor spike recovery, or concentration of chemicals also found in laboratory blanks. Nonconformances and corrective actions are discussed in Section 8 of Pace's QA manual and in Section 10 of MDH's QA manual. Copies of the laboratories' corrective action report are also available in Appendix B. The section leader and QA manager are responsible to ensure that the corrective action taken was effective. Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Section leaders are accountable to the laboratory director to ensure final acceptable resolution is achieved and documented appropriately.

B5 Quality Control Requirements

B5.1 Field Quality Control Requirements

QC procedures for field equipment will include calibrating the instruments per manufacturer's instructions or as described in the SOPs located in Appendix E and measuring duplicate samples. Pace's Field Manuals outline the field equipment precision and accuracy limits and preventive maintenance procedures. Possible corrective actions are summarized in Section B2.4.

Assessment of field sampling precision and bias will be accomplished through collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in the SOPs located in Appendix E and whenever possible, samples will be collected from the cleanest location to the dirtiest whenever the nature of the contamination is known. A summary of field QA/QC samples for this project is presented in Table 6.

Field blank samples consist of analyte-free water exposed to environmental conditions at the sampling site by transferring from one sample container to another or by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time equivalent necessary to fill a container. It measures the potential for sample cross contamination due to site conditions. Field blanks will be submitted to the laboratory with investigative samples and analyzed for the same parameters as the investigative samples. The results of field blanks should not have a reportable concentration of any target analyte above its RL (exceptions may be made for the common laboratory contaminants).

Trip blank samples are used when sampling volatile organic compounds (VOC). Analyte-free water is used for water samples and methanol (or other applicable sample preservative) is used for soil samples. They are prepared or provided by the laboratory along with the VOC sampling containers prior to a sampling event. Trip blank sample containers are not to be opened in the field and are to accompany the VOC samples during collection, storage, and transport to the analytical laboratory. The trip blanks should be listed on the chain-of-custody (COC) along with the samples and the analysis required. The purpose of the trip blank sample is to determine the extent of potential contamination introduced during sample transport and handling. The analytical results of trip blanks should not have a reportable concentration of any target analyte above its RL (exceptions may be made for the common laboratory contaminants).

Equipment blank (or rinsate blank) samples are prepared on-site by pouring analyte-free water through decontaminated sample collection equipment (e.g., bailer or pump, hand-trowel, etc.) and collecting the "rinsate" in the appropriate sample container. If collecting a blank for dissolved metals or dissolved organic carbon, the rinsate will be filtered before adding to the sample container. In addition to the field sources of contamination that may be introduced in the transferring of samples to one vessel to another, an equipment blank also tests the potential cross contamination from incomplete decontamination. Generally, blanks are collected for each parameter of interest.

Field precision will be assessed through the collection and analysis of field duplicate samples. RPDs will be calculated for the detected analytes from investigative and field duplicate samples where both the native and field duplicate sample concentrations are greater than five times the RL. The equation to be used to determine precision (RPD) and the field duplicate precision limit are presented in Section A7.2.1. An exceedance of these limits will result in corrective actions by the Barr QA manager.

B5.2 Laboratory Quality Assurance Program Overview

The purpose of the laboratories' quality assurance programs is to ensure that analytical data is scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. QA oversight is performed throughout sample processing from initial order/entry, through the analytical system, to the final report. This is done through various policies, procedures, and quality control checks. The QA managers at each laboratory have the authority and responsibility for implementing, maintaining, and improving the quality system and for ensuring compliance with all regulatory compliance quality standards. The applicable accreditations/ certifications held by the laboratories is provided in Appendix C. The QA managers work with laboratory staff to establish effective quality control and assessment processes and have the authority to stop work in response to quality problems.

B5.2.1 Internal Quality Control Procedures

Internal quality control procedures are established, implemented, and maintained. They include, but are not limited to, auditing, data integrity training, document control, control of records, measurement traceability, analysis of proficiency testing (PT) samples, and internal auditing. Detailed information regarding each of these procedures, along with other internal laboratory policies and procedures, are provided in the laboratories' QA manuals in Appendix B. These policies and procedures are established in order to meet requirements of accreditation bodies and applicable

programs, as well as client's quality objectives. QC procedures are used to continually assess performance of the laboratory and quality systems. The laboratory maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical QC checks with analyses, and by observing sample custody requirements.

B5.2.2 Laboratory Quality Control Checks

The laboratories ensure the production of quality analytical data through the use of overall quality assurance systems that are supported by documented quality control checks. The particular types and frequencies of quality control checks analyzed with samples are defined in the laboratories' SOPs and Quality Assurance Manuals (QAMs). Laboratory acceptance criteria is included with each analytical report and a summary of laboratory QA/QC limits are presented in Tables 3 and 4. An exceedance of these limits will result in corrective actions by the Barr QA manager, as described in Section D1.4.

B6 Instrument/Equipment Testing, Inspection and Maintenance

B6.1 Field Equipment

Pace staff perform routine preventive maintenance of instruments based on manufacturers' recommendations and schedules. Critical spare parts such as pH probes, and batteries will be kept on site to reduce downtime. Backup instruments and equipment will be available on site or within one-day shipment to avoid delays in the field schedule. As described in Section B7 of this QAPP, Pace staff will calibrate or analyze calibration verification standards where appropriate to determine if the instrument is operating properly prior to beginning the analysis.

Field equipment maintenance information is provided in the Pace Field SOP Manuals (Appendix E) outlines the field equipment's preventive maintenance procedures.

B6.2 Laboratory Equipment

As part of their QA program, preventive maintenance is conducted by the laboratories to minimize the occurrence of instrument failure and other system malfunctions. The maintenance is performed by qualified laboratory staff or under commercial service contracts. Responsibility for ensuring that routine maintenance is performed lies with the laboratories' section supervisor. Each laboratory section maintains a critical parts inventory. This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repair. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring onsite manufacturer repair. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks and include the following information:

- Instrument's serial number
- Date instrument was received
- Condition when received (new, used, reconditioned, etc.)
- Date instrument was placed into service
- Prior history of issues or repair (if known)
- Details and symptoms of problem

- Repairs and/or maintenance performed
- Description and/or part number of replaced parts
- Source(s) of the replaced parts
- Analyst's signature and date
- Demonstration of return to analytical control

Preventive maintenance procedures, frequencies, etc. are available for each instrument. They may be found in the various SOPs (Appendix B) for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

B7 Instrument Calibration and Frequency

This section describes procedures for maintaining the accuracy of the instruments and measuring equipment which are used for conducting field and laboratory analyses. These instruments and equipment are calibrated or verified prior to each use or on a scheduled, periodic basis.

B7.1 Field Instrument Calibration

As applicable, field instruments used to gather, generate, or measure field environmental data will be calibrated, or have the calibration verified, prior to use. Field instruments may include a photo ionization detector (PID). If acceptable calibration criteria are not achieved and applicable corrective measures (Section B2.4) do not achieve acceptable QC criteria, the following actions should be implemented:

- Remove the instrument from service
- Contact the Barr equipment technician for repair or a replacement instrument
- Notify the Barr project manager

For specific instructions on the calibration, maintenance, acceptance criteria, and conditions that will require more frequent recalibration, refer to the specific Pace Field SOPs provided in Appendix E and the manufacturers' recommendations.

B7.2 Laboratory Instrument Calibration

Procedures for initial calibration and continuing calibration verification are in place for the instruments within the laboratories. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. Section B8.2 details information on laboratory supplies and consumables.

The frequency of calibration and calibration verification, number of points calibrated, and acceptance criteria for each of the instruments to be used are provided in the laboratories' SOPs (Appendix B) and are consistent with the referenced method. Additional information is provided in Section 5.0 in Pace's Quality Assurance Manual and in Section 21 of MDH's Quality Assurance Manual (Appendix B).

In general, laboratory instruments are calibrated at multiple concentration levels for the analytes of interest. The initial calibration is verified with a second, independent source. Analysis of a standard or

extract prepared at the lowest level initial calibration standard (where applicable) provides confirmation of the established sensitivity of the method. It is not necessary to reanalyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. Calibration verification is performed at method-specified intervals. For gas chromatography/mass spectrometry (GC/MS), internal standard procedures, and/or isotope dilution procedures, mass spectra of the tuning compounds, internal standard response, and/or labeled compound recovery must meet method/SOP criteria before analyses can proceed. If criteria is not met, the system should be evaluated, and corrective action performed, as documented within the laboratories' analytical SOP, before sample analysis begins. Reanalysis of samples analyzed while the system was malfunctioning is required. Additional information on laboratory nonconforming work and corrective action is provided in Section B4.3. Records of calibration and calibration verification are maintained in laboratory reference files to provide traceability of standards and equipment.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by an accredited vendor. The calibration of each analytical balance is checked by the user each day of use with at least two Class S or S-1 weights, which assess the accuracy of the balance at low and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of the user who performed the check.

Equipment shown by verification to be malfunctioning or defective is taken out of service until it is repaired. When an instrument is taken out of service, an out-of-service sign is placed by the laboratory on the instrument. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

B8 Requirements for Supplies and Consumables

B8.1 Field Supplies and Consumables

Supplies and consumables that will be used for the projects include sample equipment, personal protective equipment, and sampling containers. Sampling equipment will be examined upon receipt from various vendors by the Pace field staff. Sampling equipment that has obvious physical damage should also not be used. In the case of sampling gloves, if any physical tears or discoloration exists on the gloves, they should not be used. Other consumable equipment will be examined on site and a determination as to its usability will be made based upon the product's physical appearance.

The laboratory will supply pre-cleaned, certified sample containers from approved vendor sources for the analytes/methods cited in Table 1. Pre-preserved (where applicable) sample containers will be shipped to the Barr field office in accordance with federal shipping guidelines. Sample containers will not be accepted if there is more than 10% breakage of the containers upon receipt. If the sample containers contain preservative and are broken in the receiving container, none of the sample containers in that container will be used for sampling. Sample containers, preservative, and holding times for the analyses are detailed in Table 7.

B8.2 Laboratory Supplies and Consumables

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors, where possible, have fulfilled the requirements for 9001 certification and/or are ISO 17025 accredited. The laboratories rely on a primary vendor for the majority of its analytical supplies.

Standards used at the laboratories are prepared from pure standard materials or purchased. Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material. The standards in solution are stored in a discrete freezer or refrigerator in the applicable laboratory section. Each standard is discretely designated. The information is stored in a standards notebook and/or electronic database. Additional purchasing of supplies is provided in Pace SOP S-MN-L-143, "Purchasing Laboratory Supplies," and additional measurement traceability information is provided in Section 5.1 of Pace's Quality Assurance Manual and in Section 7 of MDH's Quality Assurance Manual, respectively (Appendix B).

B9 Data Acquisition Requirements for Non-Direct Measurements

Existing chemical data from previous site investigations were used to design the scope for this investigation. Historical data were obtained by following the QA/QC protocol, sampling and analytical procedures, and data validation guidelines defined in the previously approved QAPP and QAPP addenda for the site. The data obtained prior to this QAPP was reviewed and is deemed acceptable for the purposes of this project.

B10 Data Management

Data generated through field activities or by the laboratory shall be reduced and verified prior to reporting. No data will be disseminated by the laboratory until it has been subjected to the procedures summarized in subsections below.

B10.1 Data Collection

Most outputs are generated through computer programs that have been validated by the manufacturer prior to laboratory purchase of the instrumentation. The instruments have programs available for the analysts to manually verify integrations and quantitations. Manual verification is routinely performed.

B10.2 Data Reduction

Data reduction includes processes that may change the instrument/computer-generated values, quantity of data values or numbers of data items, and frequently includes computation of summary statistics. In most cases, a programmable calculator, computer spreadsheet, or computer program is used to generate statistical information. The documentation allows the reviewer to verify the validity of the data reduction process.

In the data review process, the data produced are compared to information concerning the sample processing history, sample preparations, sample analysis, and associated QA data to evaluate the validity of the results. In addition, any project-specific requirements are reviewed for data compliance.

B10.2.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. The field forms are included in Appendix B where applicable, which are also stored in the document management software and are updated on an ongoing basis. The use of PIDs will generate measurements directly read from the meters following calibration per manufacturer's recommendations, as outlined in Section B7.1 of this QAPP. Such data will be written into field data sheets immediately after measurements are taken and are described in the Pace field SOPs included in Appendix B. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results

forms required for this study are being filled out, the Barr QA manager and Barr project manager will proof the forms to determine whether any transcription errors have been made by the field crew.

B10.2.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be conducted according to the following general protocol. Results will be generated by the analyst who performs the analysis and prepares the raw data for reporting. The data will be initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOP (Appendix B). The resulting data set is either manually entered into an electronic report form or is electronically transferred into the report.

Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. If errors are noted, manual editing of data is allowed per established procedures. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. Nonconformances are handled as per Section B4.3 of this QAPP.

Once the primary analyst has checked the data for accuracy and acceptability, the data and report hardcopy is forwarded to the supervisor or second qualified analyst who reviews the data to ensure that the QC criteria have been examined and any deficiencies noted and addressed.

Upon approval, the final report will be sent to Barr and reviewed based on Barr's SOPs for Routine Data Evaluation which are included in Appendix D and discussed further in Sections D1.3 and D1.4. Unacceptable data shall be appropriately qualified by the laboratory in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. More information on laboratory data reduction can be found in the individual analytical SOPs located in Appendix B.

B10.3 Barr Data Review and Validation

Data review and validation procedures shall be performed for both field and laboratory operations. More information on Barr data review and validation can be found in Sections D1.3 and D1.4, respectively, of this QAPP.

B10.3.1 Procedures to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field notebooks, on the part of field staff members. This task will be the responsibility of the Barr project manager or other Barr technical staff, who will otherwise not participate in making any of the field measurements, or in adding notes, data, or other information to the notebook.

B10.3.2 Procedures to Review Laboratory Data

Barr data assessment will be accomplished by the joint efforts of the Barr QA manager and Barr project manager. The data assessment by the Barr project manager will be based on the criteria that the sample was properly collected and handled according to the associated work plan and QAPP. One hundred percent of the data shall be reviewed.

The data reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the Barr project manager based on the extent of the deficiencies and their importance in the overall context of the project. More information on data review, verification, and validation can be found in Sections D1.3 and D1.4 of this QAPP.

B10.4 Data Retention

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities, as described in this QAPP. Barr is the custodian of the evidence file and maintains the contents of evidence files for the site, including all relevant records, reports, logs, field report, pictures, and data reviews.

C Assessment and Oversight

C1 Assessment and Response Actions

Audits of both field and laboratory activities are conducted to verify that sampling and analysis are performed in accordance with the procedures established in the work plan and QAPP. The audits of field and laboratory activities may include two separate independent parts: internal and external audits. Results of audits are used to improve sampling procedures and SOPs.

C1.1 Field Audits

C1.1.1 Internal Audits

Internal audits of field activities (sampling and measurements) will be conducted by the Barr QA manager or designee (someone not directly associated with the assessment activity) once every five years. Audits may be performed more frequently according to site circumstances (e.g., major changes to field sampling procedures, nontypical data, or new personnel). The audits will include examination of field sampling records, field instrument calibration and operating records, sample collection, sample handling, QA procedures, and COC documentation in compliance with the established procedures. If, during the course of the internal audit, the auditor observes any practice that they feel may jeopardize the data, sampling will be suspended and the Barr project manager will be contacted to discuss the issue. If it is determined that the issue cannot be resolved, sampling will be suspended and resumed only after measures to correct the practice are determined by the Barr project manager and Barr QA manager. A copy of the field audit checklist is provided in Appendix G. The Barr QA manager will provide the completed audit checklist and identify deficiencies to the Barr project manager.

C1.1.2 External Audits

External field audits may be conducted at any time during the field operations. These audits may or may not be announced and are at the discretion of the MPCA. The audit will be conducted according to the field activity information presented in this QAPP.

C1.1.3 Corrective Actions

Corrective actions for deficiencies identified in field audits will be the responsibility of the Barr project manager. Specific information for field corrective actions is provided in Section B2.4. Corrective actions will be implemented immediately if data may be adversely affected due to

unapproved or improper use of approved methods. The Barr QA manager will recommend corrective actions to the Barr project manager. Implementation of corrective actions will be performed by the field staff and will be documented in quality assurance reports to the Barr project manager, and will be included in reports to the MPCA. Follow-up audits may be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the remediation.

C1.2 Laboratory Audits

C1.2.1 Internal Audits

Internal audits of laboratory activities are conducted under the direction of the laboratory QA manager and are comprised of system, process, and electronic data audits. A system audit is an annual audit of the implementation of the quality system in the laboratory. A process audit is an audit of the operational areas in the laboratory to evaluate compliance with operational and technical procedures. An electronic data audit examines the organic chromatographic data. All audit findings are documented and reported to the laboratory director and department managers for review. Additional information regarding laboratory audits is provided in Section 7.1 of Pace's QA manual and Section 15.1 of MDH's QA manual, respectively.

C1.2.2 External Audits

As part of their NELAP accreditations, the laboratories are audited by their primary NELAP Accreditation Body along with other non-NELAP states and agencies. Copies of the most recent NELAP certificates and scopes of accreditation applicable to work in Minnesota are included in Appendix C. Pace has participated in Barr's independent QA audit program. The audit results are on file at Barr. The laboratories' NELAP accreditations require participation in the analysis of proficiency testing (PT) samples. Results of the PT samples are reviewed by the laboratory director, QA manager, and the laboratory staff.

As part of their designation as a Principle Lab for the State of Minnesota drinking water program, the MDH laboratory is subject to an on-site audit of the facilities by the US EPA Region 5 Laboratory certification program. A copy of the laboratory's current interim certification is included in Appendix C.

C1.2.3 Laboratory Analyses Corrective Actions

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. Any audit deficiencies found or PT sample results outside acceptance criteria are investigated. Managers must respond with corrective actions correcting the deficiency within a defined timeframe and how the effectiveness of the corrective action will be monitored. Should problems impacting data quality be found during an audit, any client whose data are adversely impacted will be given written notification within the corrective action period (if not already provided). Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues. Corrective actions in the laboratory are discussed in Section B4.3.

C2 Reports to Management

C2.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of the measurements made in the field. Field documentation of field instrument calibrations, well logs, boring logs, sample identifications, etc. will be contained in the final field reports. Examples of field forms used for final field reports are included in Appendix E.

C2.2 Laboratory Data Reporting

Laboratory analyses reports will generally be submitted to Barr upon completion. The laboratory project manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the chain of custody, the report format shall consist of the following:

- Date of issuance
- Project name and number
- Condition of samples upon receipt at the laboratory
- Cross referencing of laboratory sample to project sample identification numbers
- Sample collection and receipt date
- Laboratory analysis performed
- Reference method used for analysis
- Laboratory batch number
- Sample preparation and analysis dates
- Sample results (including units and percent moisture and/or solids data used in dry weight corrections, if applicable)
- Laboratory RL for each analyte

- Quality control data and acceptance criteria (including method blank results, laboratory control sample recoveries, matrix spike and matrix spike duplicate recoveries and RPDs, surrogate standard recoveries, and/or laboratory duplicate RPDs, if applicable)
- Discussion and/or qualification of any laboratory quality control checks which failed to meet acceptance criteria
- Discussion and/or qualification of any holding times that were not met
- Data qualifier definitions
- Discussion of technical problems or other observations which may have created analytical difficulties
- Any deviations from intended analytical strategy
- Signature of the laboratory project manager
- EDD (Barr's EQuIS 4 File Format)

The EDD sample data will be verified against the laboratory hard copy report by a Barr data technician to verify that the results in the EDD and the hardcopy report accurately reflect the data collected. The EDD will be entered into a Barr computer database and the data will be output in a spreadsheet format to be used in report tables.

Data tables and figures are reviewed by the Barr project manager before the report is submitted to the MPCA for review. A copy of the laboratory report will be archived in accordance with Barr's project records retention guidelines (Appendix F).

The laboratories maintain a records system which ensures that laboratory records of analysis data are retained and available for five years from the date the laboratory report was issued. See QAMs located in Appendix B for specific details.

C2.3 Reports to Agencies

Data is reviewed upon receipt from the field and laboratory. The field and laboratory data collected under this QAPP will be reported to the MCPA project manager in a Remedial Investigation report. If the validated data are required to be in electronic format by an agency, the applicable EDD will be generated by Barr. Data submitted to the MPCA will be prepared in the required EQuIS EDD format.

C2.4 Project QA Reports

The summary report will contain a section that summarizes data quality information and the results of the data quality review. Included in this section will be the Barr QA manager report on the accuracy, precision, and completeness of the data, tabulated results of QA data and calculations, results of any performance and system audits, discussion of the QA/QC activities conducted by the laboratory, a summary of the data evaluation procedures performed by Barr on the laboratory data, and a summary of corrective actions that were implemented. All QA reports will be prepared by the Barr QA manager or designee and final QA reports will be reviewed by the Barr project manager. The QA report is distributed as part of the summary report to the individuals identified above.

In the case where corrective action is needed immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the project organization or corrective action sections of this QAPP. However, these events and their resolution will be discussed thoroughly in the report.

D Data Validation and Usability

D1 Data Review, Validation, and Verification Methods

For the purposes of this document, data verification (data review) is defined as an evaluation of performance against predetermined requirements given in a document such as an analytical method, SOP, or QA manual. It is performed during or at the end of field or laboratory data collection activities. The goal of data verification is to ensure and document that the reported results reflect what was actually done. Data validation is defined as the evaluation of the technical usability of the data. It focuses on the particular data needs for a project as outlined in project-specific documentation (e.g., SAP or QAPP). Data validation begins with the outputs from data verification. Data review and validation will be performed as presented below.

D1.1 Field Data Review and Verification

Field data are reviewed by both the Barr QA manager and project manager for completeness and transcription errors. Additionally, during preparation of the final field report, Pace technical field staff verifies their documentation for accuracy and completeness. If any errors are detected, the field personnel will be contacted and corrective action (Section B2.4) will be initiated.

D1.2 Laboratory Data Review, Verification, and Validation

Laboratory data review takes place on three levels. The first level of review occurs "at the bench." Analysts are charged with the responsibility of monitoring the laboratory QA/QC activities and verifying that systems are in control. The initial review is performed by the instrument operator or analyst who is responsible for assessing the following:

- Cross-checking sample identification numbers on work sheets, sample bottles, extract vials/digestate bottles, and instrument outputs
- Verifying preparative and analytical procedures were conducted within method suggested holding times
- Verifying that calibration, tuning, linearity, and retention time drift checks are within QA acceptance criteria

- Determining peak chromatography and other instrument performance characteristics are acceptable
- Calculating recoveries and internal standard responses (when applicable), and verifying that QA acceptance criteria are met

The area supervisor and/or technical reviewer perform the second level of review and validation of analyses for data completeness and accuracy. The review of QC analyses and applicable calibrations is completed and includes the following:

- Confirming quality control blanks meet QA requirements for contamination, and that associated sample data are appropriately qualified when necessary
- Calculating LCS, MS, and surrogate recoveries and duplicate RPDs, and confirming that accuracy and precision QA criteria are met, or qualified when necessary
- Comparing injections of a sample and comparing matrix spikes with the original unspiked sample for acceptable replication
- Validating that requested analyses were analyzed

After QC review, a final report is generated. The laboratory project manager performs the third level of review as summarized below:

- Checking target analyte lists requested
- Verifying application of any qualifiers
- Checking data report or case narrative for completeness
- Verifying QAPP specific requests have been met

Additional information on data review is provided in Section 6.3 of Pace's Quality Assurance Manual and in Section 25.4 of MDH's Quality Assurance Manual (Appendix B).

D1.3 Barr Data Review and Verification

The Barr QA manager will conduct a systematic review of the data reported by the laboratory in accordance with Barr's routine level data evaluation SOPs, located in Appendix D, which are based on quality assurance elements within USEPA Contract Laboratory Program National Functional

Guidelines and are in general accordance with applicable MPCA guidance (MPCA, 2011). Data quality evaluation procedures will use the QC acceptance limits specified in Tables 3 and 4, SOPs, and/or laboratory reports. The specific requirements which will be checked during data evaluation (where applicable) are:

- Holding times
- Preservation
- Blank data
- Laboratory control sample data
- Matrix spike data
- Surrogate data
- Duplicate sample data

The data reviewer will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies.

D1.4 Barr Data Validation

Barr data validation will be accomplished through the joint efforts of the Barr QA manager and Barr project manager.

The Barr QA manager will examine the data package for completeness. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, and QC summaries. The Barr QA manager will determine whether all required items are present and request copies of missing deliverables. The Barr QA manager will review issues found during data review and will compare QC data outside laboratory limits against the project limits as listed in Table 6 to determine usability of the data. Upon completing data verification and validation in accordance with Barr's routine level data evaluation SOPs (Appendix D), a routine level quality control report will be compiled and submitted. A copy of this report can be found in Barr's "Compendium of Data Quality Assessment Documentation" in Appendix D. The QA manager will indicate whether the data are usable as reported, usable as an estimated concentration, or unusable. Qualifiers applied during data

verification and validation will be presented on the tabulated form of the data and in the QA section of the summary report.

The Barr project manager or designee will verify that the samples were collected and handled according to this QAPP. The Barr project manager will review the historical, background, and other site data to verify representativeness and comparability are being achieved. If a specific parameter value is outside the expected, or if other issues are noted during data verification or validation, corrective actions are undertaken. Examples of corrective action may include, but are not limited to, request for laboratory data review, qualification of data, reanalysis of samples, or recollection of samples. Decisions to repeat sample collection and analyses may be made by the Barr project manager or Barr principal in charge in consultation with the MPCA project manager, or designee, based on the extent of the deficiencies and their importance in the overall context of the project. Corrective action is only implemented after approval by the MPCA project manager or their designee.

D2 Reconciliation of the Data with User Requirements

The data will be compiled from each investigation and summarized in tabular form. The analytical results will be compared to the project quality objectives that are summarized in Section A7 of this QAPP.

The data reconciliation process may involve multiple steps depending on the results of the initial QA review. Data that has been qualified (by the laboratory or by Barr) will be assessed for the particular circumstances surrounding the sample. For example, if multiple compounds are detected in a method or field blank, and in the associated samples at comparable levels (as defined in Appendix D), the data result will likely be treated as a false positive and considered to not be representative or accurate. In contrast, if the sample location is critical (e.g., compliance boundary), the data may need to be rejected and resampled. This also applies to qualifications based on failure to meet precision-based criteria for matrix spike/matrix spike duplicates or field duplicates if the sample or contaminant affected is critical to the project decision-making, in which case corrective actions may result. Corrective actions may include resampling and/or reanalysis of the sample. RLs may be elevated above appropriate criteria due to dilutions or matrix interferences, affecting the sensitivity of the analysis. In this case, the necessity of the nondetect data to decision-making will be evaluated and potential corrective actions may include reporting the data result as equal to the method RLs, using the qualified data, or resampling of critical samples. In cases where completeness criteria are not met, the completeness of the subset of data most critical for decision-making will be assessed to determine whether missing data results in decision errors. Comparability of the data at the site is best assessed by comparing results to historical or background data. If deviations from historical analytical or sampling methods occurs, the data may not be comparable to historical data, so efforts to maintain consistent data collection procedures are important.

Tables

Table 1
Laboratories
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Lab	Analyses			
Pace Analytical 1700 Elm Street SE Minneapolis, MN 55414 (612) 607-6400	**Water**		**Solids**	
	Chloride	EPA 300.0	Total Organic Carbon	EPA 9060
	Cyanide, Total	SM 4500-CN E	Grain Size Distribution	ASTM D422
	Cyanide, Free (Calc)	EPA 9014	Metals	EPA 6020A
	Hardness, as CaCO3 (Calc)	SM 2340B	Metals	EPA 6010C
	Nitrogen, ammonia, as N	EPA 350.1	Mercury	EPA 7471
	Unionized ammonia	EPA 350.1 Calc	VOCs	EPA 8260B
	Chromium hexavalent	SM 3500-Cr D	Wisconsin DRO	WIDRO
	Metals	EPA 200.7	Wisconsin GRO	WIGRO
	Metals	EPA 200.8	PAHs	EPA 8270 SIM
	SVOCs	EPA 8270D	**Soil Gas**	
	PCBs	EPA 8082	VOCs	EPA TO15
	Herbicides	EPA 8151 MDA List II	Methane	EPA 3C
	Pesticides	MDA List 1 (8270 Pest)		
	Radiochemical	EPA 900.0		
MN Dept. of Health 601 Robert S N St. Paul, MN 55164 (651) 201-5300	**Water**			
	VOCs	EPA 8260B	PFAS	MDH 555

Table 2
Sampling Design and Location Summary
Freeway Landfill, Dump and Transfer Station

Proposed Investigation Task	Location Type	Project Area	Property	Number of Locations	Task Description and Objectives
1. Monitoring Well Assessment / Groundwater Level Measurements	Existing Monitoring Wells	Landfill and Dump	Multiple	27	**Question to be answered: Which direction does groundwater currently flow, and where should new wells be located?** Access/repair existing monitoring wells. Obtain water levels to update 2015 groundwater flow directions at Site and target locations for current groundwater-surface interaction.
2. Monitoring Well Sampling Event	New and Existing Monitoring Wells	Landfill and Dump	Multiple	TBD	**Question to be answered: What is current water quality across entire existing well network for all parameters of concern?** Barr will develop a sample parameter list for one monitoring well network sampling event. Groundwater samples will be collected by MPCA laboratory contractor. Barr will provide periodic oversight during sample collection.
3. Shallow Monitoring Well Installation	Monitoring Well	Landfill	Freeway Landfill	3	**Question to be answered: Are there current impacts to river?** Assess shallow groundwater flow and quality near the boundary of the landfill and the Minnesota River. The data is intend to help firm up questions regarding the landfill's potential impact to the river under current conditions
		Dump	Xcel and Freeway Dump	4	**Question to be answered: Are there current impacts to wetland?** Assess shallow groundwater flow and quality at the presumed downgradient end of the dump near the northern boundary of the dump and the wetland (groundwater direction to be confirmed in Task 1 above). The data is intend to help firm up questions regarding the dump's potential impact to the wetland under current conditions.
4. Monitoring Well Nest Installation	Monitoring Well	Landfill	Freeway Landfill	2 (4 wells)	**Question to be answered: what are the groundwater impacts migrating from Freeway Landfill's downgradient property boundary towards Kramer Quarry dewatering operations?** Assess groundwater flow and quality at the presumed downgradient corner of the landfill near the southwestern boundary of the landfill and the Kramer Quarry (groundwater direction to be confirmed in Task 1 above). The data is intend to help firm up questions regarding the groundwater conditions near the landfill property boundary. Shallow well will be screened on top of bedrock, deeper well will be an open borehole well screened at the water table of the Prairie du Chien (with no more 20 feet of open borehole)
5. Monitoring Well WT-11B Interval Sampling	Monitoring Well	Landfill	Freeway Landfill	1	**Question to be answered: Are there vertical zones of higher groundwater impacts related to bedrock fractures that are masked by the 65' open borehole in this well?** Isolate 2 to 3 intervals in the saturated portion of the open hole section of monitoring well WT-11B. Measure water level and collect a groundwater sample from each interval isolated. Water levels and groundwater samples will provide information on the vertical groundwater flow and distribution of groundwater contaminants in the upper portion of bedrock aquifer near the presumed downgradient edge of landfill.
6. Soil Cover Evaluation	Soil Boring	Dump	Freeway Dump	5	**Question to be answered: How can the existing soil be used as part of the reconstruction of the landfill?** Soil sample of existing cover soils to confirm its suitability for reuse in the landfill closure. Proposed borings will be advanced by push probe and/or auger. Locations were selected next to previously conducted borings with known cover soil thickness.
		Landfill	Freeway Landfill	15	

Table 3a
Pace Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Parameter	Method	CAS#	Units	MDL	PRL	LCS/LCSD Lower	LCS/LCSD Upper	LCS/LCSD RPD	MS/MSD Lower	MS/MSD Upper	MS/MSD RPD	Duplicate RPD
General Parameters												
Chloride	EPA 300.0	16887-00-6	mg/L	0.279	1.20	90	110	20	90	110	20	20
Cyanide, Total	SM 4500-CN E	57-12-5	mg/L	0.00853	0.020	90	110	30	80	120	30	30
Cyanide, Free (Calc)	EPA 9014	--	mg/L	--	--	--	--	--	--	--	--	--
Hardness, as CaCO3 (Calc)	SM 2340B	--	ug/L	74.9	3310	--	--	--	--	--	--	--
Nitrogen, ammonia, as N	EPA 350.1	7664-41-7	mg/L	0.0518	0.1	90	110	--	90	110	10	--
Nitrogen, unionized ammonia, as N	EPA 350.1 Calc	--	mg/L	--	--	--	--	--	--	--	--	--
Chromium hexavalent	SM 3500-Cr D	18540-29-9	mg/L	0.00293	0.01	90	110	20	85	115	20	20
Metals												
Aluminum	EPA 200.7	7429-90-5	ug/L	15.5	200	85	115	20	70	130	20	20
Antimony	EPA 200.8	7440-36-0	ug/L	0.0766	0.5	85	115	20	70	130	20	20
Arsenic	EPA 200.8	7440-38-2	ug/L	0.114	0.5	85	115	20	70	130	20	20
Barium	EPA 200.7	7440-39-3	ug/L	0.175	10	85	115	20	70	130	20	20
Beryllium	EPA 200.8	7440-41-7	ug/L	0.0543	0.2	85	115	20	70	130	20	20
Boron	EPA 200.8	7440-42-8	ug/L	2.91	10	85	115	20	70	130	20	20
Cadmium	EPA 200.8	7440-43-9	ug/L	0.0271	0.08	85	115	20	70	130	20	20
Chromium	EPA 200.8	7440-47-3	ug/L	0.161	0.5	85	115	20	70	130	20	20
Cobalt	EPA 200.8	7440-48-4	ug/L	0.0854	0.5	85	115	20	70	130	20	20
Copper	EPA 200.7	7440-50-8	ug/L	1.20	10	85	115	20	70	130	20	20
Lead	EPA 200.8	7439-92-1	ug/L	0.0392	0.1	85	115	20	70	130	20	20
Manganese	EPA 200.7	7439-96-5	ug/L	0.216	5	85	115	20	70	130	20	20
Nickel	EPA 200.7	7440-02-0	ug/L	1.07	20	85	115	20	70	130	20	20
Selenium	EPA 200.8	7782-49-2	ug/L	0.137	0.5	85	115	20	70	130	20	20
Silver	EPA 200.7	7440-22-4	ug/L	0.380	10	85	115	20	70	130	20	20
Thallium	EPA 200.8	7440-28-0	ug/L	0.0264	0.1	85	115	20	70	130	20	20
Tin	EPA 200.7	7440-31-5	ug/L	3.23	75	85	115	20	70	130	20	20
Uranium	EPA 200.8	7440-62-2	ug/L	0.267	1	85	115	20	70	130	20	20
Vanadium	EPA 200.8	7440-61-1	ug/L	0.0968	0.5	85	115	20	70	130	20	20
Zinc	EPA 200.7	7440-66-6	ug/L	2.53	20	85	115	20	70	130	20	20
SVOCs												
Acenaphthene	EPA 8270D	83-32-9	ug/L	2.45	10	48	125	20	70	130	30	30
Anthracene	EPA 8270D	120-12-7	ug/L	2.83	10	61	125	20	70	130	30	30
Benzo(a)pyrene	EPA 8270D	50-32-8	ug/L	2.77	10	75	125	20	70	130	30	30
Benzoic acid *	EPA 8270D	65-85-0	ug/L	5.00	10	30	125	20	70	130	30	30
4-Bromophenylphenyl ether	EPA 8270D	101-55-3	ug/L	2.89	10	75	125	20	70	130	30	30

Table 3a
Pace Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Butylbenzylphthalate	EPA 8270D	85-68-7	ug/L	2.89	10	54	125	20	70	130	30	30
bis(2-Chloroethyl) ether	EPA 8270D	111-44-4	ug/L	2.48	10	46	125	20	70	130	30	30
2-Chlorophenol	EPA 8270D	95-57-8	ug/L	2.38	10	37	125	20	70	130	30	30
3,3'-Dichlorobenzidine	EPA 8270D	91-94-1	ug/L	2.84	50	47	125	20	70	130	30	30
2,4-Dichlorophenol	EPA 8270D	120-83-2	ug/L	2.42	10	52	125	20	70	130	30	30
Diethylphthalate	EPA 8270D	84-66-2	ug/L	2.77	10	56	125	20	70	130	30	30
2,4-Dimethylphenol	EPA 8270D	105-67-9	ug/L	2.06	10	53	125	20	70	130	30	30
Dimethylphthalate	EPA 8270D	131-11-3	ug/L	2.63	10	56	125	20	70	130	30	30
Di-n-butylphthalate	EPA 8270D	84-74-2	ug/L	2.94	10	58	125	20	70	130	30	30
2,4-Dinitrophenol	EPA 8270D	51-28-5	ug/L	5.00	10	48	125	20	70	130	30	30
Di-n-octylphthalate	EPA 8270D	117-84-0	ug/L	4.90	10	53	125	20	70	130	30	30
bis(2-Ethylhexyl)phthalate	EPA 8270D	117-81-7	ug/L	2.96	10	54	125	20	70	130	30	30
Fluoranthene	EPA 8270D	206-44-0	ug/L	2.80	10	58	125	20	70	130	30	30
Fluorene	EPA 8270D	86-73-7	ug/L	2.58	10	52	125	20	52	125	20	30
Hexachlorobenzene	EPA 8270D	118-74-1	ug/L	3.05	10	75	125	20	70	130	30	30
Hexachlorocyclopentadiene	EPA 8270D	77-47-4	ug/L	5.00	10	30	125	20	70	130	30	30
Hexachloroethane	EPA 8270D	67-72-1	ug/L	2.16	10	33	125	20	70	130	30	30
Isophorone	EPA 8270D	78-59-1	ug/L	2.68	10	69	125	20	70	130	30	30
2-Methylnaphthalene	EPA 8270D	91-57-6	ug/L	2.40	10	44	125	20	70	130	30	30
2-Methylphenol(o-Cresol)	EPA 8270D	95-48-7	ug/L	1.93	10	35	125	20	70	130	30	30
3&4-Methylphenol(m&p Cresol)	EPA 8270D	108-39-4 106-44-5	ug/L	1.92	10	36	125	20	70	130	30	30
N-Nitrosodiphenylamine	EPA 8270D	86-30-6	ug/L	2.71	10	56	125	20	70	130	30	30
Pentachlorophenol	EPA 8270D	87-86-5	ug/L	1.32	20	33	125	20	70	130	30	30
Phenanthrene	EPA 8270D	85-01-8	ug/L	2.71	10	60	125	20	70	130	30	30
Phenol	EPA 8270D	108-95-2	ug/L	5.00	10	30	125	20	70	130	30	30
Pyrene	EPA 8270D	129-00-0	ug/L	2.85	10	52	125	20	70	130	30	30
2,4,6-Trichlorophenol	EPA 8270D	88-06-2	ug/L	2.49	10	72	125	20	70	130	30	30
1,4-Dioxane	EPA 8270C SIM	123-91-1	ug/L	0.0737	0.25	69	125	20	70	130	30	30
PCBs												
PCB-1016 (Aroclor 1016)	EPA 8082	12674-11-2	ug/L	0.0419	0.1	47	125	20	30	150	30	30
PCB-1221 (Aroclor 1221)	EPA 8082	11104-28-2	ug/L	0.043	0.1	70	130	20	70	130	30	30
PCB-1232 (Aroclor 1232)	EPA 8082	11141-16-5	ug/L	0.0365	0.1	70	130	20	70	130	30	30
PCB-1242 (Aroclor 1242)	EPA 8082	53469-21-9	ug/L	0.0375	0.1	70	130	20	70	130	30	30
PCB-1248 (Aroclor 1248)	EPA 8082	12672-29-6	ug/L	0.0405	0.1	70	130	20	70	130	30	30
PCB-1254 (Aroclor 1254)	EPA 8082	11097-69-1	ug/L	0.0422	0.1	70	130	20	70	130	30	30
PCB-1260 (Aroclor 1260)	EPA 8082	11096-82-5	ug/L	0.0355	0.1	54	125	20	45	125	30	30

Table 3a
Pace Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

PCB-1262 (Aroclor 1262)	EPA 8082	37324-23-5	ug/L	0.0365	0.1	70	130	20	70	130	30	30
PCB-1268 (Aroclor 1268)	EPA 8082	11100-14-4	ug/L	0.0457	0.1	70	130	20	70	130	30	30
Herbicides MDA List II												
2,4-D	EPA 8151	94-75-7	ug/L	0.5	0.068	44.6	158	20	64.6	148	20	20
2,4-DB	EPA 8151	94-82-6	ug/L	0.5	0.035	64.7	136	20	66.7	143	20	20
2,4,5-T	EPA 8151	93-76-5	ug/L	0.5	0.042	54.1	129	20	63.4	133	20	20
2,4,5-TP (Silvex)	EPA 8151	93-72-1	ug/L	0.5	0.04	55.3	147	20	63	145	20	20
Bentazon	EPA 8151	25057-89-0	ug/L	0.5	0.029	35.5	160	20	52.5	139	20	20
Dicamba	EPA 8151	1918-00-9	ug/L	0.5	0.032	45.2	150	20	55.4	143	20	20
MCPA	EPA 8151	94-74-6	ug/L	0.3	0.048	33.6	149	20	33.5	143	20	20
Picloram	EPA 8151	2018-02-1	ug/L	0.5	0.053	32.6	139	20	47.9	113	20	20
Triclopyr	EPA 8151	55335-06-3	ug/L	0.5	0.058	56.8	143	20	65.1	141	20	20
Pesticides MDA List 1												
Acetochlor	EPA 8270D	34256-82-1	ug/L	0.037	0.5	67.5	120	20	67.3	128	20	20
Alachlor	EPA 8270D	15972-60-8	ug/L	0.038	0.5	71.7	120	20	58.2	150	20	20
Atrazine	EPA 8270D	1912-24-9	ug/L	0.032	0.5	72.8	113	20	70.1	120	20	20
Chlorpyrifos	EPA 8270D	2921-88-2	ug/L	0.043	0.5	65.3	119	20	73.3	118	20	20
Cyanazine	EPA 8270D	21725-46-2	ug/L	0.072	0.2	49.5	140	20	60.6	140	20	20
Desethylatrazine	EPA 8270D	6190-65-4	ug/L	0.014	0.5	66.9	116	20	69.7	122	20	20
Deisopropylatrazine	EPA 8270D	1007-28-9	ug/L	0.041	0.5	44.3	110	20	48	121	20	20
Dimethenamid	EPA 8270D	87674-68-8	ug/L	0.018	0.5	63.8	116	20	63.7	123	20	20
EPTC	EPA 8270D	759-94-4	ug/L	0.048	0.5	41.7	102	20	58	109	20	20
Ethalfluralin	EPA 8270D	55283-68-6	ug/L	0.1	0.5	41	127	20	59.3	129	20	20
Fonofos	EPA 8270D	944-22-9	ug/L	0.025	0.5	59.7	118	20	73.5	108	20	20
Metolachlor	EPA 8270D	87392-12-9	ug/L	0.022	0.5	71.7	122	20	40.9	156	20	20
Metribuzin	EPA 8270D	21087-64-9	ug/L	0.026	0.5	66.6	128	20	70.9	136	20	20
Pendimethalin	EPA 8270D	40487-42-1	ug/L	0.03	0.5	55.5	137	20	55.4	155	20	20
Phorate	EPA 8270D	298-02-2	ug/L	0.046	0.3	41.2	114	20	60.2	108	20	20
Prometon	EPA 8270D	1610-18-0	ug/L	0.062	0.5	66.3	120	20	74.7	124	20	20
Propachlor	EPA 8270D	1918-16-7	ug/L	0.017	0.5	65.8	119	20	72.3	115	20	20
Propazine	EPA 8270D	139-40-2	ug/L	0.051	0.5	72	122	20	73.7	124	20	20
Simazine	EPA 8270D	122-34-9	ug/L	0.037	0.5	72.8	113	20	74.8	114	20	20
Terbufos	EPA 8270D	13071-79-9	ug/L	0.025	0.2	38.6	115	20	56.1	114	20	20
Triallate	EPA 8270D	2303-17-5	ug/L	0.047	0.5	51.4	116	20	65.5	107	20	20
Trifluralin	EPA 8270D	1582-09-8	ug/L	0.013	0.5	46.1	134	20	58	149	20	20
Radiochemical												
Gross Alpha (radiation)	EPA 900.0	12587-46-1	pCi/L	3	--	--	--	--	--	--	--	--

Table 3a
Pace Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Gross Beta (radiation)	EPA 900.0	12587-47-2	pCi/L	3	--	--	--	--	--	--	--	--

* Nonstandard Analyte

Table 3b
Pace Solids Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Parameter	Method	CAS#	Units	MDL	PRL	LCS/LCSD Lower	LCS/LCSD Upper	LCS/LCSD RPD	MS/MSD Lower	MS/MSD Upper	MS/MSD RPD	Duplicate RPD
General Parameters												
Total Organic Carbon	EPA 9060	7440-44-0	mg/kg	62.1	300	70	130	--	70	130	--	25
Grain Size Distribution	ASTM D422	--	--	--	--	--	--	--	--	--	--	--
RCRA Metals												
Arsenic	EPA 6020A	7440-38-2	mg/kg	0.176	0.50	80	120	20	75	125	20	20
Barium	EPA 6010C	7440-39-3	mg/kg	0.0416	0.50	80	120	20	75	125	20	20
Cadmium	EPA 6020A	7440-43-9	mg/kg	0.0265	0.08	80	120	20	75	125	20	20
Chromium	EPA 6020	7440-47-3	mg/kg	0.124	0.50	80	120	20	75	125	20	20
Lead	EPA 6020A	7439-92-1	mg/kg	0.0573	0.20	80	120	20	75	125	20	20
Mercury	EPA 7471	7439-97-6	mg/kg	0.00804	0.02	80	120	20	75	125	20	20
Selenium	EPA 6020A	7782-49-2	mg/kg	0.145	0.50	80	120	20	75	125	20	20
Silver	EPA 6010C	7440-22-4	mg/kg	0.0363	0.50	80	120	20	75	125	20	20
VOCs												
Acetone	EPA 8260B	67-64-1	ug/kg	311	1000	65	125	20	54	150	30	30
Allyl chloride	EPA 8260B	107-05-1	ug/kg	42	200	52	125	20	53	135	30	30
Benzene	EPA 8260B	71-43-2	ug/kg	3	20	61	125	20	65	135	30	30
Bromobenzene	EPA 8260B	108-86-1	ug/kg	3	50	64	125	20	71	141	30	30
Bromochloromethane	EPA 8260B	74-97-5	ug/kg	17	50	65	125	20	62	145	30	30
Bromodichloromethane	EPA 8260B	75-27-4	ug/kg	17	50	57	125	20	59	148	30	30
Bromoform	EPA 8260B	75-25-2	ug/kg	76	200	57	125	20	57	145	30	30
Bromomethane	EPA 8260B	74-83-9	ug/kg	59	500	60	125	20	51	129	30	30
2-Butanone (MEK)	EPA 8260B	78-93-3	ug/kg	27	250	48	125	20	51	150	30	30
n-Butylbenzene	EPA 8260B	104-51-8	ug/kg	24	50	59	125	20	63	150	30	30
sec-Butylbenzene	EPA 8260B	135-98-8	ug/kg	10	50	62	125	20	66	150	30	30
tert-Butylbenzene	EPA 8260B	98-06-6	ug/kg	10	50	64	125	20	71	148	30	30
Carbon tetrachloride	EPA 8260B	56-23-5	ug/kg	24	50	58	125	20	55	144	30	30
Chlorobenzene	EPA 8260B	108-90-7	ug/kg	3	50	66	125	20	70	142	30	30
Chloroethane	EPA 8260B	75-00-3	ug/kg	26	500	62	125	20	61	135	30	30
Chloroform	EPA 8260B	67-66-3	ug/kg	25	50	59	125	20	58	135	30	30
Chloromethane	EPA 8260B	74-87-3	ug/kg	12	200	50	125	20	37	125	30	30
2-Chlorotoluene	EPA 8260B	95-49-8	ug/kg	2	50	62	125	20	66	144	30	30
4-Chlorotoluene	EPA 8260B	106-43-4	ug/kg	3	50	63	125	20	66	140	30	30
1,2-Dibromo-3-chloropropane	EPA 8260B	96-12-8	ug/kg	174	500	54	125	20	61	150	30	30
Dibromochloromethane	EPA 8260B	124-48-1	ug/kg	6	200	60	125	20	65	141	30	30
1,2-Dibromoethane (EDB)	EPA 8260B	106-93-4	ug/kg	5	50	64	125	20	67	147	30	30
Dibromomethane	EPA 8260B	74-95-3	ug/kg	9	50	69	125	20	72	150	30	30
1,2-Dichlorobenzene	EPA 8260B	95-50-1	ug/kg	2	50	63	125	20	70	142	30	30
1,3-Dichlorobenzene	EPA 8260B	541-73-1	ug/kg	2	50	64	125	20	71	142	30	30
1,4-Dichlorobenzene	EPA 8260B	106-46-7	ug/kg	3	50	63	125	20	68	142	30	30

Table 3b
Pace Solids Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Dichlorodifluoromethane	EPA 8260B	75-71-8	ug/kg	16	200	38	125	20	30	125	30	30
1,1-Dichloroethane	EPA 8260B	75-34-3	ug/kg	6	50	63	125	20	57	140	30	30
1,2-Dichloroethane	EPA 8260B	107-06-2	ug/kg	6	50	57	125	20	58	132	30	30
1,1-Dichloroethene	EPA 8260B	75-35-4	ug/kg	15	50	59	125	20	59	139	30	30
cis-1,2-Dichloroethene	EPA 8260B	156-59-2	ug/kg	8	50	61	125	20	60	138	30	30
trans-1,2-Dichloroethene	EPA 8260B	156-60-5	ug/kg	23	50	64	125	20	55	141	30	30
Dichlorofluoromethane	EPA 8260B	75-43-4	ug/kg	69	500	67	125	20	62	148	30	30
1,2-Dichloropropane	EPA 8260B	78-87-5	ug/kg	9	50	67	125	20	64	144	30	30
1,3-Dichloropropane	EPA 8260B	142-28-9	ug/kg	7	50	64	125	20	68	140	30	30
2,2-Dichloropropane	EPA 8260B	594-20-7	ug/kg	6	200	37	126	20	34	150	30	30
1,1-Dichloropropene	EPA 8260B	563-58-6	ug/kg	23	50	64	125	20	61	142	30	30
cis-1,3-Dichloropropene	EPA 8260B	10061-01-5	ug/kg	7	50	61	125	20	62	142	30	30
trans-1,3-Dichloropropene	EPA 8260B	10061-02-6	ug/kg	7	50/200	56	125	20	57	147	30	30
Diethyl ether (Ethyl ether)	EPA 8260B	60-29-7	ug/kg	31	200	60	125	20	62	135	30	30
Ethylbenzene	EPA 8260B	100-41-4	ug/kg	3	50	62	125	20	72	138	30	30
Hexachloro-1,3-butadiene	EPA 8260B	87-68-3	ug/kg	12	250	56	125	20	38	150	30	30
Isopropylbenzene (Cumene)	EPA 8260B	98-82-8	ug/kg	2	50	65	125	20	75	148	30	30
p-Isopropyltoluene	EPA 8260B	99-87-6	ug/kg	15	50	63	125	20	72	150	30	30
Methylene Chloride	EPA 8260B	75-09-2	ug/kg	94	200	64	125	20	58	135	30	30
4-Methyl-2-pentanone (MIBK)	EPA 8260B	108-10-1	ug/kg	10	250	52	135	20	63	150	30	30
Methyl-tert-butyl ether	EPA 8260B	1634-04-4	ug/kg	6	50	59	125	20	63	139	30	30
Naphthalene	EPA 8260B	91-20-3	ug/kg	47	200	53	125	20	63	150	30	30
n-Propylbenzene	EPA 8260B	103-65-1	ug/kg	3	50	61	125	20	70	146	30	30
Styrene	EPA 8260B	100-42-5	ug/kg	2	50	66	125	20	72	146	30	30
1,1,1,2-Tetrachloroethane	EPA 8260B	630-20-6	ug/kg	16	50	59	125	20	64	146	30	30
1,1,2,2-Tetrachloroethane	EPA 8260B	79-34-5	ug/kg	9	50	58	125	20	36	150	30	30
Tetrachloroethene	EPA 8260B	127-18-4	ug/kg	18	50	67	125	20	70	150	30	30
Tetrahydrofuran	EPA 8260B	109-99-9	ug/kg	73	2000	62	125	20	62	150	30	30
Toluene	EPA 8260B	108-88-3	ug/kg	12	50	61	125	20	65	142	30	30
1,2,3-Trichlorobenzene	EPA 8260B	87-61-6	ug/kg	8	50	55	126	20	69	150	30	30
1,2,4-Trichlorobenzene	EPA 8260B	120-82-1	ug/kg	11	50	62	125	20	71	149	30	30
1,1,1-Trichloroethane	EPA 8260B	71-55-6	ug/kg	23	50	59	125	20	56	148	30	30
1,1,2-Trichloroethane	EPA 8260B	79-00-5	ug/kg	6	50	64	125	20	67	148	30	30
Trichloroethene	EPA 8260B	79-01-6	ug/kg	8	50	67	125	20	62	150	30	30
Trichlorofluoromethane	EPA 8260B	75-69-4	ug/kg	87	200	65	125	20	51	150	30	30
1,2,3-Trichloropropane	EPA 8260B	96-18-4	ug/kg	13	200	62	125	20	64	150	30	30
1,1,2-Trichlorotrifluoroethane	EPA 8260B	76-13-1	ug/kg	58	200	65	125	20	60	142	30	30
1,2,4-Trimethylbenzene	EPA 8260B	95-63-6	ug/kg	10	50	59	125	20	67	149	30	30
1,3,5-Trimethylbenzene	EPA 8260B	108-67-8	ug/kg	8	50	59	125	20	71	146	30	30
Vinyl chloride	EPA 8260B	75-01-4	ug/kg	10	20	57	125	20	45	132	30	30
Xylene (Total)	EPA 8260B	1330-20-7	ug/kg	12	150	62	125	20	75	140	30	30

Table 3b
Pace Solids Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

WIDRO												
WDRO C10-C28	WI MOD DRO	--	mg/kg	2.60	10	70	120	20	70	120	20	20
WIGRO												
Gasoline Range Organics	WI MOD GRO	--	mg/kg	0.754	10	80	120	20	80	120	20	20
PAHs												
Acenaphthene	EPA 8270 SIM	83-32-9	ug/kg	0.409	10	52	125	20	30	125	30	30
Acenaphthylene	EPA 8270 SIM	208-96-8	ug/kg	0.495	10	50	125	20	30	133	30	30
Anthracene	EPA 8270 SIM	120-12-7	ug/kg	0.468	10	65	125	20	30	150	30	30
Benzo(a)anthracene	EPA 8270 SIM	56-55-3	ug/kg	1.08	10	60	125	20	30	150	30	30
Benzo(a)pyrene	EPA 8270 SIM	50-32-8	ug/kg	0.687	10	69	125	20	30	150	30	30
Benzo(b)fluoranthene	EPA 8270 SIM	205-99-2	ug/kg	0.373	10	61	125	20	30	150	30	30
Benzo(e)pyrene	EPA 8270 SIM	192-97-2	ug/kg	0.719	10	71	125	20	30	150	30	30
Benzo(g,h,i)perylene	EPA 8270 SIM	191-24-2	ug/kg	0.633	10	60	125	20	30	150	30	30
Benzo(k)fluoranthene	EPA 8270 SIM	207-08-9	ug/kg	0.845	10	67	125	20	30	150	30	30
Chrysene	EPA 8270 SIM	218-01-9	ug/kg	1.36	10	67	125	20	30	150	30	30
Dibenz(a,h)anthracene	EPA 8270 SIM	53-70-3	ug/kg	0.461	10	63	125	20	30	131	30	30
Fluoranthene	EPA 8270 SIM	206-44-0	ug/kg	0.428	10	75	125	20	30	150	30	30
Fluorene	EPA 8270 SIM	96-73-7	ug/kg	0.313	10	54	125	20	30	147	30	30
Indeno(1,2,3-cd)pyrene	EPA 8270 SIM	193-39-5	ug/kg	0.670	10	63	125	20	30	150	30	30
Naphthalene	EPA 8270 SIM	91-20-3	ug/kg	0.771	10	49	125	20	30	131	30	30
Phenanthrene	EPA 8270 SIM	85-01-8	ug/kg	1.92	10	65	125	20	30	150	30	30
Pyrene	EPA 8270 SIM	129-00-0	ug/kg	1.53	10	64	125	20	30	150	30	30

Table 3c
Pace Soil Gas Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Parameter	Method	CAS #	Alternate Name	MDL (ppbv)	PRL (ppbv)	MW	MDL (ug/m3)	PRL (ug/m3)	LCS Lower	LCS Upper	DUP RPD
1,1,1-Trichloroethane	EPA TO15	71-55-6	--	0.0558	0.2	133.4047	0.309	1.11	70	135	25
1,1,2,2-Tetrachloroethane	EPA TO15	79-34-5	--	0.0419	0.1	167.8498	0.292	0.698	70	146	25
1,1,2-Trichloroethane	EPA TO15	79-00-5	--	0.0452	0.1	133.4047	0.250	0.555	70	135	25
1,1,2-Trichlorotrifluoroethane	EPA TO15	76-13-1	Freon 113	0.0724	0.2	187.3762	0.564	1.56	63	139	25
1,1-Dichloroethane	EPA TO15	75-34-3	--	0.0546	0.2	98.9596	0.225	0.823	70	134	25
1,1-Dichloroethene	EPA TO15	75-35-4	--	0.0679	0.2	96.9438	0.274	0.806	70	137	25
1,2,4-Trichlorobenzene	EPA TO15	120-82-1	--	0.493	1	181.4487	3.72	7.54	60	133	25
1,2,4-Trimethylbenzene	EPA TO15	95-63-6	--	0.0904	0.2	120.1938	0.452	0.999	70	137	25
1,2-Dibromoethane (EDB)	EPA TO15	106-93-4	--	0.0468	0.1	187.8616	0.366	0.781	70	140	25
1,2-Dichlorobenzene	EPA TO15	95-50-1	--	0.0814	0.2	147.0036	0.498	1.22	70	137	25
1,2-Dichloroethane	EPA TO15	107-06-2	--	0.0365	0.1	98.9596	0.150	0.411	70	136	25
1,2-Dichloropropane	EPA TO15	78-87-5	--	0.0490	0.2	112.9864	0.230	0.939	70	136	25
1,3,5-Trimethylbenzene	EPA TO15	108-67-8	--	0.0798	0.2	120.1938	0.399	0.999	70	133	25
1,3-Butadiene	EPA TO15	106-99-0	--	0.0567	0.2	54.0914	0.128	0.450	64	141	25
1,3-Dichlorobenzene	EPA TO15	541-73-1	--	0.0951	0.2	147.0036	0.581	1.22	70	137	25
1,4-Dichlorobenzene	EPA TO15	106-46-7	--	0.164	0.5	147.0036	1.00	3.06	70	134	25
2-Butanone (MEK)	EPA TO15	78-93-3	Methyl Ethyl Ketone	0.123	1	72.1057	0.369	3.00	65	143	25
2-Hexanone	EPA TO15	591-78-6	Methyl Butyl Ketone	0.179	1	100.1589	0.745	4.16	60	148	25
2-Propanol	EPA TO15	67-63-0	isopropyl alcohol	0.279	1	60.1	0.697	2.50	65	135	25
4-Ethyltoluene	EPA TO15	622-96-8	--	0.114	0.5	120.1938	0.570	2.50	70	132	25
4-Methyl-2-pentanone (MIBK)	EPA TO15	108-10-1	Methy Isobutyl Ketone	0.124	1	100.1602	0.518	4.16	70	135	25
Acetone	EPA TO15	67-64-1	--	0.499	1	58.0798	1.21	2.41	59	132	25
Benzene	EPA TO15	71-43-2	--	0.0471	0.1	78.1134	0.153	0.325	70	134	25
Benzyl chloride	EPA TO15	100-44-7	--	0.228	0.5	126.58	1.20	2.63	56	150	25
Bromodichloromethane	EPA TO15	75-27-4	--	0.0537	0.2	163.8289	0.366	1.36	70	142	25
Bromoform	EPA TO15	75-25-2	--	0.135	0.5	252.7309	1.42	5.25	69	150	25
Bromomethane	EPA TO15	74-83-9	--	0.0575	0.2	94.9387	0.227	0.789	61	141	25
Carbon disulfide	EPA TO15	75-15-0	--	0.0692	0.2	76.131	0.219	0.633	66	134	25
Carbon tetrachloride	EPA TO15	56-23-5	--	0.0671	0.2	153.823	0.429	1.28	60	145	25
Chlorobenzene	EPA TO15	108-90-7	--	0.0588	0.2	112.5585	0.275	0.936	70	130	25
Chloroethane	EPA TO15	75-00-3	--	0.0969	0.2	64.5145	0.260	0.536	65	143	25
Chloroform	EPA TO15	67-66-3	--	0.0395	0.1	119.3779	0.196	0.496	70	132	25
Chloromethane	EPA TO15	74-87-3	--	0.0742	0.2	50.4877	0.156	0.420	58	140	25
cis-1,2-Dichloroethene	EPA TO15	156-59-2	--	0.0543	0.2	96.9438	0.219	0.806	70	136	25
cis-1,3-Dichloropropene	EPA TO15	10061-01-5	--	0.0659	0.2	110.9706	0.304	0.923	70	136	25
Cyclohexane	EPA TO15	110-82-7	--	0.101	0.5	84.1608	0.353	1.75	70	133	25
Dibromochloromethane	EPA TO15	124-48-1	--	0.0830	0.2	208.2799	0.719	1.73	68	149	25
Dichlorodifluoromethane	EPA TO15	75-71-8	--	0.0584	0.2	120.9138	0.293	1.01	69	130	25
Dichlorotetrafluoroethane	EPA TO15	76-14-2	--	0.0615	0.2	170.9216	0.437	1.42	68	130	25
Ethanol	EPA TO15	64-17-5	--	0.424	1	46.07	0.812	1.92	65	146	25
Ethyl acetate	EPA TO15	141-78-6	--	0.0518	0.2	88.106	0.190	0.733	68	136	25

Table 3c
Pace Soil Gas Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Ethylbenzene	EPA TO15	100-41-4	--	0.0690	0.2	106.167	0.305	0.883	70	133	25
Hexachloro-1,3-butadiene	EPA TO15	87-68-3	--	0.181	0.5	260.762	1.967	5.42	59	140	25
m&p-Xylene	EPA TO15	106-42-3	--	0.158	0.4	106.167	0.699	1.77	70	133	25
Methylene Chloride	EPA TO15	75-0902	--	0.267	1	84.9328	0.944	3.53	67	132	25
Methyl-tert-butyl ether	EPA TO15	1634-04-4	--	0.181	1	88.1492	0.663	3.66	70	132	25
Naphthalene	EPA TO15	91-20-3	--	0.248	0.5	128.1732	1.32	2.66	55	136	25
n-Heptane	EPA TO15	142-82-5	heptane	0.0913	0.2	100.2034	0.380	0.833	64	136	25
n-Hexane	EPA TO15	110-54-3	--	0.0867	0.2	86.1766	0.311	0.716	70	130	25
o-Xylene	EPA TO15	95-47-6	--	0.0780	0.2	106.167	0.344	0.883	70	132	25
Propylene	EPA TO15	115-07-1	--	0.0816	0.2	42.0804	0.143	0.350	37	150	25
Styrene	EPA TO15	100-42-5	--	0.0794	0.2	104.1512	0.344	0.866	70	139	25
Tetrachloroethene	EPA TO15	127-18-4	--	0.0455	0.1	165.834	0.314	0.689	70	133	25
Tetrahydrofuran	EPA TO15	109-99-9	--	0.0870	0.2	72.1066	0.261	0.600	62	141	25
Toluene	EPA TO15	108-88-3	--	0.0916	0.2	92.1402	0.351	0.766	70	130	25
trans-1,2-Dichloroethene	EPA TO15	156-60-5	--	0.0706	0.2	96.9438	0.285	0.806	70	132	25
trans-1,3-Dichloropropene	EPA TO15	10061-02-6	--	0.0953	0.2	110.9706	0.440	0.923	70	135	25
Trichloroethene	EPA TO15	79-01-6	--	0.0470	0.1	131.3889	0.257	0.546	70	135	25
Trichlorofluoromethane	EPA TO15	75-69-4	--	0.0641	0.2	137.3684	0.366	1.14	59	140	25
Vinyl acetate	EPA TO15	108-05-4	--	0.0754	0.2	86.0902	0.270	0.716	57	150	25
Vinyl chloride	EPA TO15	75-01-4	--	0.0485	0.1	62.4987	0.126	0.260	70	141	25
Methane (reported as %)	EPA 3C	74-82-8	--	0.94	4	--	--	--	70	130	30

Table 4
MDH Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Parameter	Method	RL (ug/L)	LCS Low	LCS High	LCS RPD	MS Low	MS High	MS RPD
VOCs								
Acetone	EPA 8260B	20.0	70	130	<30%	70	130	<30%
Allyl Chloride	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Benzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Bromobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Bromochloromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Bromodichloromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Bromoform	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Bromomethane	EPA 8260B	2.0	70	130	<30%	70	130	<30%
n-Butylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
sec-Butylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
tert-Butylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Carbon Tetrachloride	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Chlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Chlorodibromomethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Chloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Chloroform	EPA 8260B	1.0	80	120	<30%	70	130	<30%
Chloromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
2-Chlorotoluene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
4-Chlorotoluene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260B	5.0	70	130	<30%	70	130	<30%
1,2-Dibromoethane (EDB)	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Dibromomethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2-Dichlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,3-Dichlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,4-Dichlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Dichlorodifluoromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1-Dichloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2-Dichloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1-Dichloroethene	EPA 8260B	1.0	80	120	<30%	70	130	<30%

Table 4
MDH Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

cis-1,2-Dichloroethene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
trans-1,2-Dichloroethene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Dichlorofluoromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2-Dichloropropane	EPA 8260B	1.0	80	120	<30%	70	130	<30%
1,3-Dichloropropane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
2,2-Dichloropropane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1-Dichloropropene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
cis-1,3-Dichloropropene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
trans-1,3-Dichloropropene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Ethylbenzene	EPA 8260B	1.0	80	120	<30%	70	130	<30%
Ethyl Ether	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Hexachlorobutadiene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Isopropylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
p-Isopropyltoluene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Methylene Chloride	EPA 8260B	2.0	70	130	<30%	70	130	<30%
Methyl Ethyl Ketone (MEK)	EPA 8260B	10.0	70	130	<30%	70	130	<30%
Methyl Isobutyl Ketone (MIBK)	EPA 8260B	5.0	70	130	<30%	70	130	<30%
Methyl tert-Butyl Ether (MTBE)	EPA 8260B	2.0	70	130	<30%	70	130	<30%
Naphthalene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
n-Propylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Styrene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1,1,2-Tetrachloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1,2,2-Tetrachloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Tetrachloroethene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Tetrahydrofuran (THF)	EPA 8260B	10.0	70	130	<30%	70	130	<30%
Toluene	EPA 8260B	1.0	80	120	<30%	70	130	<30%
1,2,3-Trichlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2,4-Trichlorobenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1,1-Trichloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1,2-Trichloroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Trichloroethene (TCE)	EPA 8260B	1.0	70	130	<30%	70	130	<30%

Table 4
MDH Water Analyses
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Trichlorofluoromethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2,3-Trichloropropane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,1,2-Trichlorotrifluoroethane	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,2,4-Trimethylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
1,3,5-Trimethylbenzene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
Vinyl Chloride	EPA 8260B	1.0	80	120	<30%	70	130	<30%
o-Xylene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
p&m-Xylene	EPA 8260B	1.0	70	130	<30%	70	130	<30%
PFAS								
Perfluorobutanic acid	MDH 555	0.05	80	120	--	70	130	<20%
Perfluoropentanoic acid	MDH 555	0.05	80	120	--	70	130	<20%
Perfluorohexanoic acid	MDH 555	0.05	80	120	--	70	130	<20%
Perfluorooctanoic acid	MDH 555	0.05	80	120	--	70	130	<20%
Perfluorobutane sulfonate	MDH 555	0.05	80	120	--	70	130	<20%
Perfluorohexane sulfonate	MDH 555	0.05	80	120	--	70	130	<20%
Perfluorooctane sulfonate	MDH 555	0.05	80	120	--	70	130	<20%

Table 5
Data Quality Objectives
Freeway Landfill QAPP

Problem	Goal of the Study	Information Inputs	Study Boundaries	Decision Rule	Tolerable Limits on Decision Errors	Optimize Design for Obtaining Data
Soil						
Potential closure options involve the need for a significant amount of fill. The characteristics, quality, and suitability for reuse of the existing fill and cover materials is unknown.	Characterize the quality of the cover soils to determine if they are suitable for reuse as part of a remedial design.	Data from previous soil borings will be reviewed. Soil samples will be collected from planned boring locations. Physical and chemical data will be collected. The soil parameters are shown on Table 3b.	Spatial study boundaries include the project site and adjacent properties, as shown on Figures 2a and 2b. Consent to access on third party properties will be required to collect the data. Temporal boundaries do not directly apply to the collection of soil samples; however, the sampling must occur prior to finalizing remedial design.	Decision regarding the suitability of the existing cover soils for use as fill or cover soil as part of the closure design will be based on whether chemical concentrations and physical properties are appropriate for planned use.	Sampling will follow the summary of expected sampling found in Table 2. Decision will be based on analytical results, which will minimize potential for false negatives. False positive decision errors may be addressed with duplicate sampling. Additional sampling may occur if required to better define the suitability of the soils.	The design of the sampling is summarized in Table 2. The basis for the design is MPCA requirements and criteria.
Groundwater						
Previous investigations have shown that groundwater has been impacted by the presence of waste in the Landfill and Dump.	Further characterize the quality of groundwater to evaluate if nearby receptors are being affected by impacted groundwater.	Information that will be evaluated includes: groundwater elevation data from new and existing wells, groundwater concentrations from new and existing wells, the potentially affected receptors and their relevant criteria.	Spatial study boundaries include the project site and adjacent properties, as shown on Figures 2a and 2b. Consent to access on third party properties will be required to collect the data. Temporal influence on groundwater data will be based on physical conditions, including the ongoing pumping of the adjacent quarry and periodic flooding of the adjacent Minnesota River. Data can be collected during any conditions but the current conditions will be noted to assist in data evaluation.	Determination if there is an unacceptable risk to receptors based on the presence of impacted water will be based on state and federal criteria, including surface water criteria, health risk limits, and maximum contaminant levels.	Sampling will follow the summary of expected sampling found in Table 2. Decision will be based on analytical results, which will minimize potential for false negatives. False positive decision errors may be addressed with duplicate sampling. Additional sampling may occur if it is decided there are impacts requiring further characterization.	The design of the sampling is summarized in Table 2. The basis for the design is MPCA requirements and criteria.
Soil Gas						
Previous investigations have shown that waste materials in the Landfill and Dump are generating elevated levels of landfill gases, including methane. The presence of waste materials may also be generating other soil gases that have not been investigated. The waste materials are present adjacent to buildings that may be periodically occupied.	Characterize the soil gas concentrations to determine if the potential for vapor intrusion exists in nearby buildings.	Information that will be evaluated includes the soil gas concentrations and the season during which the samples were collected. Potential occupancy and building conditions from adjacent buildings will also be evaluated.	Spatial study boundaries include the project site and adjacent properties, as shown on Figures 2a and 2b. Consent to access on third party properties will be required to collect the data. Seasonal conditions (e.g., winter vs. non-winter) may affect soil gas concentrations. The season during which samples were collected will be noted. Additional sampling may be required, per MPCA best management practices.	Determination if there is a vapor intrusion risk to potential building occupants will be based on the MPCA Intrusion Screening Values for Volatile Organic Compounds and the explosive limits for methane and other landfill gases.	Sampling will follow the summary of expected sampling found in Table 2. Decision will be based on analytical results, which will minimize potential for false negatives. False positive decision errors may be addressed with duplicate sampling. Additional sampling may occur if it is decided there are impacts requiring further characterization.	The design of the sampling is summarized in Table 2. The basis for the design is MPCA requirements and criteria.

Table 6
Field and Laboratory QA/QC Sample Requirements
Freeway Landfill, Dump and Transfer Station Investigation QAPP

	Parameter	Frequency	Comments
Field	Field Blank	1 collected every 10 samples	A sample of analyte-free water exposed to environmental conditions at the sampling site by transferring from one sample container to another or by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time equivalent necessary to fill a container. Collected instead of an Equipment Blank if disposable/single use sampling equipment is used. Target analytes should not have a reportable concentration above the method reporting limit.
	Equipment Blank	1 collected every 10 samples	A sample of analyte-free water collected when rinsing sampling equipment. It measures the potential for sample cross contamination due to insufficient decontamination of sampling equipment. Collected when reusable sampling equipment is used. Target analytes should not have a reportable concentration above the method reporting limit.
	Trip Blank	Generally when VOC samples are collected, but may be less frequent if conditions are not indicative for the potential of cross contamination (i.e. low PID readings)	Trip blank samples are used when sampling volatile organic compounds (VOC) only. Analyte-free water is used for water samples and methanol (or other applicable sample preservative) is used for soil samples. They are prepared or provided by the laboratory along with the VOC sampling containers prior to a sampling event. Trip blank sample containers are not to be opened in the field and accompany the VOC samples during collection, storage, and transport to the analytical laboratory. The trip blanks should be listed on the chain-of-custody (COC) along with the samples and the analysis required. The purpose of the trip blank sample is to determine the extent of potential contamination introduced during sample transport and handling.
	Field Duplicate	1 collected every 10 samples	Sample collected in duplicate using the same collection methods to verify reproducibility.
Laboratory	Method Blank	1 per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte free media processed simultaneously with and under the same conditions as samples. Used to assess possible sources of laboratory contamination present at concentrations that may impact analytical results. Target analytes should not have a reportable concentration above the method reporting limit.
	Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	1 LCS or 1 LCS/LCSD set per batch of 20 or fewer samples, with every analytical batch or as stated in the method, whichever is more frequent	Analyte-free media spiked with a known concentration of analyte processed with and under the same conditions as samples. Recovery is used to evaluate overall analytical method accuracy independent of sample matrix effects. If analyzed in duplicate, the calculated RPD is used to assess the overall analytical method precision.
	Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	1 MS or 1 MS/MSD set per batch of 20 or fewer samples, where applicable	A sample spiked with a known concentration of analyte processed with and under the same conditions in order to assess the accuracy of a method in a given sample matrix. If analyzed in duplicate, the calculated RPD is used to assess the precision of a method in a given sample matrix.
	Laboratory Duplicate	1 per batch of 20 or fewer samples, where applicable	A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method. It may be a duplicate of a sample or a duplicate of a matrix spike.
	Surrogates	Surrogates are added to each sample for organic analyses (blanks, spiked samples, project samples, QC samples) prior to sample extraction.	Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Recovery is used to evaluate the analytical method efficiency.

Table 7
Sample Containers, Preservation and Holding Times
Freeway Landfill, Dump and Transfer Station Investigation QAPP

Matrix	Parameter	Container	Preservative	Recommended Holding Time
Water	Chloride	50mL glass or plastic	Cool <6°C	28 days
	Chromium hexavalent	Glass jar or vial, unspecified size	Cool <6°C	24 hrs
	Cyanide, Free	250mL glass or plastic	NaOH to pH>12	14 days
	Cyanide, Total	250mL glass or plastic	NaOH to pH>12	48 hours
	Hardness, as CaCO3 (Calc)	Calculation	NA	NA
	Herbicides	1, 1L amber glass bottles	Cool <6°C	7 days extraction/40 days after extraction
	Metals	250mL, plastic	HNO3	180 days
	Nitrogen, ammonia, as N	Glass or plastic, unspecified size	H2SO4	28 days
	PCBs	1, 1L amber glass bottles	Cool <6°C	7 days extraction/40 days after extraction
	Pesticides	1, 1L amber glass bottles	Cool <6°C	7 days extraction/40 days after extraction
	PFAS	Nalgene HDPE 250 ml wide mouth	Cool <6°C	14 days (per MDH SOP)
	Radiochemical	Glass or plastic, unspecified size	HNO3	180 days
	SVOCs	1, 1L amber glass bottles	Cool <6°C	7 days extraction/40 days after extraction
	Unionized ammonia	Calculation	NA	NA
	VOC	3, 40mL VOAs	HCl to <2 pH, Cool <6°C, Minimize headspace	14 days
Soil	Grain Size Distribution	Zipper-style plastic bag or glass jar	None	None
	Mercury	4 oz. jar w/PTFE-lined lid	None	28 days
	Metals	4 oz. jar w/PTFE-lined lid	None	180 days
	PAHs	4 oz. jar w/PTFE-lined lid	Cool ≤ 6 °C	14 days extraction/40 days after extraction
	Total Organic Carbon	4 oz. jar w/PTFE-lined lid	Cool <6°C	28 days
	VOCs	2 Tared 40 mL vial or wide mouth jar – 25 g capacity Encore, or similar approved sample container and storage device (Terracore)	MeOH (1:1 ratio), cool <6°C	14 days
	Wisconsin DRO	4 oz. jar w/PTFE-lined lid, pre-weighed by laboratory	Cool <6°C	10 days
	Wisconsin GRO	Glass jar or vial w/PTFE-lined lid	MeOH (1:1 ratio), cool <6°C	21 days
Gas	Methane	5 L Summa Canister	None	30 days
	VOCs	5 L Summa Canister	None	30 days

Table 8
Action Level Criteria for Comparison
Freeway Landfill, Dump and Transfer Station Investigation QAPP

	Criteria for Comparison
Drinking Water	EPA Maximum Contaminant Levels
	MDH Human Health-Based Water Guidance Table
Surface Water	Minnesota Surface Water 2Bd Chronic 7050 - 360 Hardness
	Minnesota Surface Water 2Bd Final Acute Value 7050 - 360 Hardness
Soils and Solids	Minnesota Soil Leaching Values (SLVs)
	Minnesota Tier 2 Industrial Soil Reference Values (SRVs)
	Minnesota Tier 2 Recreational Soil Reference Values (SRVs)
Soil Gas	Minnesota Intrusion Screening Values (ISVs)

Figures

FREEWAY TRANSFER STATION

FREEWAY LANDFILL

FREEWAY DUMP

Minnesota River

BLOOMINGTON

BURNSVILLE

SAVAGE

Project Areas

Municipal Boundary

N

0 1,000 2,000

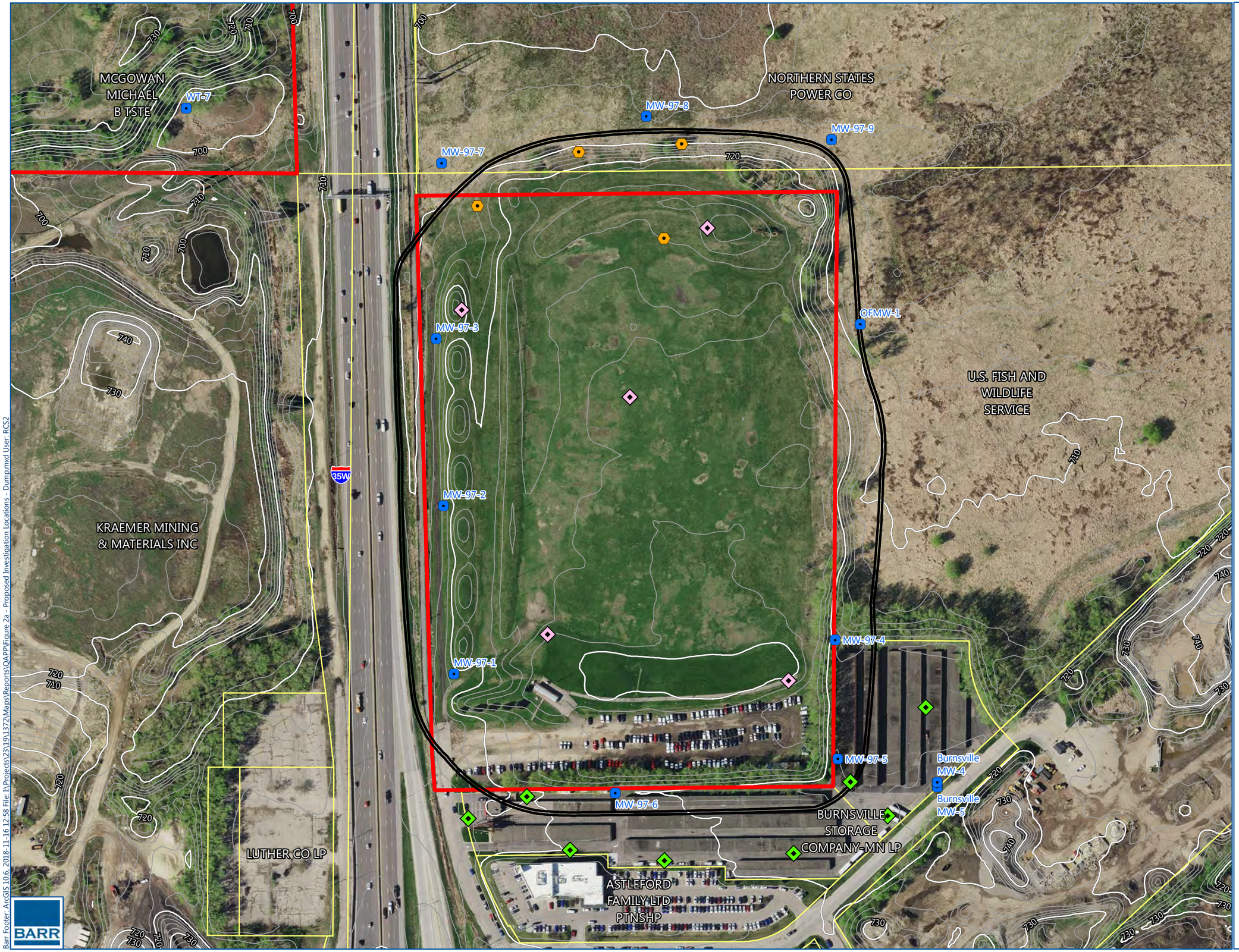
Feet

SITE MAP
Freeway Dump and Landfill
Phase B
Dakota County, Minnesota

FIGURE 1

BARR

Barr Footer: ArcGIS 10.6, 2018-06-07 16:14 File: I:\Projects\23\19\1372\Maps\Reports\Investigation_Report\Fig 1 - Site Overview.mxd User: kac2



Inferred Waste Extent

Project Areas

Parcel Boundary

10-Foot Contour

2-Foot Contour

Existing Well Loaciton

Phase B Proposed Locations

Proposed Shallow Monitoring Well Location

Proposed Cover Soil Evaluation Boring Location

Proposed Waste Extent Evaluation Boring Location

N

0 200 400

Feet

PROPOSED INVESTIGATION LOCATIONS - Freeway Dump

Phase B Work Scope

Dakota County, Minnesota

FIGURE 2a

BARR

Barr Footer: ArcGIS 10.6, 2018-11-16 12:58 File: I:\Projects\23\19\1372\Maps\Reports\QAPP\Figure 2a - Proposed Investigation Locations - Dump.mxd User: RCS2

Inferred Waste Extent
Project Areas
Parcel Boundary
10-Foot Contour
Existing Well Location

Phase B Proposed Locations

Proposed Shallow Monitoring Well Location
Proposed Monitoring Well Nest Location
Proposed Test Excavation Location
Proposed Soil Cover Evaluation Boring

WT-9
WT-10
WT-12B
WT-14
WT-11B
WT-13
WT-6
J-14
J-13
J-1
MW-8 & MW-8D
MW-7
MW-6
MW-3
MW-5
MW-4 & MW-4D
MW-2
MW-1

Hennepin County
Dakota County
Minnesota River
U.S. SALT INC
MCGOWAN MICHAEL B TSTE
KRAEMER MINING & MATERIALS INC
NORTHERN STATES POWER CO
U.S. FISH AND WILDLIFE SERVICE
MCGOWAN MICHAEL B TSTE
35W

N

0 350 700
Feet

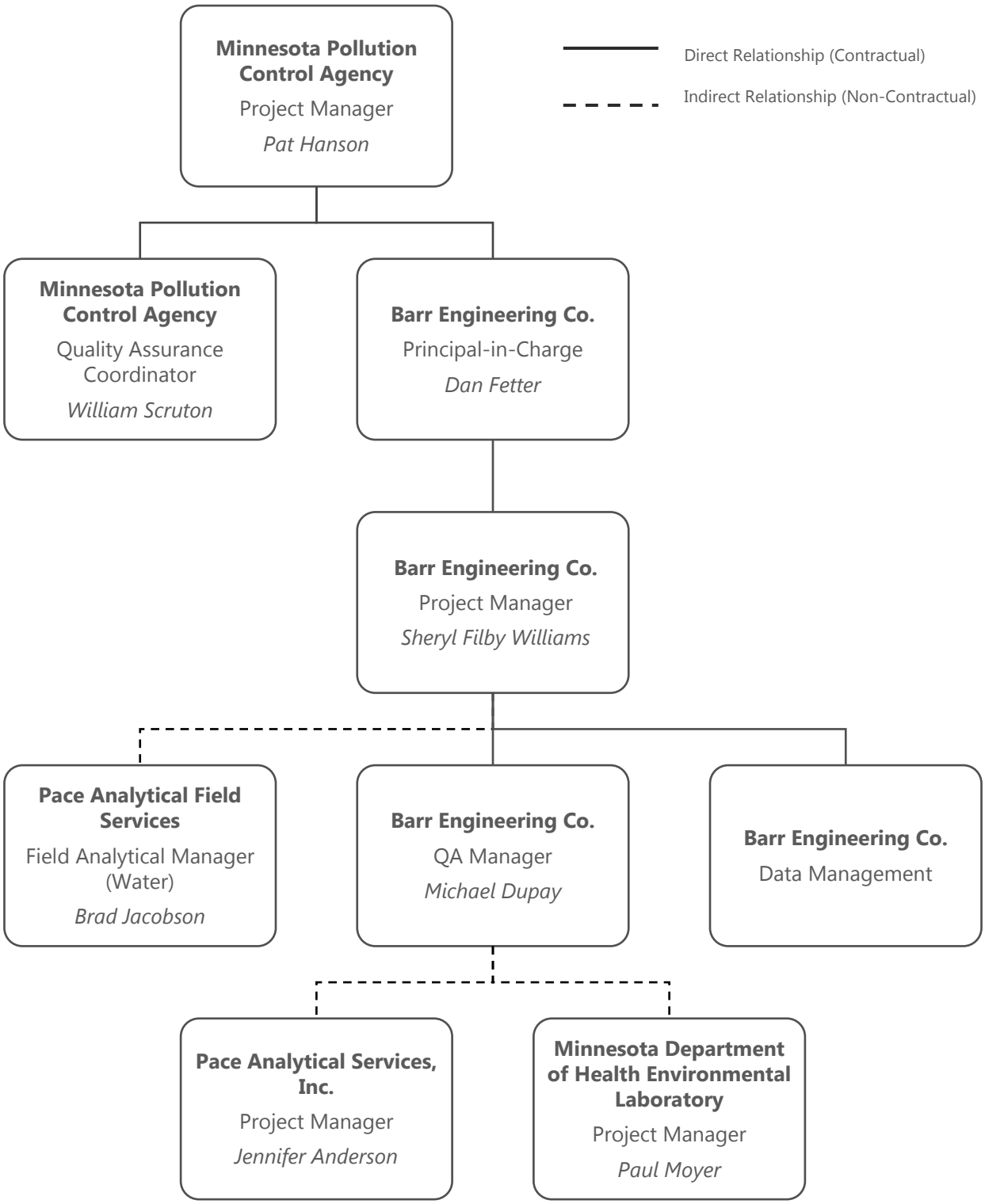
PROPOSED INVESTIGATION LOCATIONS - Freeway Landfill
Phase B Work Scope
Dakota County, Minnesota

FIGURE 2b

BARR

Barr Footer: ArcGIS 10.6, 2018-11-16 13:00 File: I:\Projects\23\19\1372\Maps\Reports\QAPP\Figure 2b - Proposed Investigation Locations - Landfill.mxd User: RCS2

Figure 3
Project Organization Chart



Appendices

Appendix A

Resumes

DANIEL J. FETTER, PE
Vice President, Senior Civil Engineer

BARR

Experience

Dan Fetter has 30 years of experience in the areas of regulatory analysis, site investigation, remedial design, brownfields redevelopment, cost estimating, hazardous waste management, and remedial action coordination. He specializes in addressing legacy environmental issues at contaminated sites and industrial facilities and developing practical, cost-effective environmental solutions for redeveloping contaminated land. His experience includes:

Brownfields redevelopment

- Directing investigation and cleanup activities for the new Mississippi Watershed Management Organization's headquarters located on the east bank of the Mississippi River in Minneapolis. The project integrated an array of Barr services related to site clearing, contamination investigation and remediation planning, assistance with obtaining grant funding, and design of environmentally friendly stormwater management. The new development created a recreational space with integrated stormwater management that provide recreational and educational benefits and will help spur the redevelopment of the Mississippi River corridor.
- Directing investigation and cleanup planning for the new Capitol Region Watershed District headquarters in St. Paul. The redevelopment included reuse of an existing building that will be retrofitted to address vapor intrusion concerns related to past industrial uses of the property, including petroleum and solvent releases. A unique approach was developed with the Minnesota Pollution Control Agency (MPCA) for on-site stormwater management that relied on investigation and modeling to demonstrate that low-level groundwater impacts would not be adversely affected by new stormwater infiltration basins. Site construction and remediation is planned for 2018.
- Directing investigations, cleanup planning, and geotechnical assessments for a new building development at the Minneapolis impound lot, which is the site of the old Irving Avenue Dump, a former state Superfund site. The project included environmental and geotechnical assessments, development of cleanup plans to address vapor intrusion risks (methane and hydrogen sulfide), and treatment of Resource Conservation and Recovery Act (RCRA) hazardous wastes that would be encountered during redevelopment excavations. Site construction and remediation is planned for 2018.
- Assisting a group of public stakeholders on a series of projects to position for redevelopment of an urban neighborhood with a history of heavy industrial use along Bassett Creek in the City of Minneapolis. The stakeholders include the City of Minneapolis, Bassett Creek Watershed Management Commission, Hennepin County, MPCA, and U.S. Environmental Protection Agency (EPA). The work included summarizing and synthesizing available environmental information across a 284-acre redevelopment area and publishing the information on an interactive GIS-based mapping website to help assist with due diligence and regulatory coordination around emerging development interests. Follow-up work is occurring at specific parcels as development advances, including planning and investigation for site redevelopment, assessing the feasibility and cost for various new land uses, and environmental

investigations and planning for an upcoming streambank stabilization project where Bassett Creek passes through several contaminated sites.

- Directing environmental evaluations for the City of South St. Paul at industrial and brownfield properties that are associated with the planned levee improvements along the western bank of the Mississippi River. The levee project and environmental evaluations are also being coordinated with long range redevelopment planning for this aging industrial neighborhood.
- Directing investigation and cleanup planning for the City of St. Paul on three brownfield redevelopment sites along the Central Corridor, a development area around St. Paul's first light-rail transit (LRT) route. Work was funded under the city's U.S. EPA brownfield redevelopment grant, and it included Phase I and Phase II assessments, preparation of response action plans, assistance with U.S. EPA grant administration procedures, and coordination with the City's development partners.
- Assisting the city of New Brighton with one of the largest and most complex brownfield redevelopments in the state. The work includes conducting Phase Is, Phase IIs, and preparation of response action plans in support of the city's planned acquisition and redevelopment of the 100-acre Northwest Quadrant redevelopment area adjoining I-694 and I-35W. The redevelopment involves 15 properties that include nine petroleum release sites, a former refinery and Superfund site, two former dumps with landfill gas (methane) migration concerns, and other concerns related to past solvent and chemical use. The work includes assessing the soil, groundwater, and vapor impacts and developing and implementing response action plans in support of a mixed-use redevelopment and new public infrastructure (e.g., roads, piped utilities, stormwater ponds, and foundations). The majority of the cleanup has been completed, and the city and its developer partners have developed about eighty percent of the project, including four corporate offices, a luxury apartment building, two new parks, and a 25-acre residential area with 126 homes and townhomes.
- Assisting the City of St. Louis Park with investigation and management of old dump materials that were encountered during a park redevelopment. The project involved improving park features and expansion of a dry retention basin to address neighborhood flooding concerns. The project included partial removal of dump materials, establishment of an appropriate soil cover over the remaining areas of the dump, and coordination with the MPCA.
- Directing the investigation and cleanup planning for the new Surly Brewing Co. development located on the border of Minneapolis and St. Paul. The redevelopment site has a long history of industrial use, including a variety of environmental legacy concerns. Work has included helping to secure $2 million in environmental grant funding, conducting Phase I/II site assessments, assessing geotechnical requirements, response action planning, regulatory coordination, site demolition with beneficial on-site reused of crushed aggregate, and rehabilitation of an existing water supply well in support of the new brewery development. The brewery opened in December 2014.
- Assisting the city of New Brighton with cleanup and redevelopment of two petroleum-release sites into new commercial businesses. Reviewed the past investigation results

and prepared development response action plans (DRAPs) to address the residual contamination in support of the planned commercial redevelopments. All work is being coordinated with the MPCA's petroleum brownfield program.

- Assisting the city of New Brighton with several demolition efforts to clear land of aging commercial and industrial facilities in preparation for redevelopment. The work included planning and coordination of hazardous substance abatement (including asbestos, lead paint, and mercury switches), assistance with public bidding, and oversight and testing during demolition work.
- Assisting several of Barr's clients in successfully obtaining more than $9 million dollars in grant and reimbursement funding for numerous environmental projects. The funding sources have included brownfield grants from the U.S EPA, Minnesota Department of Employment and Economic Development (DEED), Metropolitan Council, Hennepin County, Ramsey County, Minnesota Petrofund tank program, Wisconsin PECFA tank program, and special bonding requests to state and federal legislatures.
- Planning and coordinating a unique U.S. EPA Superfund cleanup at 35 residential properties located adjacent to a former wood-treating facility. Previous cleanups had addressed the majority of the contamination from the historical wood-treating operations, but recent data identified low-level dioxins in residential yards and interior house dust. A remedial action for residential dust reduction was negotiated and implemented at the request of the U.S. EPA. The work involved coordinating access to homes, temporarily relocating residents to motels, carpet removal and replacement, duct cleaning, and extensive cleaning of nearly every interior surface of the homes. To control potential future sources of contaminated dust, the residential yards were covered with three inches of clean topsoil and re-vegetated, and the residential driveways were covered with three inches of clean gravel. Ongoing efforts include arrangements for periodic supplemental cleaning of homes to remove accumulated dust and application of dust suppressant to unpaved roads in the neighborhood. A permanent remedy is being negotiated with U.S. EPA.
- Assisting Xcel Energy with planning and managing historical impacts to soil and groundwater as part of a $700-million project involving demolition and reconstruction of two electric-generation plants that were upgraded and switched from coal to natural gas-the Riverside power plant in Minneapolis and the High Bridge power plant in St. Paul. Developed a soil-management plan to address historical concerns from the past 100 years of power-plant operations including petroleum releases, asbestos-containing materials, and buried ash, slag, and coal. The soil management was also coordinated with development of updated plans for stormwater management and closure of the handling facilities for coal, ash, and slag.
- Designing and negotiating regulatory acceptance for a risk-based redevelopment plan to convert a former demolition dump with PAH and lead contamination into a new park and recreation area. The innovative design work involved coordination of the in-place dump closure with the park redevelopment (including ball fields, retaining walls, landscaping, geotechnical design, parking lots, and utilities). The project also involved

protection and enhancement of an adjoining wetland and creek in coordination with the watershed district and regulatory authorities.

- Directing environmental planning and negotiated regulatory liability assurances on a series of projects for the city of Golden Valley which led to redevelopment of several adjoining contaminated properties into a new office and warehouse business park, along with the associated streets and utilities. The work involved investigating the properties, identifying environmental concerns, preparing a comprehensive corrective action plan, and assisting with implementation of institutional controls. All efforts were coordinated with the redevelopment plans to focus the environmental cleanup on the actual future land use. The design work included developing a soil management plan to address the poor geotechnical site conditions and the soil and groundwater contamination (petroleum, chlorinated VOCs, and PAHs).
- Directing a remedial investigation, focused feasibility study, and prepared a response action plan for a site in Minneapolis that had formerly been an automotive battery recycling operation. Worked with the Minnesota Department of Transportation (MnDOT) to implement the remedial action, which involved excavation and on-site stabilization of the lead-contaminated soil. The City of Minneapolis plans to redevelop the site.
- Designing a series of response action plans associated with redevelopment of a former railyard with petroleum and solvent contamination into a business park with new roads, office buildings, and parking. The environmental response plan includes safe, onsite management for most of the contaminated soil combined with a geotechnical soil correction for the proposed buildings.
- Assisting the city of Inver Grove Heights to address historical petroleum releases and farm dumps that were encountered as part of their construction of new frontage roads, stormwater ponds, and related utilities along the Highway 52 corridor.
- Assisting the cities of New Brighton and Burnsville with new stormwater ponds that were constructed near historical petroleum release sites. The work included review of previous environmental investigations and development of remedial plans to address residual groundwater impacts that could impact the new pond's water quality.
- Conducting numerous Phase I environmental site-assessment projects involving property transfers.

Environmental assessment and investigations

- Directing environmental assessment and cleanup planning in support of Barr's master planning efforts to treat and store stormwater for reuse as part of a district system in the Prospect Park neighborhood in Minneapolis, Minnesota. The planned district system involves constructing new stormwater infrastructure in phases to serve eight acres of emerging development owned by four different developers. Barr's work was conducted for the Mississippi Watershed Management Organization and involved close coordination, facilitation, and design development across a group of diverse stakeholders, including the watershed, the City, Hennepin County, MPCA, the neighborhood association, the University of Minnesota, and local developers. Barr

helped the stakeholders develop an approach to assign costs and benefits that were ultimately adopted in final landowner agreements to finance the project in a public-private partnership.

- Assisting Bassett Creek Watershed Management Commission on multiple projects related to stormwater management and stream bank improvements. The work has included Phase I ESAs for the creek corridors and Phase II investigations in support of stream bank stabilization in the presence of brownfield properties.
- Serving as senior civil engineer on the Mouse River enhanced flood protection project. Directed hazardous, toxic, and radioactive waste (HTRW) assessment activities in support of a 13-mile-long enhanced flood protection project on the Mouse River near Minot. The HTRW supported planning, design and permitting efforts, including desktop review of available environmental records for the project area. Phase II environmental drilling investigations were also completed to further assess some project segments in Minot that are advancing towards construction where historical environmental sites posed additional risk to the project (e.g., known petroleum releases).
- Serving as senior civil engineer on the fast-track project to realign the 100-year-old Trout Brook storm-sewer interceptor in support of a new highway interchange near downtown St. Paul. The project area involved petroleum contaminated soil and groundwater that had to be managed during the complex interceptor replacement in a congested urban setting. BNSF Railway agreed to a rare 30-hour shutdown of two mainline railroad tracks to allow project work that included removal and replacement of railroad track, contaminated soil excavation, dewatering, and installation a new box culvert. The project involved coordination with numerous government agencies, consultants, and contractors.
- Directing Barr staff working with MnDOT on a variety of environmental projects under an emergency contract that was funded by federal stimulus funds. The sites involved environmental investigations (Phase I/IIs), response action plans, and oversight of contamination cleanup for new highway construction projects throughout northern Minnesota involving petroleum releases and old dumps.
- Assisting the City of Oslo, Minnesota to address environmental legacy concerns as part of a fast-track flood control project to control flooding on the Red River of the North. The project work included a hazardous, toxic, and radioactive waste (HTRW) assessment; coordination of pre-demolition surveys to identify hazardous substances in more than 20 buildings and structures; Phase II field investigations to delineate a petroleum release in an area where the city's water supply tank was to be relocated for a new flood wall; and coordination with environmental regulatory agencies. The petroleum release was remediated in conjunction construction of a new water-supply tank for the city.
- Assisting the City of Hopkins and the Nine Mile Creek Watershed district to address environmental legacy concerns as part of a streambank stabilization project on a 1.4-mile long corridor of the city with numerous contaminated sites including petroleum releases, old dumps, manufactured gas plant sites, solvent sites, and demolition fill.

The project work included performing Phase I and II investigations, preparing a response action plan, and successfully obtaining $364,000 in grants from the Hennepin County Environmental Response Fund to reimburse investigation and cleanup costs. The environmental cleanup approach was designed in conjunction with the elements of the creek restoration project that addressed stabilization of eroding banks; creation of new channel segments; maintenance dredging of stormwater ponds; and construction of new stormwater outfalls as well as park paths, bridges, and bike trails.

- Assisting Hennepin County on a series of projects under Barr's master services agreement, including Phase I and Phase II environmental site assessments and development of response action plans. The work has spanned a wide variety of projects including brownfield redevelopment, stormwater projects that encountered legacy contamination, and litigation support to the county as an environmental expert to help resolve a dispute between the county and their highway construction contractor over the cost of unexpected contamination.
- Directing a Phase I corridor study and targeted Phase II environmental investigations in support of MnDOT's reconstruction of the I-35W and Highway 62 interchange (Crosstown Highway). The Phase I/II work was conducted to assess for subsurface environmental concerns that may affect the reconstruction of this critical 5 mile urban transportation corridor for the Twin Cities. The reconstruction of the 5-mile-long project corridor will involve 24 bridges, new ramps/retaining walls/sound walls, stormwater management ponds, and some reconfiguration of adjacent local streets and utilities.
- Directing environmental investigations and related property cleanup for the first light rail transit project in the Twin Cities metropolitan area. The project involved a 12-mile rail transit corridor through an urban setting. Preliminary planning and cost estimating was conducted with MnDOT. Following that, Dan directed targeted environmental investigations, developed a response action plan, and implemented the necessary response actions during rail line construction. The project was successfully completed by a design-build project team involving an innovative, multi-party public/private partnership.
- Assisting with a RCRA facility investigation and implemented a RCRA closure plan for an Oregon site with a release of petroleum distillates to soil and groundwater.
- Directing screening site inspections (SSIs) under CERCLA at three former municipal dumps in Minnesota. The SSIs were conducted with the Minnesota Pollution Control Agency and the U.S. EPA to develop a hazard ranking score that was used to evaluate sites for the EPA Superfund National Priority List and MPCA Permanent List of Priorities.

Remediation

- Assisting Capitol Region Watershed District and MnDOT with a fast-track project to realign a 100-year-old storm-sewer interceptor to make way for new highway interchange bridges near downtown St. Paul. The project area involved petroleum contaminated soil and groundwater that had to be managed during the complex

interceptor replacement. BNSF Railway agreed to a rare 30-hour shutdown of two mainline railroad tracks to allow removal and replacement of railroad track, installation a new box culvert, open-cut excavation, and backfilling. Months of planning preceded the effort and involved government agencies, consultants, and investigative contractors. The excavation needed to be completely dewatered prior to construction, requiring permits for disposing of contaminated groundwater and impacted soils and the design of a sophisticated track-monitoring system to verify that dewatering did not affect the surrounding railway. Construction was completed successfully and rail service restored on time, minimizing disruptions and enabling the MnDOT's highway project to move forward.

- Assisting International Paper Company with several efforts to address concerns from a former wood-treating facility located in Cass Lake, Minnesota. The work has included investigations and a feasibility study to evaluate many alternatives for addressing widespread areas of dioxin in soil at the site and in nearby residential areas. The potentially impacted areas under study involve hundreds of acres of land, including more than 100 residences in surrounding neighborhoods. Also directed interim remedial actions to remove areas of soil at the site with high concentrations of dioxin, cover residential yards near the site with clean soil, and arrange for periodic cleanings of residences and dust suppression on unpaved roads. The site is located within the Leech Lake Band of Ojibwe Reservation, and investigation and cleanup efforts are subject to complex negotiations between the International Paper, U.S. EPA, state agencies, local government, and the tribe.
- Helping a large iron mine in northern Michigan respond to regulatory concerns about historical tailings releases to wetlands and streams. Work involved evaluating the extent of the releases, evaluating options for dredging tailings from streams, and assisting with permitting work in wetlands and surface water.
- Directing the cleanup, decommissioning, and demolition of a large bulk-petroleum-storage facility at a former mine in northern Michigan. The work included recovery and recycling of the tank contents, demolition and recycling of the metal tanks, and evaluation and management of petroleum-impacted soil.
- Directing a remedial design and remedial action under CERCLA (Superfund) at a former waste-oil disposal facility at Douglassville, Pennsylvania. The work included negotiating, planning, designing, and providing project management for a $15 million cleanup effort that involved excavation, on-site stabilization, and on-site landfilling of 46,000 cubic yards of used-oil filter-cake sludge. Detailed procedures were developed for monitoring waste treatment, controlling and monitoring air emissions, and collecting and treating wastewater generated from runoff.
- Conducting a feasibility study for the former Reserve Mining scrapyard and landfill located at the current North Shore Mining facility near Silver Bay, Minnesota. The work involved evaluation of a range of on-site and off-site alternatives for managing buried scrap, debris, and drummed waste (including some RCRA hazardous wastes) associated with a nearby taconite plant. The remedial alternatives were developed to address direct contact and groundwater pathway risks that were identified by Barr's

remedial investigation at the site. The work was conducted for the Minnesota Pollution Control Agency.

- Directing long-term operations and improvements for a groundwater remediation system at a Superfund site that addresses a large solvent release from an old chemical dump in Oakdale, Minnesota. The work has involved regulatory negotiations and evaluating various enhancements to the system to ensure that remedial objectives are met while economically maintaining the groundwater remediation system.
- Conducting a focused feasibility study to evaluate remedial options and potential environmental response costs for a former wood tar site located in Kipling, Michigan. The study considered a range of both onsite and offsite remedial options that could support site redevelopment.
- Conducting an evaluation of potential remedial costs for the Cliffs-Dow wood tar site in Marquette, Michigan. The study considered a range of both onsite and off-site remedial options that could support site redevelopment.
- Designing and coordinating a remedial action under CERCLA (Superfund) at a former coal gasification facility in Dubuque, Iowa, that had extensive coal tar contamination in the soil and groundwater. The design, which was coordinated with the city, the Iowa DOT, and MidAmerican Energy, included redeveloping a portion of the site into a new highway corridor. The remedial action included excavation, processing, and offsite thermal treatment of coal tar and heavily contaminated soil at a coal-fired power plant. Soil with residual contamination was managed onsite under a clean cover and a groundwater extraction and treatment system with sanitary sewer discharge was installed to address the groundwater risks.
- Directing the cleanup, decommissioning, and demolition of a large bulk-petroleum-storage facility at a former mine in northern Michigan. The work included recovery and recycling of the tank contents, demolition and recycling of the metal tanks, and evaluation and management of petroleum-impacted soil.
- Assisting with preparation of RI/FS work plans and supporting documents for several contaminated sites, including former coal gasification facilities in Chicago and Iowa and a former lead-battery recycling facility in Minneapolis.
- Assisting with feasibility studies for evaluating remedial options for contaminated soil, groundwater, and wastes at numerous sites, including a former railroad switchyard with an extensive petroleum release, a former uncontrolled municipal dump that contained lead contamination, and a Chicago railyard with lead and PCB soil contamination. The Chicago railyard study included development of a probabilistic cost evaluation for possible remedial alternatives.
- Conducting an underground-storage-tank management project for the U.S. Postal Service that involved more than 125 tanks at 90 locations in Minnesota and North Dakota. The project included site visits and reports summarizing recommendations to comply with new tank regulations and to minimize environmental liabilities associated with tank operation. Subsequent work involved design and construction observation

during replacement of tanks at several post offices and management of contaminated soil and groundwater at sites where petroleum had been released.

- Planning and coordinating a soil remediation at a former automotive battery-cracking operation at a railyard in La Crosse, Wisconsin. Lead-contaminated soil was stabilized in situ prior to excavation and off-site disposal. The work was coordinated with the city of Lacrosse and the Wisconsin Department of Natural Resources in accordance with NR 700 rules.
- Developing probabilistic remedial cost estimates for two contaminated rail yards and a waste oil disposal site. Responsibilities included developing potential remedial strategies, evaluating key technical/regulatory uncertainties, assigning probabilities, and developing an estimated range for remedial costs.
- Providing technical expertise and negotiating with the Wisconsin Department of Natural Resources for two former manufactured gas plant (MGP) sites that were located adjacent to rivers in urban settings. The work included assessing impacts to soil, groundwater, and surface water in accordance with Wisconsin NR 700 rules and evaluating MPG-related structures still on the sites. The work at one of the sites included coordination of an Interim Removal Action to address potential impacts to the surface water and preparation of a site investigation work plan. The work at the second site included preparation of detailed plan and cost estimate for implementing a remedial action to stabilize and cap MGP waste along a river bank as part of a planned redevelopment of the site into a city park.
- Assisting with remedial investigations/remedial alternative evaluations at numerous Holiday gas stations in Wisconsin. All work was conducted in accordance with NR 700 and Department of Commerce rules and guidance regarding petroleum release sites and PECFA-reimbursement requirements.
- Assisting with the remedial design to address solvent-contaminated soil near a former drum burial area at a site in Monroe, Wisconsin. Developed site-specific, performance-based soil cleanup goals for land treatment in accordance with NR 718 and 720.
- Providing technical review and recommendations the City of New Brighton in support of their response to citizen complaints for sites involving noise and odor concerns.

While with another consulting firm, Dan focused on the investigation and remediation of soil and groundwater at contaminated sites. His work included:

- Conducting feasibility studies for material handling and thermal treatment of contaminated soil at a large petrochemical facility on the EPA s National Priority List.
- Observing tank removals and performing remedial investigations at numerous underground-storage-tank sites in accordance with MPCA guidance documents.
- Assisting with the design and implementation of various remedial actions at sites with contaminated soil and groundwater.
- Conducting numerous environmental property assessments prior to land purchase or development.

- Assisting with the development of equipment for soil-gas testing and thermal treatment of contaminated soil.

Education BS, Civil Engineering, University of Minnesota, 1988

Registration Professional Engineer: Minnesota, Iowa, Michigan, Wisconsin

SHERYL FILBY WILLIAMS, PG
Vice President, Senior Hydrogeologist

Experience

Sheryl Filby Williams has more than 17 years of experience with environmental and hydrogeologic investigations and a master's degree in hydrogeology from the University of Minnesota. She is a licensed professional geologist in the states of Minnesota and Wisconsin. Her experience at Barr has included:

- Managing a long-term assessment and remediation project at a former manufacturing site in Minnesota. Project activities have included long term operation and maintenance of two groundwater remediation systems, multiple soil and groundwater investigations, groundwater modeling, a feasibility study, vapor intrusion study, and an in-situ biobarrier remediation system installation. The project is part of the Minnesota Pollution Control Agency's VIC program.
- Managing a long-term assessment and remediation project in Minnesota. Project activities have included soil and groundwater investigations, long-term planning related to institutional controls, vapor intrusion investigations, and stakeholder communication. The project is part of the EPA's Superfund program.
- Managing a long-term assessment and remediation project at an industrial facility in Iowa. Project activities have included site investigation, groundwater modeling, regular groundwater sampling, and annual reporting.
- Managing a brownfield redevelopment project in Duluth, Minnesota. Project activities have included assistance with grant writing to obtain funding for the project and development and implementation of a response action plan during utility construction activities. This project is part of the Minnesota Pollution Control Agency's Voluntary Investigation and Cleanup (VIC) program and has used funding from the U.S. Environmental Protection Agency and Minnesota Department of Employment and Economic Development.
- Managing a long-term assessment and remediation project at a former manufactured gas plant site in Michigan. Project activities have included product recovery, regular groundwater sampling, rotasonic drilling, groundwater flow and solute transport modeling, and feasibility studies for remediation options.
- Performing Phase I assessments of industrial and commercial properties in Minnesota as an "environmental professional" under the ASTM standard (E 1527-05) for Phase I environmental site assessments.
- Using the groundwater modeling codes MODFLOW, RT3D, MT3D, PEST, MLAEM, SLAEM, AQTESOLV, and MODFLOW SURFACT in combination with Surfer and GIS to assist in site decisions across the U.S.
- Using groundwater modeling to evaluate how historical pumping has affected a
- Performing rotasonic drilling and vertical aquifer profile sampling to investigate potential dense non-aqueous phase liquid (DNAPL) migration at a manufactured-gas-plant site.
- Performing a subsurface bedrock and hydrogeologic investigation using rotasonic drilling in Minneapolis.

- Performing a Geoprobe investigation as part of a Phase I/II investigation for the City of Duluth.
- Performing a geophysical investigation (electromagnetic survey) of a site in St. Paul to investigate possible underground tanks, pipes, and structures.
- Digging test trenches and assisting with on-site excavation.
- Performing a geophysical investigation involving electromagnetic survey, seismic reflection, seismic refraction, and multi-channel analysis of surface waves for the purpose of delineating bedrock depth and investigation of historic structures.

Sheryl's master's research experience includes developing a one-dimensional finite-difference unsaturated-zone model (including soil moisture distribution, heat transfer, evapotranspiration routine, and freeze/thaw calculation), coupled with MODFLOW, to evaluate water resources and paleohydrologic changes in Minnesota's Shingobee watershed. This project was funded by NASA's land-surface-hydrology program.

Education

MS, Geology (hydrogeology emphasis), University of Minnesota, Institute of Technology, 2001

BA, Geology, Gustavus Adolphus College, 1998

Registration

Professional Geologist: Minnesota and Wisconsin

Publications

Filby, S.K., Locke, S., Person, M., Winter, T., Rosenberry, D., Nieber, J., Gutowski, B., and E. Ito. 2002. "Mid-Holocene hydrologic model of the Shingobee Watershed, Minnesota." Quaternary Research 58, 246-254.

Filby, S.K., York, J.P., Person, M.A., and E. Ito. 2000. "A comparison of lake core records to paleoclimate model results in Minnesota." Geological Society of America Abstracts with Programs 32:27.

Person, M.A., York, J., Filby, S.K., Gutowski, B., Neiber, J., and R. Daanen. 2000. "A novel model." Resource 7: 4, 13-14.

York, J.P., Filby, S.K., Person, M.A., Wright, H., and W. J. Gutowski. 2000. "A Holocene hydrologic model of the Crow Wing watershed, Minnesota." Journal of Geochemical Exploration 69-70, 419-422.

Person, M.A., Filby, S.K., and Peter Eadington. 1999. "Oil Brine Migration within the Papuan Fold Belt." AAPG Annual Convention.

Filby, S.K. 1997. "Boulder pavement under the New Ulm till in the Mankato bend area of the Minnesota River Valley." Geological Society of America Abstracts with Programs 29, 15.

MICHAEL DUPAY
Senior Environmental Scientist

Experience

Michael Dupay has 11 years of experience and degrees in chemistry and criminal justice from Hamline University in St. Paul, Minnesota. His experience includes evaluating and creating standard operating procedures for an analytical laboratory, using a laboratory-information-management system (LIMS), and presenting findings before small and large groups. Michael's work at Barr includes:

- Providing advanced statistical services, including principle component analysis (PCA), discriminant analysis (DA), and agglomerative hierarchical clustering (AHC).
- Performing bench-scale testing to test project hypotheses on small scales prior to expansion into pilot-scale testing.
- Providing on-site consulting services to Flint Hills Resources' environmental laboratory. Managed laboratory coordination and data evaluation for a refinery-wide mercury mass-balance effort and a mercury-removal pilot project.
- Developing and revising standard operating procedures for data validation.
- Providing quality assurance and quality control (QA/QC) of CLP reports for a complex contaminated waste site in Michigan.
- Developing and designing the QA/QC team intranet webpage which includes tracking of tasks and laboratory QA/QC issues.
- Performing advanced statistical analysis of data to determine likely chemical relationships in complex water systems to determine possible root causes for a client's water-quality issues.
- Performing computer modeling using PHREEQC, a program for speciation, batch-reactions, one-dimensional transport, and inverse geochemical calculations.
- Performing evaluations and assessments of analytical laboratories
- Assisting in the production of quality assurance project plans (QAPPs)
- Providing general QA/QC of data and writing validation reports
- Serving as laboratory liaison for related questions and supply ordering.
- Assisting with an environmental forensic analysis of hydrocarbons.

Prior to joining Barr, Michael served as a student worker in a laboratory environment for the Drug Chemistry section of the Minnesota Bureau of Criminal Apprehension (BCA). His duties for the BCA included:

- Mixing and maintaining reagent supplies.
- Evaluating and developing standard operating procedures.
- Tracking drug standards and evidence (electronic chain of custodies once the evidence was submitted by law enforcement).
- Validating new equipment and procedures used by the drug chemistry laboratory.

Education

BA, Chemistry, Hamline University, 2005

BA, Criminal Justice, Hamline University, 2005

Certificate of Forensic Sciences, Hamline University, 2005

Training

Essentials of Drinking Water Treatment, University of Wisconsin – Madison, 2017

Basic Assessor Training, ISO/IEC 17025 and NELAC (ASI Course 300), Advanced Systems, Inc., 2010

Contaminant Forensics of Petroleum, Chlorinated Hydrocarbons and Metals (CHEM 405), Northwest Environmental Training Center, 2008

Understanding Water Chemistry for Practical Application, University of Wisconsin – Madison, 2007

Erin Evans
Quality Manager

Summary

Erin Evans has 13 years of environmental testing experience and joined Pace Analytical Services in 2005. She facilitates quality program efforts and is directly responsible for ensuring the overall quality performance of the Field Services Division. Erin Evans reviews and evaluates technician performance and is active in training field personnel in specialized sampling. She is an active member of the Safety Committee. She reviews project documentation for completeness, method compliance, and contract fulfillment. Erin Evans employs spreadsheets to organize and display test results and compiles data into final test reports.

Quality Manager

- Maintains ISO 17025, FSMO, and ASTM 7036 accreditations. Acts upon request of departments to attain certifications necessary for specific projects, and provides factual information on certifications and capabilities on a project basis.
- Reviews and evaluates data integrity and validity including review of client reports; initiates and facilitates the corrective action process as appropriate including a tracking system to ensure follow up/resolution of corrective action.
- Coordinates visits and responses to external audits; conducts appropriate internal audits to ensure departments are performing work in accordance with most current procedures for each task; coordinates pre-audit information gathering and post-audit responses for audits.
- Maintains and oversees distribution of current QAM, SOPs, and Procedure Manuals, and monitors adherence to established Pace procedures. Develops new procedural documents as needed.
- Oversees training requirements of personnel performing test procedures including proficiency testing requirements. Maintains documentation of required training. Evaluates personnel performance and initiates retraining as required.

Groundwater, Surface Water Experience

- Use of field instrumentation to measure pH, specific conductance, dissolved oxygen, redox and water elevations.
- Groundwater sample collection and field monitoring from large volume deep wells using pumping equipment in dedicated and non-dedicated situations.
- Use of groundwater sampling equipment including bailers, bladder pumps, and keck pumps.

Wastewater, Stormwater Experience

- Installation and use of primary wastewater effluent flow monitoring equipment including weirs and flumes, and electronic secondary level and flow measuring devices.
- Installation and operation of programmable wastewater autosamplers.
- Preparation of flow proportional wastewater composites for laboratory analysis.
- Use of continuous pH monitoring instrumentation.
- Use of field instrumentation to measure pH, dissolved oxygen and residual chlorine.
- Confined space entry including operation of associated safety equipment and monitors.
- Sample preparation and sample collection and handling procedures for PFC analysis.
- Sample collection using a Composite Liquid Waste Sampler (COLIWASA)
- Sample collection for drinking water and bacteria analysis.

Education

- Bachelor of Science, Environmental Science, University of Oregon

Memberships & Affiliations

- Source Evaluation Society (SES)

Training & Certifications

- 40-Hour Industrial Hazardous Materials Training (29 CFR 1910.120)
- Confined Space Entry
- Fall Protection

Shane D Olund

Quality Assurance Officer
Environmental Laboratory Section
Public Health Laboratory Division
Minnesota Department of Health
phone: 651-201-5357
Email: shane.olund@state.mn.us

Professional Organizations

Association of Public Health Laboratories 2018 – Present
Environmental Laboratory Sciences Committee Member 2018 – Present

Work Experience:

Quality Assurance Officer - Minnesota Department of Health
03/2013 to present

- Serve as quality assurance officer for the Environmental Laboratory of the Minnesota Department of Health, Public Health Laboratory
- Oversee the quality of data produced in units of the laboratory - Organics, Radiochemistry, Metals, Inorganics and Microbiology
- Function as project manager as the environmental laboratory seeks ISO accreditation, starting with a limited scope of analytes for non-potable waters
- Ensure compliance for the environmental laboratory with EPA rules and regulations to maintain the lab's certification for drinking water parameters through EPA Region 5
- Oversee the proficiency testing program for non-potable and drinking water analytes for which proficiency testing samples are available
- Review contracts and interagency agreements for accuracy on an annual basis
- Review and contribute to Quality Assurance Project Plans for special projects the environmental laboratory participates in
- Create and maintain policies and procedures for the quality management system for the environmental laboratory
- Participate in routine meetings with clients to identify the scope of future work, resolve quality assurance-related issues and maintain positive relationships with clients
- Periodically review data for accuracy
- Manage the environmental laboratory's corrective action system
- Serve as an administrator in the design, implementation and operation of a Quality Management System software package (MasterControl)
- Manage the calibration and verification of support equipment from both outside vendors and laboratory staff

Environmental Analyst - Minnesota Department of Health
05/2008 to 03/2013

- Member of the NELAC/ISO17025 Accreditation Team guiding the environmental lab through its national accreditation process

- Secondary data reviewer for the inorganic unit of the environmental laboratory
- Assistant radiation safety officer
- Lead analyst for ultra-low level methyl mercury and total mercury samples using cold vapor atomic fluorescence spectroscopy (CVAFS)
- Maintain, optimize and troubleshoot CVAFS
- Lead analyst for radium 226 and radium 228 using gas proportional counter (GPC)
- Maintain, optimize and troubleshoot GPC
- Assist environmental laboratory accreditation program in ultra-low level mercury assessments
- Train personnel for MDH radiological emergency preparedness program
- Maintain and update standard operating procedures for ultra-low level mercury, radium 226 and radium 228
- Analyst for radon 222 using liquid scintillation counter (LSC)
- Analyst for orthophosphate using flow injection analysis (FIA)

Chemist - Interpoll Laboratories, Inc.
04/2007 to 05/2008

- Lead analyst for anions (F-, Cl-, NO2-, Br-, NO3-, PO4-, and SO4-) using ion chromatography (IC)
- Lead analyst for alkalinity, ammonia, and total phosphorus using flow injection analysis (FIA)
- Maintain, optimize, and troubleshoot IC and FIA
- Assist in total coliform presence/absence, total coliform membrane filtration and colony count detection
- Assist in total residual chlorine analysis using spectrophotometer
- Assist in sample receiving and sample log in

Chemist/Quality Assurance Officer - U. S. GEOLOGICAL SURVEY
05/1997 to 4/2007

- Co-project leader for multi-agency study of mercury contamination of a San Francisco Bay estuarial salt marsh
    - Plan and organize field sampling trips in collaboration with other agency project leaders
    - Interpret and report mercury lab data to other project leaders
- Lead analyst for ultra-low level methyl mercury samples for stable isotopes using inductively coupled plasma – mass spectroscopy (ICP-MS)
- Maintain, optimize, and troubleshoot ICP-MS
- Responsible for QA/QC validation for isotopic data and upload into database
- Implemented isotopic dilution method currently utilized for stable isotope methyl mercury analysis
- Lead analyst for ultra-low level methyl mercury determinations in water, suspended solids, soils, and biological samples
- Led a USGS method development and prove-out study for the determination of total mercury at low levels in sediments and suspended sediments

- Troubleshoot instrumentation when analysis fails QA/QC objectives
- Train new personnel in detailed analytical practices, instrumentation and ultra-clean sampling procedures
- Prepare and distribution of data reports to customers and collaborators
- Extensive experience in scientific studies involving the collection and preservation of multi-media (water, sediment, plants, and biota) samples
- Led the development and refinement of a procedure for the determination of acid volatile sulfide and ultra-low level reactive mercury in sediments as well as establish QA objectives
- Participated in the validation of USEPA Method 1631 – analysis of total mercury

Education:

University of Wisconsin - Madison, Madison, WI
B.S., Chemistry Course
December 1998
Minor: Environmental Studies

Publications:

- Ericksen, J.A., Gustin, M.S., Lindberg, S.E., Olund, S.D., and Krabbenhoft, D.P., 2005, Assessing the Potential for Re-emission of Mercury Deposited in Precipitation from Arid Soils Using a Stable Isotope: Environmental Science & Technology, v. 39, p. 8001-8007.
- Olund, S.D., DeWild, J.F., Olson, M.L., and Tate, M.T., 2004, Methods for the Preparation and Analysis of Solids and Suspended Solids for Total Mercury: U.S. Geological Survey Techniques and Methods book 5, chap. A8, 15 p.
- DeWild, J.F., Olund, S.D., Olson, M.L., and Tate, M.T., 2004, Methods for the Preparation and Analysis of Solids and Suspended Solids for Methylmercury: U.S. Geological Survey Techniques and Methods book 5, chap. A7, 13 p.
- DeWild, J.F, Olson, M.L., and Olund, S.D., 2002, Determination of Methyl Mercury by Aqueous Phase Ethylation, Followed by Gas Chromatographic Separation with Cold Vapor Atomic Fluorescence Detection: U.S. Geological Survey Open-File Report 01–445, 14 p.

Honors, Awards, and Special Accomplishments:

- MDH Star Honors Positive Impact Award - 2016
- USGS STAR award for method development and prove-out study for analysis of total mercury in solids and suspended solids – 2004
- USGS STAR award for development of AVS and reactive mercury methods of analysis – 2000

Other Information:

- Dynamics of Mercury and Methylmercury in San Francisco Bay Estuarial Marshes; oral presentation at the 8th International Conference on Mercury as a Global Pollutant – 2006
- Instructor for mercury sampling techniques at the NAWQA Ecological Training and Methods workshop – 2002

Bradley Jacobson
Department Manager, Water

Summary	Bradley Jacobson has 29 years of environmental testing experience and joined Pace Analytical Services in 1988. He provides supervisory and project management duties for wastewater projects and groundwater projects. He is responsible for project initiation, contact follow-up, generating cost quotes and proposals, developing sampling plans and preparing Test Plan documents. Bradley Jacobson is a proficient field team leader who reviews and evaluates technician performance and is active in training field personnel in specialized sampling. He reviews project charges and coordinates invoicing and reviews project documentation for completeness, method compliance, and contract fulfillment. Bradley Jacobson is responsible for scheduling and coordinating daily projects including personnel, supply and equipment resources and arranging sample analyses with the laboratory. Bradley Jacobson employs spreadsheets to organize and display test results and compiles data into final test reports.
Air Experience	· Basic Probe Tending
Groundwater, Surface Water Experience	· Groundwater sample collection and groundwater field monitoring procedures including for trace organics and metals pllutants. · Use of field instrumentation to measure pH, specific conductance, dissolved oxygen, redox and water elevations. · Groundwater sample collection and field monitoring procedures at UST sites, petroleum refineries and terminals including use of field instrumentation to measure petroleum product levels. · Groundwater sample collection and field monitoring from large volume deep wells using pumping equipment in dedicated and non-dedicated situations. · Use of groundwater sampling equipment including bailers, bladder pumps, and keck pumps. · Surface water and sediment sampling employing Kemmerer Samplers and Eckman Dredges.
Wastewater, Stormwater Experience	· Installation and use of primary wastewater effluent flow monitoring equipment including weirs and flumes, and electronic secondary level and flow measuring devices. · Installation and operation of programmable wastewater autosamplers. · Preparation of flow proportional wastewater composites for laboratory analysis. · Use of continuous pH monitoring instrumentation. · Use of field instrumentation to measure pH, dissolved oxygen and residual chlorine. · Confined space entry including operation of associated safety equipment and monitors. · Sampler preparation and sample collection and handling procedures for Priority Pollutant and Total Toxic Organic (TTO) analysis following EPA protocols. · Non-point source sampling and flow monitoring for the National Pollution Discharge Elimination System (NPDES). · Sample preparation and sample collection and handling procedures for PFC analysis. · Sample collection using a Composite Liquid Waste Sampler (COLIWASA) · Collection of Volitile Organic Compounds (VOCs) using the QCED Volatile composite Sampler. · Sample collection for wastewater toxicity sampling. · Low-level mercury sampling, including the use of clean hands/dirty hands procedures. · Sample collection for drinking water and bacteria analysis. · Stormwater pond sediment core sampling, Vibracore sediment sampling
Contaminated Site, Hazardous	· Representative sampling and handling of various waste matrices and sampling conditions.

Waste Experience	· Use of field instrumentation to measure landfill gas methane levels and organic vapor levels (OVA, HNu) · Soil sampling techniques that employ soil auger, bar punching and soil sieve.
Education	· BA degree from University of Minnesota Morris; Major in Biology and English
Papers, Publications, Special Projects and Experience	· New Technology Evaluations for Major Clients · Determination of % Volatiles in UV cure adhesives · Audit Surveys
Training & Certifications	· 40-Hour Industrial Hazardous Materials Training (29 CFR 1910.120) · 10-Hour OSHA Training · First Aid/CPR · Confined Space Entry · Fall Protection · Respirator Training

Curriculum Vitae

Paul F. Moyer
14020 Flintwood Way
Apple Valley, MN 55124

Home Phone Number: (651) 470-4229
E-mail Address: paul.moyer@state.mn.us
Country of Citizenship: United States

EDUCATION

University of Pittsburgh Graduate School of Public Health	Master of Science Environmental and Occupational Health	May 1999
University of Pittsburgh	Bachelor of Science Microbiology Chemistry Minor	May 1987

LICENSES AND CERTIFICATIONS

DOT Hazardous Materials Certification
HAZWOPER 40 Hour Certification (Technician Level)

PROFESSIONAL ORGANIZATIONS

Association of Public Health Laboratories	2003 – Present
- Environmental Health Committee	2012 - Present
o Committee Chair	2016 - Present
National Society of Toxicology (SOT)	1999 – Present
Northland Chapter of SOT	2006 – Present
Society for Risk Analysis (SRA)	2008 – 2010
Society for Chemical Hazard Communication (SCHC)	1997 – 2002
- Continuing Education Committee	2001 – 2002

WORK EXPERIENCE

2011 – present
Environmental Laboratory Section Manager
Minnesota Department of Health, Public Health Laboratory, St. Paul, MN

- Manage the overall operation of the Environmental Laboratory Section.
  - Assure that preanalytic, analytic, and postanalytic phases of all testing systems are of highest quality and meet all regulatory and client requirements.
  - Assure that quality control and quality assurance programs are established and maintained.
  - Assure that the laboratory facilities provide an appropriate and

safe environment for the tests performed.
    - Assure that the test methodologies selected are capable of providing the type and quality of results required by clients.
    - Assure the employment of a sufficient number of laboratory personnel with education or experience appropriate for the complexity of testing conducted and assure that all personnel have had appropriate training and have demonstrated their ability to perform the testing operations accurately and reliably.
    - Assure that a procedure manual is available to and followed by all personnel responsible for any testing process.
- Manage the staff of the Environmental Laboratory Section according to all applicable personnel policies and rules so that the needs, goals, and objectives of both the Section and the Division are met.
- Consult on technical matters with Department program staff and other clients of the Environmental Laboratory Section so that they are aware of significant laboratory findings and they recognize the laboratory as an essential resource to assist them in achieving their own particular objectives.

2006-2011 Environmental Research Scientist, Toxicologist
Minnesota Department of Health, Health Risk Assessment Unit, St. Paul, MN

- Serve as Project Manager and Team Lead in planning, designing, and carrying out major sections of the Health Risk Limits rulemaking effort.
- Serve on the team that reviews and develops health-based guidance for toxicants found in groundwater and other media.
- Represent MDH as an expert panel member for "Beyond Science and Decisions: From Issue Identification to Dose-Response Assessment Workshops" (http://www.allianceforrisk.org/ARA_Dose-Response.htm)
- Prepare and deliver information and educational material including presentations, posters, and Web content regarding health-based guidance for various audiences including partners, stakeholders, the public, MDH management, key legislators, and Governor's Office staff;
- Consult with partners and the public on hazard and exposure assessments;
- Serve as a member of the Environmental Health Division's Radiological Emergency Preparedness Ingestions Pathway Response Team and serve in a reserve capacity for other emergency response functions including MNTRAC; and other designated incident management functions.
- Conduct other chemical risk assessments.
- Employ and influence MDH processes and policies to help build and maintain relationships with colleagues from other programs to help facilitate collaborative efforts on intra- and interagency projects.
- Computer proficiency includes: Microsoft Excel, PowerPoint, Outlook, Visio, and Word, and experience with: Microsoft Access.

2002-2006 Chemical Threat Response Coordinator,
Minnesota Department of Health, Public Health Laboratory, St. Paul, MN

- Served as the CDC Laboratory Response Network (LRN) Chemical Terrorism Laboratory Coordinator for Minnesota, which included providing input and support for Level 1, 2, and 3 chemical laboratory activities;
- Provided input into the CDC Preparedness Cooperative Agreement;
- Integrated and designed chemical, biological, and radiological procedures for an all-hazards approach to laboratory emergency response planning.
- Provided outreach activities to promote cooperation with first responders including the state hazardous materials teams and the 55th National Guard Civil Support Team;
- Performed GC/MS and LC/MS/MS chemical analysis following CDC LRN-C protocols for chemical agent exposure measurements in clinical matrices;
- Served as a laboratory representative on working groups for the development of emergency preparedness and response plans at the division, department, and state levels;
- Presented at regional and national meetings regarding emergency response activities such as the development and implementation of plans for handling unknown environmental samples and continuity of operations;

2000- 2002 Hazard Communication Manager,
ChemADVISOR, Inc., Pittsburgh, PA

- Supervised a department of three hazard communication specialists including determining project assignments, reviewing prepared documents (safety data sheets, labels, hazard summaries, transportation classifications, etc.), and serving as a technical advisor to staff and clients;
- Provided input on annual budget and financial projections for the hazard communication services section;
- Served as co-author/instructor for the various hazard communication related courses including MSDS awareness, MSDS authoring, and Canadian Hazard Communication; and
- Provided demonstrations, instruction, and input into the development of MSDS authoring and regulatory database software products.

1997- 2000 Hazard Communication Specialist,
ChemADVISOR, Inc. Pittsburgh, PA

- Prepared hazard communication documents (e.g. safety data sheets and product labels) for a wide spectrum of chemical products for various clients;
- Conducted hazard determination and scientific literature summaries including hazard and occupational risk assessments for products containing a mixture of hazardous chemicals; and
- Applied relevant government regulations pertaining to hazardous chemical hazard communication classifications and documents.

1991-1997 Research Specialist III, Department of Surgery, University of Pittsburgh, Pittsburgh, PA

- Harvest, maintain, and propagate human and other mammalian cell cultures;
- Conduct assays in molecular biology, including nucleic acid extractions, Northern blotting, and polymerase chain reaction (PCR);
- Conduct assays in protein chemistry including protein extraction and isolation, Western blotting, and assays in enzyme activity using ion exchange chromatography and HPLC;
- Reported experimental results and recommendations to the principle investigator; and
- Conducted literature reviews to determine suitability of adapting analytical laboratory techniques and methods to experimental design applications.

1988-1991 Analytical Laboratory Supervisor, Biodecision Laboratories, Pittsburgh, PA

- Supervised three Laboratory Analysts in their daily duties;
- Set-up, calibrated, and standardized liquid chromatography, gas chromatography, and microbiological assays according to U.S. Food and Drug Administration guidelines for pharmacokinetic bio-availability studies; and
- Collected and interpreted study data
- Prepared study reports

1987-1988 Analytical Laboratory Analyst, Biodecision Laboratories, Pittsburgh, PA

- Performed protocols for the analysis of pharmaceutical products from various biological matrices using organic chemistry extraction techniques;
- Assisted with study set-up including preparation of standards and controls; and
- Assisted with daily maintenance of laboratory instrumentation and supplies.

PROFESSIONAL PRESENTATIONS

Minnesota Drug Overdose and Substance Abuse Pilot Surveillance System (MNDOSA) – A Response to the Opioid Crisis. Association of Public Health Laboratories Annual Meeting, Pasadena, CA June 2018

A Response to Emerging Concerns: PHL Roles in PFC Monitoring in Minnesota. Paul Moyer. Association of Public Health Laboratories Annual Meeting, Raleigh, NC June 2013

Minnesota's Study to Measure Mercury Levels in Newborns by Analyzing Newborn Blood Spots, Paul Moyer. Association of Public Health Laboratories Annual Meeting, Seattle, WA May 2012

Doing More with Less: Tools for Screening the Potential Health Risks of Emerging Contaminants with Limited Toxicological Information. Paul Moyer and Helen Goeden. Minnesota Water Resources Conference, October 2009

Integration of Life-stage and Exposure Duration Assessments into Derivation of Standards. Society of Toxicology – Northland Chapter Meeting, Duluth, MN April 2008

Integration of Life-stage and Exposure Duration Assessments into Derivation of Standards. Helen Goeden, Paul Moyer, and Christopher Greene., Society of Toxicology Annual Meeting, Seattle, WA March 2008

Risk Assessment in a World Without Boundaries: Apportionment of Human Health Risk in the Development of Health-Based Water Criteria. Christopher Greene, Helen Goeden, and Paul Moyer, Minnesota Water Resource Conference, Minneapolis, MN October 2007

Stop, Think, and Drink: Protecting Health Through Assessing the Risk of Groundwater Contaminants. Paul Moyer, Christopher, and Helen Goeden., Minnesota Water Resource Conference, Minneapolis, MN October 2007

Use of Multiple Intake Rates in the Derivation of Groundwater Standards. Helen Goeden, Paul Moyer, and Christopher Greene, International Society of Exposure Analysis - Annual Meeting, Durham, NC October 2007

Minnesota's Public Health Laboratory Business Continuity Planning, North Central Consortium Meeting, East Lansing, MI September 2005

A Tiered Approach to All-Hazards Laboratory Testing of Unknown Environmental Samples (Technical Details), Laboratory Response Network Conference, New Orleans, LA May 2005.

A Tiered Approach to All-Hazards Laboratory Testing of Unknown Environmental Samples (Partner Integration), Public Health Preparedness Conference, Washington, D.C. February 2005

Putting the "C" (Chemical) into the Comprehensive Response Plan: The Minnesota Approach. Laboratory Response Network Conference, Atlanta, GA November 2004.

PROFESSIONAL EDUCATION COURSES AND SEMINARS

8-Hour HAZWOPER Emergency Response Refresher, January 2009 - present

Intermediate Topics in Chemical Mixtures Health Risk Assessment, Society for Risk Analysis Workshop, December 2008

Chemical Specific Adjustment Factors: Evaluating and Using Data to Quantify Inter- and Intraspecies Extrapolation for Risk Assessment, Society for Risk Analysis Workshop, December 2008

MDA/MDH Legislative Training Session and Mock Testimony, December 2008

State of Minnesota, Emerging Leaders Institute, September 2008 – March 2009

DOT Hazardous Materials Certification Update, April 2008

Mini-Pigs as an Alternative Non-Rodent Species in Toxicology and Safety Studies, Dose-Response Modeling for Occupational and Environmental Risk Assessment, Society of Toxicology Continuing Education Course, March 2008

Dose-Response Modeling for Occupational and Environmental Risk Assessment, Society of Toxicology Continuing Education Course, March 2008

Nanotoxicology: The Science of Developing a Safe Technology, Society of Toxicology Continuing Education Course, March 2008

Teaching Technical Topics, CDC Sponsored 20 Hour "Train-the-Trainer" Course, August 2006

HPLC Method Development for LC/MS, The Minnesota Chromatography Forum, May 2006

Beginning Gas Chromatography, The Minnesota Chromatography Forum, January 2006

NFPA 472 80 Hour HAZMAT Certification (Technician Level), Rescue Associates Inc., August 2005

Public Health Leadership Skills, Public Health Institute, University of Minnesota, May 2005

Integrated Emergency Management Course for Metropolitan Medical Response System Communities sponsored by FEMA (40 hrs), February 2005

Senior Officials Workshop (SOW) for WMD/Terrorism Incident Preparedness, The National Emergency Response & Rescue Training Center, January 2005

Integrating Laboratory Resources for National Food Security, Association of Food and Drug Officials, June 2003

HAZWOPER 40 Hour Certification (Technician Level), Midwest Center for Occupational Health and Safety, April 2003

Minnesota Incident Management System 16 Hour Training, Midwest Center for Occupational Health and Safety, February 2003

Initial Response to Terrorism Incidents Basic Concepts, Hennepin Technical College, November 2002

Central Region Food Emergency Response Network Workshop, USFDA Forensic Chemistry Center, November 2002

Continuing Occupational Health and Safety Courses offered by the Society for Chemical Hazard Communications, 1997-2002 (40+ Hours)

Appendix B

Laboratories Quality Assurance Manuals, Standard Operating Procedures, and Forms (Pace – Minneapolis, MN, and Minnesota Department of Health – Saint Paul, MN)

Pace Analytical Services LLC
315 Chestnut Street
Virginia, MN 55792

Phone: 218-735-6700
Fax: 218-742-1010

STANDARD OPERATING PROCEDURE

Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC)

Reference Method: SM5310C and EPA 9060A

Local SOP Number:	S-VM-I-019-rev.09
Effective Date:	Date of Final Signature
Supersedes:	S-VM-I-019-rev.08

APPROVALS

Laboratory General Manager	31 OCT 2016 Date
Jodi Carlson (for Tim Harrison) Laboratory Quality Manager	31 OCT 2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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SECTION **PAGE**

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used to determine the concentration of Total Organic Carbon (TOC) in aqueous samples and soils using the OI Analytical Aurora Instrument. This SOP follows analytical methods SM 5310C and EPA 9060A.

2. Summary of Method

2.1. When analyzing water samples, the sample is injected into a reactor filled with a sodium persulfate solution where it is oxidized. The product of the reaction is CO_2 gas, which is then blown into the non-dispersive infrared detector (NDIR). The NDIR uses infrared energy to measure the CO_2. This measurement is proportional to the carbon in the sample. In order to analyze for TOC the water sample must first have the Inorganic Carbon (IC) removed by the addition of phosphoric acid followed by purging with nitrogen.

2.2. When analyzing soil samples, the sample is burned at $900 \pm 20^{o}C$ to convert the carbon to CO_2 gas. The CO_2 gas is detected using a non-dispersive infrared detector (NDIR). Samples are pre-treated with acid to remove Inorganic Carbon (IC) prior to analysis.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. **Parameters**: This SOP applies to total organic carbon.

4. Applicable Matrices

2.1 This SOP pertains to drinking waters, ground waters, surface waters, domestic and industrial waste samples and solid samples.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) is 1.0 mg/L for this method for waters.

5.2. The reporting limit (LOQ) for soil is 300 mg/Kg for this method.

5.3. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Carbonate and bicarbonate carbon represent interferences under the terms of this test and must be removed or accounted for in the final calculation.

6.2. When dealing with a water matrix, this SOP is applicable only to homogenous samples that can be injected into the apparatus reproducibly by means of an auto-sampler syringe.

6.3 Solid samples tend to be non-homogeneous as received, which can produce widely varying results. Solids are dried at low temperature and ground to improve homogeneity.

7.0 Sample Collection, Preservation, Shipment and Storage

Table 7.1 Sample, Collection Preservation, Shipment and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	40 mL VOA vials, a minimum of 2 vials for 5310C and 4 for 9060A	Acidified with 1:1 sulfuric acid to pH<2; no headspace	≤6°C but above freezing	Must be analyzed within 28 days of collection.
Solids	4oz jar	≤6°C but above freezing	≤6°C but above freezing	Must be analyzed within 28 days of collection.

8.0 Definitions

8.1 Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9.0 Equipment and Supplies (Including Computer Hardware and Software)

9.1 Table 8.1 Equipment and Supplies

Supplies	Description	Vendor / Item #
Autopipettors	Checked for quarterly accuracy. Pipet tips Are checked per lot when received at the laboratory.	Eppendorf, or equivalent replacement
Volumetrics	Class A, 100 mL and 1L	Fisher, or equivalent vendor
Narrow mouth storage bottles	FEP (fluorinated ethylene propylene) with screw closure, 135-mL to 1-L capacities	Fisher, or equivalent vendor
40 mL VOA vials	Clear glass vials	C&G, or equivalent vendor
Filters	0.45 µm , 47mm filter	Pall Corporation P/N66068
pH strips	To take the pH of the samples prior to analysis	Fisher, or equivalent vendor
Disposable pipets	To take a small drop of sample for pH confirmation	Fisher, or equivalent vendor
Analytical Balance	Balance capable of weight to 0.0001g	Sartorius, or equivalent vendor
Sample boat	Small quartz cup for solids analysis	OI Analytical
Watchglass	Glass or HDPE	Fisher
Crucible	Glass or Porcelain, 20ml capacity	Fisher

Supplies	Description	Vendor / Item #
TOC Analyzer	Aurora with NDIR Detector equipped with autosampler and IR detector	OI Analytical/ Model 1030
TOC Autosampler		OI Analytical/ Model 1088
Compressed Nitrogen		Praxair or equivalent
Compressed Oxygen		Praxair or equivalent
Software	Laboratory Information System for reporting data	Horizon, current version in master list
Software	TOC Reporter for TOC 1030 Aurora	OI Analytical/ V1.4.2

10. Reagents and Standards

10.1. ***Table 10.1: Reagents, Intermediate and Working Solutions***

Reagent	Concentration/ Description	Vendor/ Item #
DI Water	Carbon Dioxide free water	
Phosphoric Acid (H_3PO_4)	5% (v/v) – Fill 1 L volumetric flask with 500 mL DI water. Pour 50 mL phosphoric acid into the volumetric. Transfer to 1000 mL bottle and label. Expires 3 weeks from preparation.	BDH/BDH3104-2.5LPC or equivalent replacement
Sodium Persulfate	10% (w/v) – Dissolve 100 g of sodium persulfate into a 1 L volumetric flask, add 500 mL DI water. Dilute to volume with DI water.	JT Baker V035-07, or equivalent replacement
Primary Stock Potassium Hydrogen Phthalate (KHP)	1000 mg/L Used to prepare Stock, Intermediate and working standards (see section 10). This is used for ICAL, MS/MSD and CRDL. Expires 5 years from date of receipt unless otherwise specified from the vendor.	Fluka/076067-250ML-F Or equivalent replacement
Secondary Stock Potassium Hydrogen Phthalate (KHP)	10,000 mg/L Used to prepar ICV, CCV and LCS standards and spikes. Expires 5 years from date of receipt unless otherwise specified from the vendor.	Ultra Scientific/25 IQC-101-5 Or equivalent replacement
Sulfuric Acid	Concentrated lab grade for sample preservation if found to be inadequately preserved from the field	Fisher or equivalent replacement
Soil Primary Stock Calcium Carbonate (CaCO3)	120,000 mg/Kg C Used as received for Calibration and MS/MSD analyses	Fisher or equivalent replacement
Soil Secondary Stock Calcium Carbonate (CaCO3)	120,000 mg/Kg C Used for verification of soil Initial Calibration	Acros Organics or equivalent vendor
Nutrients in Soil (TOC)	Varies by lot.	ERA or equivalent
Phosphoric Acid	5% Used for testing soil samples for the presence of Inorganic Carbon.	Fisher or equivalent vendor
Solid TOC QC	Used for Soil TOC LCS to verify preparation procedure	ERA Nutrients in Soil

10.2 Working Standard Dilutions and Concentrations for Waters

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Calibration Std 1	0.1 mL	Water	99.9 mL	100 mL	1.0 mg/L
Calibration Std 2	0.5 mL	Water	99.5 mL	100 mL	5.0 mg/L
Calibration Std 3	0.2 mL	Water	19.8 mL	20 mL	10 mg/L
Calibration Std 4	0.5 mL	Water	19.5 mL	20 mL	25 mg/L
Calibration Std 5	1.0 mL	Water	19.0 mL	20 mL	50 mg/L
Initial Calibration Verification Standard	0.25 mL	Water	99.75 mL	20 mL	25 mg/L
Continuing Calibration Verification Standard and LCS	1.25 mL *	Water	498.75 mL	500 mL	25 mg/L
Spiking Standard (for MS/MSD)	0.5mL	Water	19.5 mL	20 mL	25 mg/L

* Prepared from 10,000 ug/mL stock standard, second source

10.3 Working Standard Concentrations for Soils

Standard	Standard Amount	Final Concentration Mg C	Final Concentration Mg/Kg
Calibration Std 1	0.0025 g	0.3	300
Calibration Std 2	0.0050 g	0.6	600
Calibration Std 3	0.0125 g	1.5	1500
Calibration Std 4	0.0250 g	3.0	3000
Calibration Std 5	0.0500 g	6.0	6000
Calibration Std 6	0.1000 g	12.0	12000
Initial Calibration Verification Standard	0.0250 g	3.0	3000
Continuing Calibration Verification Standard	0.0250 g	3.0	3000
Spiking Standard (for MS/MSD)	0.0250 g	3.0	3000

Soil standards are prepared by weighing out CaCO3 into the cup to be burned by the solids analyzer. A SRM is analyzed for TOC soils LCS.

11. Calibration and Standardization

11.1 ***Table 11.1. Calibration and Standardization.***

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression	Correlation coefficient ≥ 0.995	If not met, stop analysis. Review for preparation or calculation errors. Perform and document any necessary maintenance. Reanalyze before sample analysis.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ±10%	If not met, stop analysis. Review for preparation or calculation errors. Reanalyze 1 additional time, if it doesn't pass stop analysis and recalibrate the

			instrument.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 samples thereafter. Samples must be bracketed with a closing CCV standard.	% Diff ±10%	If not met, stop analysis. Review for preparation or calculation errors. Reanalyze 1 additional time, if it doesn't pass stop analysis and recalibrate the instrument. Reanalyze all samples since the last passing CCV.
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately follow each ICV and CCV	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in the blanks should be non-detect	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x the blank detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination; If sample result <10x the blank detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Reporting limit standard	Immediately following the initial calibration	60-140%	If not met, stop analysis. Review for preparation or calculation errors. If the RL does not pass, evaluate the next level in the initial calibration to see if it meets the criteria. If the data quality objectives are met with the higher RL, request the RL be raised. If not, recalibrate the instrument to meet criteria.

11.2 Prepare calibration and other QC standards according to the calibration prep log and load into the autosampler rack in the correct spots.

11.2.1 Soil standards are added onto quartz wool in the solid sample cups and are analyzed one at a time using the 1030 soil module.

11.3 Calibration using blank and five calibration standards. The suggested initial calibration levels are in Section 10.

12 Procedure

12.1 Sample Preparation for Liquids:

12.1.1 Pour DI into 40mL VOA for the method blank (MB). For aliquots fill the VOA vials at least ½ full. The actual volume does not need to be recorded, just ensure enough volume present for the instrument to draw an aliquot.

12.1.2. Pour the secondary stock standard at 25 mg/L into 40 mL VOA vials for the LCS and CCVs.

12.1.3 Shake sample vigorously to suspend any sediment present in the sample prior to taking an aliquot for analysis.

12.1.4 Take the pH of the sample by taking a small aliquot with a disposable pipet, note < or > 2 on the batch worklist. Do not put the pH strip into the sample container. The pH from field preservation should be <2 pH units, if the sample pH is > 2, add additional sulfuric acid, ~0.5 mL. Shake and recheck the pH. Note that additional acid was added to the samples on the batch worklist. Pour a sample aliquot into 40mL VOA vial.

12.1.5 For DOCs, if the samples have not been field filtered, filter the samples through a 0.45 µm filter. Any time samples are laboratory filtered, also prepare a laboratory filter blank by filtering DI through the same filters to ensure that no laboratory contamination was present. Collect the filtered aliquot into the 40 mL VOA vials for analysis.

12.1.6 To prepare the matrix spike samples, with pipette add 0.5mL of Primary Stock Standard and 19.5mL of sample into a 40 mL VOA vial for a final concentration of 25 mg/L. Pour into 40 mL VOA for MS and MSD's. For DOC analysis, the MS/MSD would be prepared from a filtered aliquot of sample.

12.1.7 Load into autosampler as instructed by the manufacturer.

12.2 Sample Preparation for Soils:

12.2.1 Place a representative 2-10g subsample into a dish and slowly add 5% H3PO4 drop-wise until effervescence stops and air-dry overnight. (Alternately, place the sample into a crucible and dry in a 105^{o}C oven overnight.)

12.2.1.1 After drying, grind samples with a mortar and pestle to break up larger particles and homogenize the sample.

12.2.1.2 Rocks and detritus should be excluded from the sample portion used for analysis.

12.2.1.3 Larger fragments of leaves, twigs and wood should be removed, unless the client has indicated that they are to be included. If they are to be included, mechanical grinding may be necessary to reduce particles to a size where they can be homogenized in the sample.

12.2.2 Weigh a small portion of sample (0.002 to 1.0g) into a tared sample cup. Record the sample weight in the TOC Soil Log Book. Make sure that all cups are conditioned before weighing, and do not touch cups with your bare fingers.

12.2.3 Load the sample cup into the 1030 soil module.

12.3 Sample Analysis for Water:

12.3.1. Load samples and batch QC into designated auto-sampler location making sure the correct spot is used.

12.3.2. Schedule begins with a rinse followed by a CCB/MB and CCV/LCS. Run a CCV/CCB every 10 samples and at the end of the run.

12.3.3 **For 5310C analysis**: The sample is injected into the sparge vessel, acidified and is heated to 70 ^{o}C and purged with nitrogen to drive off the inorganic carbon. Then the sample has sodium persulfate added is heated to 98 ^{o}C and is purged for 2 minutes and the CO2 generated is detected by the NDIR. The TOC analyzer reads two injections of the sample that should be within 10% RPD to be a valid analysis for 5310C. The average of the two injections is reported. If the average readings are at or below the RL, precision is NOT calculated. If the RPD is > 10% for a value above the RL, the sample must be rerun. No result above the curve will be accepted. The sample must be diluted and rerun.

12.3.4 **For 9060A analysis**: The sample is treated the same as 5310C for injection with the exception that the samples are pre-purged for 10 minutes prior to loading for analysis. For 9060A, the analyzer is set up to read four injections of the samples. The average and the individual readings are reported for the final results. If the RSD is > 40% for a value above the RL, the sample must be rerun or flagged. No result above the curve will be accepted. The sample must be diluted and rerun.

12.4 Sample Analysis for Soil

12.4.1 Samples cups are conditioned by acid and DI rinsing and then dried in the oven.
12.4.2 Samples are analyzed one at a time. Load the prepared sample cup into the cup holder, close the sliding door to the sample chamber, and press the green start arrow.
12.4.3 The sequence will pause and wait for the next sample cup to be loaded. The next sample cup may be placed in the holder when the holder is retracted from the furnace. The green start arrow must be pressed for each sample.
12.4.4 Soils are analyzed duplicate or quadruplicate and must be entered into the sequence individually.

Quality Control

13.1. ***Table 13.1. Quality Control for Water***

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination; If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	DI water spiked with all target compounds	One per 20 samples	90-110%	Analyze a new LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 10 samples	80-120%	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD	MS Duplicate	One per 10 samples	%Diff ≤ 20%	Report results with an appropriate footnote.

13.2. ***Table 13.2. Quality Control for Soil***

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	SiO_2	One per 20 samples	Target analytes must be less than reporting limit.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination; If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Solid TOC standard 0.0250 g of CaCO3 or 0.2 g of SRM	One per 20 samples	70-130% or within QC provider's certified limits.	Analyze a new LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run ***Exceptions:*** If LCS recovery is > QC limits and the samples are non-detect, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with 3.0 mg C which is 0.0250 g of CaCO3.	One per 10 samples	70-130% until laboratory limits are established	If LCS and MBs are acceptable, the MS/MSD may be reported with appropriate footnote indicating matrix interferences
MSD	MS Duplicate 3.0 mg C which is 0.0250 g of CaCO3.	One per 10 samples	%Diff ≤ 25%	Report results with an appropriate footnote.
Duplicate Analysis	As required	Every sample, as required	%RPD ≤ 30% until laboratory limits are established.	Repeat replicates to meet %RPD. If samples are not able to be homogenized, report average and range with footnote for %RPD failure.
Quadruplicate Analysis	As required	Every sample, as required	%RSD ≤ 30% until laboratory limits are established.	Repeat replicates to meet %RSD. If samples are not able to be homogenized, report average and range with footnote for %RSD failure.

14. Data Analysis and Calculations

14.1. SW846 9060A average Sample result (quadruplicate)

$$\text{Quad Average Result} = \frac{(TOC1) + (TOC2) + (TOC3) + (TOC4)}{4\{\text{number of injections}\}}$$

14.2 Soil Sample results (mg/kg) = mg C result/ sample weight in kg
Soils are dried prior to analysis, so no correction for %solids is necessary.

14.3 Percent Recovery: (for QC, RLV use no value for the sample result):

((spiked sample result – sample result)/(spike concentration))*100

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See table in section 13.

16 Corrective Actions for Out-of-Control Data

16.1 See table in section 13.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-VM-Q-016, Method Detection Limit Studies for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Training Procedures.

18.4. **Periodic performance evaluation (PE)** – samples are analyzed to demonstrate continuing competence per SOP S-VM-Q-017, or equivalent replacement. Results are stored in the QC office.

19. Method Modifications

19.1. SM 5310C states that a laboratory control spike and method blank should be analyzed every ten samples. Since the CCV and CCB are equivalent to a laboratory control spike and method blank respectively, the laboratory uses CCV/CCB pairs as equivalent to LCS/MB pairs every ten samples. The QC criteria for LCS/MB is the same as the CCV/CCB criteria.

19.2 EPA 9060A soil is a duplicate replicate method. Quadruplicate replicate analysis may be performed upon client request. MDL studies are complete for both duplicate and quadruplicate sample analysis.

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. QC Recoveries low:

21.1.1 Check to make sure you are using the correct calibration and not an older one or using the wrong matrix calibration.

21.1.2 Look for leaks in the system according to manufacturer guidelines.

21.1.3 Look at tube with drierite to see if a plug in present or excessive discoloring.

21.1.4 Reanalyze the QC to check for potential miss-load or made incorrectly.

21.2. QC Recoveries high:

21.2.1 Look at analytical run for the presence of sample that may have contaminated the system, which required cleaning the system.

21.3 Follow manufacturer guidelines for other issue in the instrument manual or call OI for telephone support.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. Use caution when removing sample cups from the 1030 soil module. The cups are heated to 900^{o}C, and may still be hot after the sample analysis is finished.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-VM-S-001, Waste Handling.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Standard Methods for the Examination of Water and Wastes – Method 5310 C, 2011.

25.2. 40CFR Part 136 National Institute of Standards and Technology (NIST) O.I. Analytical TOC analyzer user manual.

25.3. Pace Quality Manual - Pace Analytical Services, Inc., current revision.

25.4. NELAC Standard, most current version.

25.5. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" – most current version.

25.6. USEPA Test Methods for Evaluating Solid Wastes, SW 846, 3rd Edition, Methods 9060A, Nov 2004.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-VM-I-019-rev.09	Table 7.0 - Changed Soils to Solids Section 12.2.2 - Changed sample weight from 0.0002g to 0.002g. Section 12.4.1 - Removed cup maintenance macro procedure. Table 13.2 - Changed frequency of Soil MS/MSD from 1 per 20 samples to 1 per 10 samples. Section 25 - Added year to EPA 9060A Method reference.	12Oct2016

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STANDARD OPERATING PROCEDURE

AMMONIA NITROGEN BY SEMI-AUTOMATED COLORIMETRY

Reference Methods: EPA 350.1 Rev. 2 1993

LOCAL SOP NUMBER:	S-VM-I-015-Rev.07
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-VM-I-015-Rev.06

APPROVALS

Laboratory General Manager — Date: 15 December 2016

Laboratory Quality Manager (for Kathleen Mitchell) — Date: 15 DECEMBER 2016

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace-Virginia, MN to determine the concentration of Ammonia by EPA Method 350.1.

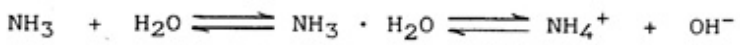
2. SUMMARY OF METHOD

2.1. The sample is buffered at a pH of 9.5 with borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is distilled into a solution of sulfuric acid. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue. The blue color formed is intensified with sodium nitroferricyanide.

2.2. The absorbance of the reaction product is measured at 630nm, and is directly proportional to the original ammonia concentration.

2.3. The NH3 as N result in mg/l can be used in the calculation for Organic Nitrogen. See TKN SOP (S-VM-I-016 most recent version).

2.4. The concentration of unionized ammonia is a function of pH and temperature. When ammonia dissolves in water, most of the ammonia reacts with the water to form ammonium ions (NH_4^+). A chemical equilibrium is established which contains un-ionized ammonia (NH^3), ionized ammonia (NH_4^+), and hydroxide ions (OH^-). The following equation expresses the equilibrium of these chemical species.



3. SCOPE AND APPLICATION

3.1. This automated method covers the determination of ammonia in non potable water for the clean water program, (CWP) ,the National Pollution Discharge Elimination System (NPDES), and Unionized Ammonia Calculation,

4. APPLICABLE MATRICES

4.1. This SOP is applicable to water matrix and has been modified for ammonia analysis of soils.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

5.2. Water: The applicable range is 0.1-20 mg/L NH_3 as N. Dilutions will extend the range.

5.3. Soil: The applicable range is 3.0-600 mg/Kg NH_3 as N. Dilutions will extend the range.

6. INTERFERENCES

6.1. Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.

6.2. Known Residual Chlorine must be removed by pretreatment of the sample with sodium thiosulfate or other reagents before distillation.

6.3. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line via the buffer to prevent these problems.

6.4. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Distillation removes most interference.

6.5. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response. Take care to prevent contamination by cleaning glassware well with 10 percent HCl following with 3x DI H_2O rinse. Ammonia in the lab atmosphere is a known source of contamination: seal samples, standards, and reagents well; sulfuric acid readily absorbs ammonia.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Table 7.1 Sample Collection, Preservation and Storage

Sample Type	Collection per Sample	Preservation	Storage	Hold Time
Aqueous	Water samples should be collected in polyethylene or glass bottles	Preserve water samples with H_2SO_4 to a pH <2	Above freezing ≤6°C Ammonia can be absorbed from the surrounding environment; prevent samples from contact with excess ambient air. Take care all samples are stored away from ammonia sources.	Max hold time is 28 days
Soil	4 oz soil jar		Above freezing ≤6°C Ammonia can be absorbed from the surrounding environment; prevent samples from contact with excess ambient air. Take care all samples are stored away from ammonia sources.	Max hold time is 28 days

7.1 NPDES discharge samples, and samples with known interferences are required to be distilled unless a distillation study and MDH approval is granted to not distill. If distilled samples are not analyzed the same day, seal distillation tube with parafilm. All Wisconsin samples require distillation.

7.2 Samples collected and delivered to the laboratory with the following problems: insufficient sample volume and improper container type or preservation; will involve notification to the client for immediate correction. Samples in compromised containers, and samples over the hold time will not be analyzed; the client will be required to provide a new sample. Most non-conformances are addressed during the sample receiving process.

8. DEFINITIONS

8.1. Definitions of terms found in this SOP can also be found in the Pace Quality Manual. When definitions are not consistent with TNI defined terms, an explanation is provided in this SOP or the Pace Analytical Services' Quality Manual Glossary.

8.2. Un-ionized Ammonia - NH_3. Ionized Ammonia – NH_4^+

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

Table 9.1: Instrumentation and Supplies

Equipment	Vendor	Model / Version	Description / Comments
Lachat	Zellweger Analytics	Quikchem FIA+8000 Series	With colorimetric detector, wavelength filter 630 nm
Lachat Reagent Pump	Zellweger Analytics	RP-150 Series	
Auto sampler	Cetac	ASX-500 Model No. 510	
Auto dilutor	Zellweger Analytics	8000 Series	
Tubing and peristaltic pump	Zellweger Analytics		Or equivalent vendor
Computer Software	Omnion	Version 2.0	
Hardware	Midwest Comp Depot	3035	
Analytical Balance	Sartorius, or equivalent vendor		Accuracy 0.001 g

Table 9.2: Glassware and additional supplies

Supplies	Description	Vendor / Item #
Safety glasses		Fisher, or equivalent vendor
Gloves	Latex or vinyl	Fisher, or equivalent vendor
Autopipettors	Checked for quarterly accuracy. Pipette tips Are checked per lot when received at the laboratory. 2-10 mL, 0.2-1.0 mL	Eppendorf, or equivalent replacement
Volumetrics	Class A	Fisher, or equivalent vendor
Helium	Compressed gas used for degassing DI water	
Tube Rack	Holds Distillation Tubes	Lachat/17012
Micro Dist® Tubes	Distillation Tubes	Lachat/A17117a (Kit #)
Membrane Filter Cap	Membranes and caps for sealing samples	Lachat/A17117a (Kit #)
Micro Dist® distillation Block	Distillation Block	Lachat/A17102
Equipment Press	Used for capping sample tubes (distillation)	Lachat/17023
Residual Chlorine Strips		Fisher or equivalent

10. REAGENTS AND STANDARDS

Table 10.1: Reagents, Intermediate and Working Solutions

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
DI Water	Aerated with helium	Resistivity =/>18 Megohm/cm
Micro Distillation: Sodium Borate Buffer	Dissolve 9.50g sodium tetraborate decahydrate in about 500mL reagent water. Add 22g NaOH. Dilute to 1L and mix well	Fisher, or equivalent vendor Room temperature for 3 months.
Distillation Stock: 0.1M Sulfuric Acid(H_2SO_4)	Add 11.1 ml H_2SO_4 in about 900mL reagent water. Dilute to 1L and mix.	Fisher, or equivalent vendor Room temperature for 6 months.
Distillation: 0.016M Sulfuric Acid Trapping Solution	Add 80ml 0.1M H_2SO_4 to 50mL reagent water. Dilute to 500ml, mix.	Fisher, or equivalent vendor Room temperature for 3 months.
Distillation: 2%H_2SO_4 Add to prepared standards etc., to match sample matrix:0.2% final conc.	Add 2ml of conc. H_2SO_4 to 90ml reagent water, dilute to 100ml, mix.	Fisher or equivalent vendor Prepare fresh day of use.
Sodium Nitroferricyanide **Prepare Weekly**	Dissolve 3.5g sodium nitroferricyanide in about 800mL reagent water. Dilute to 1L.	Fisher or equivalent vendor Room temperature Prepare weekly
Both Distilled/UN: Sodium Hypochlorite Solution **Prepare Fresh**	Add 250 ml bleach (purchased unaltered-5.25% Sodium Hypochlorite) to 500mL flask. Dilute to 500mL with reagent water. Invert to mix.	Local store Prepare fresh daily
Sodium Phenolate **Prepare Fresh 3-5days** Discard when dark brown	Dissolve 88mL 88% liquefied phenol and 32g NaOH in 600mL reagent water. Dilute to 1L	Fisher or equivalent vendor Room temperature Prepare fresh 3-5 days
NH_3 Buffer	Dissolve 50g Na_2EDTA and 5.5g NaOH in about 900mL reagent water. Dilute to 1L, mix.	Fisher or equivalent vendor Room temperature 1 month
Distilled: Carrier Solution 0.003M Sulfuric Acid	Add 30 mL 0.1M H_2SO_4 to~900mL reagent water. Dilute to 1L, mix.	Fisher or equivalent vendor Room temperature 1 month
UN Distilled Carrier degas DI	DI H20 aerated with helium	
Sodium Thiosulfate for dechlorination of sample.	Dissolve 3.5 g in DI water and dulute to 1L	Fisher or equivalent

Table 10.2: Standards, Storage Conditions

Standard Type	Description	Expiration	Storage
Calibration Stock Standard Solution	▪ 1000 mg/L, Hach Cat#23541-53 or equivalent	▪ Specified by manufacturer.	▪ Specified by Manufacturer.
Calibration Working Standard - undistilled (WS 1)	Prepare a working standard of 100 mg/L by diluting 1 mL of calibration stock from above with 9 mL DI Water Aerated with helium for a final volume of 10 mL.	▪ Prepared on day of use.	NA Make fresh for each day of use.
Second Source Stock Standard	Purchased standard that is a second source from the calibration stock 100 mg/L. NC Labs A-37A or equivalent	▪ Specified by manufacturer.	▪ Specified by Manufacturer.
QC/Secondary Working Standard (ICV1 /LCS) (Distilled)	Prepared by diluting 1 mL of the secondary stock standard at 100 mg/L from above with 8 mL reagent water and 1 ml of 2% H_2SO_4 solution for a final volume of 10 mL and 10 mg/L concentration.	▪ Prepared day of use.	Make fresh for each LCS used in run.
(ICV2) (Distilled)	Prepared by diluting 0.5 mL of the secondary stock standard at 100 mg/L from above with 9.5 mL reagent water for a final volume of 10 mL and 5 mg/L concentration.	▪ Prepared day of use.	Make fresh for each LCS used in run.
QC/Secondary Working Standard (ICV/LCS) (Undistilled)	Prepare by diluting 0.2 mL of the secondary stock standard at 100 mg/L from above with 9.8 mL reagent water for a final volume of 10 mL and a final concentration of 2 mg/L	▪ Prepared day of use.	Make fresh for each LCS used in run.

10.3. Use current calibration standards for analytical method – Prepare each standard as specified in table 10.3 (undistilled) or 10.4 (distilled) using 1000ppm stock standard, use degassed DI preserved to dilute.

Table 10.3: Calibration Standards for Undistilled analysis

Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7
4 mg/L	**2 mg/L - CCV**	**1.0 mg/L**	**0.5 mg/L**	**0.1 mg/L**	**0.05 mg/L**	**Blank**
0.8mL of 100 mg/l (WS 1)	0.8 std (cup 1) (WS 1) also CCV check	5.0 mL of 4 mg/l std (cup 1)	2.5 mL of 4 mg/l std (cup 1)	2mL of 1 mg/l std (cup 3)	1mL of 1mg/l std (cup 3)	none
19.2 mLs DI water	39.2 mL DI water	15 mls DI water	17.5mL DI water	18mL DI water	19mL of DI water (RLV*)	DI water

10.4. Final 2% H_2SO_4 solution ~2ml/L (0.2%) to match calibration standards to samples. (See table 10.4).

Table 10.4: Calibration Standards for Distilled analysis.

Cup 1	Cup 2	Cup 3	Cup 4	Cup 5	Cup 6	Cup 7	Cup 8
20 mg/L Std	**10 mg/L** (CCV)	**5.0 mg/L** (CCV)	**2.5 mg/L**	**1.0 mg/L**	**0.5 mg/L**	**0.1 mg/L**	**Blank**
1ml of 1000 calibration stock std.	0.5ml of 1000 calibration stock std.	0.25ml of 1000 calibration stock std.	6.25ml of 20 mg/L Std (cup 1)	2.5 ml of 20 mg/l Std (cup 1)	2.5ml of 10 mg/l Std (cup 2)	5.0 ml of 1.0 mg/l Std (cup 4)	NONE
5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4	5ml 2% H_2SO_4
44.0 ml aerated DI water	44.5 ml aerated DI water	44.75 ml aerated DI water	38.75ml aerated DI water	42.5ml aerated DI water	42.5ml aerated DI water	40.0ml aerated DI water	45ml aerated DI water

10.5. Prepare 1 MB and 1LCS per sample batch (max of 20 samples), these must include final H_2SO_4 concentration of 0.2%.

11. CALIBRATION AND STANDARDIZATION

Table 11.1.Calibration and Criteria.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve	For Undistilled 1st Order Distilled 2nd order Calibrate Daily	Curve Fit $r \geq 0.995$	If any of the criteria are not met, terminate analysis, fix the problem and recalibrate.
Calibration Check Verification (ICV/CCV) second source. Need to analyze two levels for 2nd order curve.	After Calibration,10% samples analyzed	90-110% % Recovery based on expected values	If criteria are not met, terminate analysis, fix the problem. One additional repetition may be conducted. If it fails, recalibrate prior to any sample analysis. All samples bracketed by the failing ICV or CCV must be reanalyzed following passing QC.

Calibration Check Blank(CCB/RB)	After Calibration,10% samples analyzed	< Reporting Limit Direct read	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x blank detects report the data as it is not impacted by the blank detections; If sample result <10x blank detects and cannot be reprepared/reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Reporting Limit Verification (CRDL)	After Calibration	60-140% % Recovery based on expected values	If the criteria is not met, review the data for the quality objectives. If the next point in the calibration passes criteria and raising the RL meets the client specifications, the RL may be adjusted in LIMS, otherwise recalibrate the instrument.

11.1. Calibration standards (table 10.3 for Undistilled and 10.4 for Distilled) should be analyzed creating a new calibration curve each time an ammonia analysis is run.

11.2. Lachat Quikchem software will construct and display the calibration curve and calculate all sample results. The calibration curve can also be prepared by plotting instrument response (optical density or absorbance) against standard concentration. Calculate sample concentration by comparing the sample response with the standard curve.

11.3. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed. Multiply calculated sample concentrations by appropriate dilution factor used.

11.4. The diluent bottle for this method is distilled blank solution (see section 10.4 for blank preparation).

11.5. Use the appropriate number of standards covering the desired analytical range. A curve should be made up as defined in table 10.3 (Undistilled) or 10.4 (Distilled) with the calibration standards.

12. PROCEDURE

12.1. Discharge/effluent samples under CWA (NPDES) and samples with known interferents, like leachates, high color require distillation prior to analysis. See also sample handling. If Distillation is not required, proceed to Sample Analysis.

12.2. For the items below the final distillate is collected in the user fill tubes and are ready to be analyzed:

12.2.1. QC Requirements: Per batch, Maximum of 20 samples, 1 Method blank; 1 LCS (2nd source); 1 MS/MSD per 10 samples.

12.2.1.1. Liquid Method Blank**:** 9 mL DI water, 1 mL 2% H_2SO_4. MB made up separately for each one needed.

12.2.1.2. Soil Method Blank: weigh out 0.20 Teflon boiling chips weighed to the nearest 100th on a calibrated balance and 1.0 mL of the Borate Buffer. Bring volume to 6 mL with DI water.

12.2.1.3. Liquid: Quality control standard (Distilled), (LCS): Using 100 mg/L second source stock standard: 1.0 mL of l standard, 1.0 mL of 2% H_2SO_4 to 8.0 mL DI water for a final concentration of 10 mg/L. Distill 6 mL.

12.2.1.4. Soil: prepare one LCS by weighing out 0.20 Teflon boiling chips weighed to the nearest 100th on a calibrated balance and 1.0 mL of the Borate Buffer. Add 1.0 mL of the 100 mg/L standard and 9.0 mL DI to create a 10 mg/L standard. Add 6.0 mL of the 10 mg/L solution to the digestion vessel containing the 0.2g boiling chips.

12.2.1.5. Liquid: Matrix spikes, (MS), and Matrix spike duplicates, (MSD):

Distilled - Using 9.9 mL of sample add 0.1 mL of 1000 mg/l stock solution for a final spike concentration of 10 mg/L- distill 6 mL.

12.2.1.6. Soil Matrix spikes, (MS), and Matrix spike duplicates, (MSD):

Distilled – Create an MS/MSD set by weighing out 0.20 g of sample to the nearest 100th on a calibrated balance and place into a distillation vessel and 1.0 mL of Borate Buffer. Thoroughly homogenize the sample prior to taking an aliquot of sample. Do not target or select small sample particles. For further details on sample homogenization S-MN-L-147 Sub-sampling Sample Homogenization. Repeat this step for the MSD.

Prepare 20 mL of a 10 mg/L spiking solution by adding 0.2 mL of a 1000 mg/L stock solution and adding 19.8 mL DI water. Add 6 mL of this solution to each the MS and MSD sample.

12.2.1.7 Liquid Samples: Shake the sample container to homogenize the sample. Test for residual chlorine using a residual chlorine test strip. If the sample contains residual chlorine, remove by adding Sodium Thiosulfate (Table 10.1) One mL will remove 1 mg/L in a 500 mL sample.
Measure out 6 mL of sample into the digestion vessel for distilled samples.

12.2.1.8 Soil Samples: Thoroughly homogenize the sample prior to taking an aliquot of sample. Do not target or select small sample particles. For further details on sample homogenization S-MN-L-147 Sub-sampling

Sample Homogenization. Weigh out 0.20 g of sample to the nearest 100th place on a calibrated balance and place into a distillation vessel. Add 1.0 mL Borate Buffer. Bring to a volume of 6 mL with DI water.

12.3. Distillation Procedure

12.3.1. User should fill distillation tubes (table 9.2) with the M side up: Add 1.0mL of 0.016M H2SO4 trapping solution to each tube. Put the membrane filter and cap on M end. Repeat until 1 tube per distillation (max of 21 per distillation set) is ready. Place in large tube rack.

12.3.2. In smaller tube rack add the sample tubes. Add 6.0 mL of each required standard, blank, sample. Include appropriate QC (MB, LCS, MS, and MSD) at the required frequency.

12.3.3. Add minimum volume of borate buffer solution, not to exceed 1.0 mL, to obtain a pH of 9.5. Confirm (in a separate cup or by dispensing a drop on a pH strip) that the borate buffer solution when added to 6mL of a standard / sample will adjust the pH to 9.5. Transfer pH to the prep log.

12.3.4. Using the press, hold the tube in the middle, put in press and move lever so it clamps the sample tube to the filler tube up to the ring on the sample tube. Repeat for all samples.

12.3.5. Place all tubes for this set onto the distillation block.

12.3.6. Set the timer for 30min. The volatilized NH3 is trapped in the tube above the filter.

12.3.7. When the timer is complete, put on heat resistant gloves, and take the first sample off of the distiller. Within 4 sec, pull off the bottom sample tube (move back and forth) and discard in a bucket. WAITING TOO LONG will destroy the sample; the distillate will go back into the sample tube. Place tube in large tube rack.

12.3.8. Repeat step 12.3 (distillation procedure) for each sample. Let cool 10 min.

12.3.9. Rotate tubes to collect all fluid from the sides. Turn tube upside down, and flick any solution in D side so goes into the M side of the tube.

12.3.10. Break off the D side and discard.

12.3.11. Dilute sample up to the 6 ml mark on tubes with DI water. Cap the top of the tube until analysis. If not analyzing w/in an hour, wrap parafilm around sealed top.

12.3.12. Repeat steps for next distillation set(s). If samples are not analyzed the same day, cap end and parafilm both ends.

12.3.13. Analyze samples, using the 0.003M H2SO4 carrier solution (see above).

12.3.14. Evaluate analysis for accuracy and precision requirements, see Sample Analysis

12.4. Undistilled Ammonia:

12.4.1. QC Requirements: Per batch, maximum of 20 samples: 1 Method Blank, 1 LCS (second source), 1 MS/MSD per 10 samples.

12.4.1.1. Liquid Method Blank: 10 mL DI H2O.

12.4.1.2. LCS: Prepared from 100 mg/L second source stock standard: 0.2 mL of 100 mg/L std, 9.8 mL DI H2O for a final concentration of 2.0 mg/L.

12.4.1.3. Matrix spikes (MS) and Matrix spike duplicates (MSD): For each, use 9.8 mL sample and 0.2 mL of 100 mg/L working standard for a final concentration of 2.0 mg/L MS and MSD.

12.4.1.4. Liquid samples: Pour a 10 mL sample aliquot for analysis.

12.5. Sample analysis:

12.5.1. Instrument Set Up: Turn on instrument. Log on to computer. Set up manifold on channel 1 with (20.5 cm x 0.8 mm i.d.) sample loop, (200 cm x 0.5 mm i.d.) backpressure loop, 630 nm interference filter, and 650 cm heater set to 70^{o}C. Pump degassed DI water through all reagent lines, check for leaks/smooth flow. Switch lines (in order) to:

12.5.1.1. Undistilled Carrier: Degassed distilled water
tubing: gray/gray i.d.0.051mm.
Distilled 0.003M H2SO4 Carrier tubing: gray/gray i.d. 0.051mm.

12.5.1.2. Buffer: NH3 buffer -tubing: red/red i.d. 0.045mm.

12.5.1.3. Sodium hypochlorite Undistilled (250/500). Sodium hypochlorite Distilled (250/500): white/white

12.5.1.4. Sodium Nitroferricyanide -tubing orange/orange i.d. 0.035 mm.

12.5.1.5. Sodium phenolate -tubing: red/red i.d. 0.045 mm.

12.5.1.6. Allow all reagents to pump for *at least* 5 minutes to achieve a steady baseline and for the system to equilibrate.

12.5.2. Open Applicable Method file ~ for undistilled samples: (unh14.met.); For Micro distilled samples: (unh14.met).

12.5.3. Calibration Fit Type: 1st Order Polynomial.

12.5.4. Cal Fit Type: Replace.

12.5.5. Weighting Method: None.

12.5.6. Concentration Scaling: None.

12.5.7. Force Through Zero: No.

12.5.8. Concentration Units: mg/L.

12.5.9. Inject to Peak Start: 30 s.

12.5.10. Peak Base Width: 30.979.

12.5.11. % Width Tolerance: 100%.

12.5.12. Threshold: 10,000.

12.5.13. Chemistry: Direct.

12.5.14. **Clear previous calibration curve**: review, fit, clear.

12.5.15. Undistilled: Open Tray file, (unh14.tra), Method file, (unh14.met), and DQM file, (unh14.dqm): or applicable files.

12.5.16. Distilled Open Tray file, (nh514.tra), Method file, (nh514.met), and DQM file, (nh514.dqm): or applicable files.

12.5.17. Enter sample numbers in tray. Program in CCV, CCB, RLV, MB, LCS, MS, MSDs IDs. Undistilled can be calc after analysis, there is no Data Quality Management (DQM) requirements programmed in. Save tray with date and analysis ID. Data quality management (DQM) for distilled Information: CCV= 10mg/L, QC (LCS) = 10mg/L, CRDL = 0.1mg/L; MS final concentration = 10mg/L.

12.5.18. Place calibration standards in Auto sampler locations. Pour out samples from plastic cups into sample tray. Begin calibration: *run tray*. Enter a result file ID (year, month, day, number Lachat run for the day). Click ok, save all files.

12.5.19. Once the calibration is completed, the curve is reviewed for standard RSD values (recommended <10%) and correlation coefficient >/= 0.995. If curve passes the criteria continue with analysis.

12.5.20. Automatic calibration verification per Lachat software - Data Quality Management (DQM).

12.5.20.1. The reporting limit verification standard of 0.1 mg/L, cup 7 (distilled) and cup 5 (undistilled) will automatically be checked at the beginning of every run. Recovery must be within 60-140%.

12.5.20.2. A mid-range standard will automatically be analyzed and the recovery must be within 90-110%. This will also serve as the continuing check standard automatically analyzed after every 10 samples. See Section 11.

12.5.20.3. The calibration blank, in cup 8 will automatically be analyzed and the results must be <RL. This will automatically be checked every 10 samples.

12.6. Unionized ammonia may be determined by utilizing the attached chart from Emmerson, et al. (1975), or by utilizing the website (http://aquanic.org/images/tools/ammonia.htm).The formulas used for the calculations were supplied by Dr. Ted Batterson, Michigan State University. These calculations were last updated on April 14, 2009. Use the temperature of the sample taken at the time of sampling.

13. QUALITY CONTROL

13.1 Table 13.1.Qualty Control and Criteria

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or blank solid for soil samples	One per 10 samples	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

Laboratory Control Sample (LCS)	DI water spiked with all target compounds or blank solid for soil samples	1 per sample run prior to analysis of samples.	% Recovery based on expected values. 90-110% or qualify data	Reanalyze a the LCS; If problem persists, check spike solution; Perform system maintenance prior to reanalysis LCS run ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	10 % of samples	Liquid 90-110% % Recovery	If LCS and MBs are acceptable, the MS/MSD data should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigated and document the cause in the associated narrative as well as qualifying properly.
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	10% of samples	RPD 10%	Report results with an appropriate footnote.

14. DATA ANALYSIS AND CALCULATIONS

14.1. **Liquid -** Raw Data value (mg/L) X Dilution Factor = NH3 as N (mg/L)

14.2. **Soil -** $\frac{\text{Raw Data Value (mg/L) X 0.006 L X Dilution Factor}}{(0.02 \text{ X \% solids})}$ = NH3 (mg/Kg)

14.3. The following calculation can be used to calculate the LCS percent recovery:

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. See table in section 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. See table in section 13.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. METHOD PERFORMANCE

18.1. **Method Detection Limit (MDL) Study** – An MDL study must be conducted per S-MN-Q-269, Method Detection Limit Studies, or equivalent replacement for each matrix per instrument

18.2. **Demonstration of Capability (DOC) –** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Training Procedures, or equivalent replacement.

18.3. **Periodic performance evaluation (PE)** – samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, Performance Evaluation (PE)/Proficiency Testing (PT) Program or equivalent replacement. Results are stored in the QA office.

18.4. The analyst must read and understand this procedure with documentation maintained in his/her training file.

19. METHOD MODIFICATIONS

19.1. This method was modified to include the analysis of ammonia in soils.

19.2. The distillation procedure and reagents used in the SOP follows the procedure developed by the Micro Distillation unit manufacturer. See User Manual reference in Section 25.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. TROUBLESHOOTING

21.1. See Section 13.

22. SAFETY

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a

sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

23.1. The Environmental Protection Agency (USEPA) requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult SOP S-VM-S-001, Waste Handling, or equivalent replacement.

23.2. The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

24. POLLUTION PREVENTION

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

25.1. U.S. EPA, Determination of Ammonia Nitrogen by Semi-automated Colorimetry, Method 350.1 rev 2 1993.

25.2. QuickChem Method 10-107-06-1-X, Determination of Ammonia by Flow Infection Analysis (Low Flow Micro Dist ® Sample Preparation), written by Amy Huberty/Lachat Applications Group. September 8, 2008

25.3. Standard Methods Online, SM4500NH3-B, 2011, Preliminary Distillation Step.

25.4. Lachat Instruments QuikChem Methods Manual

25.5. Lachat Instruments QuikChem 8000 Automated Ion Analyzer FIA Training Manual.

25.6. Lachat Instruments FIA Software

25.7. Lachat Instruments FIA Hardware Installation and System Operation Manual.

25.8. Lachat Instruments QuikChem Method 10-210-00-1-B, Revised December 2000.

25.9. Micro Dist ® Operation and Applications User Manual Oct 2007 Ed. 3.

25.10 Pace Quality Manual - Pace Analytical Services, Inc., current revision.

25.11 NELAC Standard, most current version.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1 Attachment I – Canadian Water Quality Guidelines for the Protection of Aquatic Life. Unionized ammonia calculation chart.

26.2 Attachment II – Prep Log example.

27. REVISIONS

Document Number	Reason for Change	Date
S-VM-I-015-rev.07	Changed 'Pace Analytical Inc.' to 'Pace Analytical LLC.' Section 25.3 - Updated Standard Method Reference year to 2011. Table 9.1 - Changed wavelength to 630 nm. Table 10.3 - Changed RLV to CRDL. Table 10.4 - Added 5 mg/L CCV Section 12.3 - Added Borate buffer solution addition and pH confirmation procedure. Section 12.4 - Added Undistilled Ammonia sample and QC prep Section 12.5.17 - Added 5 mg/L CCV, changed RLV to CRDL. Section 12.5.20.1 - Changed location of cup for CRDL and Blank. Section 19.2 - Added Distillation Instrument Manual reference.	12/09/16

Attachment I – Unionized Calculation Chart
Canadian Water Quality Guidelines for the Protection of Aquatic Life (page 3)

AMMONIA **Canadian Water Quality Guidelines for the Protection of Aquatic Life**

EQUATION 1.

$$pKa = 0.0901821 + 2729.92 / T$$

Where,

T = Temperature in K; Absolute zero = - 273.15 °C

T (in K) = T (in °C) + 273.15

EQUATION 2.

$$f = 1 / [10^{(pKa - pH)} + 1]$$

Where,

f = fraction of total ammonia that is un-ionized
pKa = dissociation constant from equation 1

Using the equations above, a table describing the percent of NH_3 in low ionic strength water for temperatures (0 – 30°C) and pH (pH= 6 – 10) is presented (Table 3). The ionic strength of the water is also an important influence on the un-ionized ammonia concentration. As the ionic strength increases in hard or marine waters, there is a decrease in the un-ionized NH_3 concentration (Environment Canada 1997; Emerson et al. 1975). Freshwater systems with up to 200 - 300 mg· L^{-1} total dissolved solids may have a negligible reduction in percent NH_3. The effect of ionic strength is much smaller than the effects of temperature and pH (Soderberg and Meade 1991).

Table 3. Percent un-ionized aqueous ammonia solutions for 0-30°C and pH 6-10 (Emerson et al. 1975)

Temp (°C)	pH								
	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10
0	0.008	0.026	0.082	0.261	0.820	2.55	7.64	20.7	45.3
5	0.012	0.039	0.125	0.394	1.23	3.80	11.1	28.3	55.6
10	0.018	0.058	0.186	0.586	1.83	5.56	15.7	37.1	65.1
15	0.027	0.086	0.273	0.859	2.67	7.97	21.5	46.4	73.3
20	0.039	0.125	0.396	1.24	3.82	11.2	28.4	55.7	79.9
25	0.056	0.180	0.566	1.77	5.38	15.3	36.3	64.3	85.1
30	0.080	0.254	0.799	2.48	7.46	20.3	44.6	71.8	89.0

In surface waters, both nitrification and volatilization are important competitive fate processes for ammonia (Environment Canada 1999). Volatilization increases with increasing wind speed, temperature, and pH. In addition, the partial pressure of ammonia in solution increases with increasing pH, and in aqueous solutions, ammonia may form complexes with a number of metal ions. It may be sorbed onto suspended and bed sediments and to colloidal particles. Ammonia may also be exchanged between sediments and overlying water. Ammonia concentrations in water vary seasonally and regionally. In natural waters, concentrations of total ammonia are generally less than 0.1 mg·L^{-1}. Higher levels of ammonia are generally indicative of organic pollution (McNeely et al. 1979 as cited in WQB 1989).

The data on Canadian environmental concentrations presented below have been selected from a database prepared by Environment Canada in support of the Second Canadian Environmental Protection Act Priority Substances List (CEPA PSL II) assessment for ammonia (Environment Canada 1998). Detection limits were often not reported in the database.

From 1993 to 1996, concentrations of dissolved ammonia in twenty rivers sampled in the Northwest Territories ranged from 0.0002 to 0.294 mg·L^{-1} (n=521), with an average concentration of 0.0148 mg·L^{-1}. Total ammonia sampled from two rivers in the Northwest Territories waters ranged from 0.002 to 0.19 mg·L^{-1} (n=4) (Environment Canada 1998).

Concentrations of dissolved ammonia collected from 165 rivers and lakes across British Columbia between 1990 and 1996 ranged from ND (not detected) to 180 mg·L^{-1} (n=5135), with an average concentration of 0.689 mg·L^{-1}. The maximum concentration was detected in the Fraser River at a hydro station south of Mission City in 1993. Total ammonia levels from 32 water bodies in British Columbia ranged from ND to 8.4 mg·L^{-1} (n=2129), with an average concentration of 0.0858 mg·L^{-1} (Environment Canada 1998). Similarly, concentrations of total ammonia sampled from 232 waterbodies in the province of Alberta between 1990 and 1996 ranged between ND and 10.2 mg·L^{-1} (n=2599), with an average concentration of 0.183 mg· L^{-1} (Environment Canada 1998). Dissolved ammonia concentrations from 414 rivers and lakes in Alberta ranged between ND and 8.8 mg·L^{-1} (n=1929), with an average concentration of 0.110 mg·L^{-1} (Environment Canada 1998).

In 1987, total ammonia concentrations in the South Saskatchewan River, 140 m below the outfall diffuser of Saskatoon's sewage treatment plant outfall, reached a maximum of 4.26 mg·L^{-1} (WQB 1989). For five kilometres downstream of the sewage treatment plant outfall, a few of the transects had mean total ammonia concentrations within the effluent plume that surpassed the Saskatchewan Surface Water Quality Objective for total ammonia for the protection of aquatic life of 0.44 mg·L^{-1} (WQB 1989).

Attachment II – Prep Log

Batch Information: WETA NH3 23251 8/9/13 **Template Version: F-VM-I-044-Rev.01**

Prep Method:	EPA 350.1	Analysis Method:	EPA 350.1	Extracted By:	JLB	Extracted By Date:	08/09/2013 12:15:05.643
SNNa2B2Sol3:	3700	Batch Notes:		Reviewed By:		Reviewed By Date:	

Sample Information:

QC Rule	Sample Type	Lab Sample ID	Matrix	Initial Volume (mL)	Initial Weight (g)	H2SO4 Solution (mL)	Borate Buffer (mL)	Block ID	Final Volume (mL)	pH	Sample Notes	NH3-LCS (mL)	NH3-SRK (mL)
3501 W_P	BLANK	**88078**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	LCS	**88079**	Water	10	10	20198 ()	18556 ()		10	9.5		19306 (1)	
3501 W_P	PS	**1223663002**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	MS	**88080**	Water	10	10	20198 ()	18556 ()		10	9.5			13561 (.1)
3501 W_P	MSD	**88081**	Water	10	10	20198 ()	18556 ()		10	9.5			13561 (.1)
3501 W_P	PS	**1223661004**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223543004**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223543005**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223660002**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223480002**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223481001**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1222999001**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223528001**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223457001**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223661005**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	MS	**88082**	Water	10	10	20198 ()	18556 ()		10	9.5			13561 (.1)
3501 W_P	MSD	**88083**	Water	10	10	20198 ()	18556 ()		10	9.5			13561 (.1)
3501 W_P	PS	**1223533001**	Water	1	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223481003**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223483001**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223481002**	Water	10	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223491001**	Water	1	10	20198 ()	18556 ()		10	9.5			
3501 W_P	PS	**1223546002**	Water	10	10	20198 ()	18556 ()		10	9.5			

Fri, 9 Aug 2013 12:16:14 -0500

STANDARD OPERATING PROCEDURE

Analysis of Samples for Gross Alpha and Gross Beta Content

Methods: EPA 900.0, EPA 9310, ASTM D1890-90, SM 7110C

SOP NUMBER:	S-PGH-R-001-rev.19
REVIEW:	R. Kinney
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	PGH-R-001-18
REVIEW DATE:	Upon Procedural Change

APPROVALS

_____ Department Manager/Supervisor	02/08/18 Date
_____ Senior Quality Manager	02/08/18 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date

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Table of Contents

1. Purpose

1.1 This SOP documents the analytical procedure for gross alpha and gross beta content in samples by EPA 900.0, EPA 9310, ASTM D1890-90 and SM 7110C.

2. Scope and Application

2.1 This procedure is used to rapidly screen a variety of matrices for both high and low activities of alpha and beta emitting radionuclides. The purpose of this method is two-fold: 1) to provide adequate information concerning the activity within samples, and thus determine if further, more detailed analyses are required, and 2) to support accountability that radioactive material license limits are not exceeded.

2.2 Gross screening analyses are not expected to be as accurate nor as precise as more detailed radiochemical separations. Rather, they are intended to provide rapid information associated with a particular action level with minimal chemical preparation. Additionally, these types of analyses are not intended to give "absolute" activity measurements, but rather "order-of-magnitude" estimates.

2.3 This method is applicable to the analysis of gross alpha and gross beta in drinking water, wastewater, soil, sludge, air filter, contamination smears, oil, and organic matrices. Without qualification, this procedure, as written, is compliant with Method 900.0 of "*Prescribed Procedures for Measurement of Radioactivity in Drinking Water,* EPA-600/4-80-032", Method 9310 of "*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW846), Volume 1C, Third Edition", and Method 7110C of "*Standard Methods for the Examination of Water and Wastewater*".

2.4 Waters with no visible suspended solids and low total dissolved solids (<500 ppm) may be processed as received without further preparation by the evaporation method.

2.5 Waters with high total dissolved solids (>500 ppm) may be processed for gross alpha using the specified co-precipitation method to achieve adequate limits of detection. Samples with suspended solids must be filtered prior to preparation and analysis of gross alpha by the SM7110C co-precipitation method. The evaporative method can still be used for the gross beta determination.

2.6 Waters with suspended solids may be filtered. The solids may then be discarded, counted directly or the filter may be digested and analyzed at the customer's request.

2.7 Filters and smears are direct counted, by attaching the filter or smear to a planchet.

2.8 Solids and oils are placed directly on the counting planchet and affixed with PMMA before counting.

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2.9 Samples which do not fall into any of these categories are digested then analyzed as an aqueous sample.

2.10 Pace Analytical services, LLC. (PASI) applies isotope decay correction only in instances where the total impact in the analysis result is 2% or greater. Assuming a maximum hold time of 180 days, a 2% isotope decay would occur only for radioisotopes with a half-life of 17.14 years or less. The parameters reported using this SOP are not affected by this policy. Because gross alpha and gross beta measurements do not determine specific radioisotopes, it is impossible to apply decay correction factors. Decay correction is not utilized for gross alpha and/or gross beta analysis.

3. Summary of Method

3.1 Aqueous samples (including drinking waters, waste waters, and other miscellaneous aqueous samples) requiring “total” gross alpha and gross beta analysis are evaporated to near dryness. Concentrated nitric acid is added to each concentrated sample and evaporated to near dryness to remove chloride. The concentrated sample (in HNO_3) is transferred to a stainless steel counting planchet and dried under a heat lamp.

3.2 Aqueous samples requiring “dissolved” analysis are filtered through a 0.45μm membrane filter and analyzed as for “total” analysis as summarized in Section 3.1 of this SOP.

3.3 Aqueous samples requiring analysis for “suspended” solids are filtered through a 0.45μm membrane filter. The resulting solids are dried and analyzed directly for gross alpha and gross beta content or are dissolved and analyzed.

3.4 A portion of dried sludge or soil sample that has been pulverized to less than 200-mesh and homogenized is transferred to a stainless steel counting planchet. 1N Nitric acid solution is added to the solids and the samples are evaporated to dryness under a heat lamp.

3.5 A small portion of organic sample is slowly evaporated to dryness under a heat lamp or on a hot plate. Residual solids are treated with 16M HNO_3 and peroxide solution to reduce organic content. Resulting solids are transferred to a stainless steel counting planchet and evaporated to dryness under a heat lamp.

3.6 Small portions of oil samples are smeared onto the planchet and left under the heat lamp to dry.

3.7 Air filter samples may be counted directly for gross alpha and gross beta content or dissolved, with a portion of the resulting digestate analyzed as for “total” analysis as summarized in Section 3.1 of this SOP.

3.8 Contamination smear samples are mounted onto a stainless steel counting planchet and counted directly for gross alpha and beta content.

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3.9 Samples residues (excluding directly mounted filters or smears) that exhibit fluctuations in mass following drying may be heated to a dull red heat to convert hygroscopic (water-absorbing) salts to oxides that are less hygroscopic.

3.10 Aqueous samples with high total dissolved solids content (>500 ppm) may be analyzed for gross alpha using a co-precipitation technique to achieve adequate detection limits. Alpha emitting elements contained in the sample are co-precipitated with stable barium (as sulfate) and with stable iron (as hydroxide). The resulting precipitate mixture is filtered through a 0.45µm nitrocellulose filter, stored for three hours to allow decay of radon progeny and counted directly for gross alpha content.

3.11 All prepared samples are analyzed in a gas flow proportional counting system for gross alpha and/or gross beta content.

3.12 Solid samples which cannot be direct plated due to the matrix, such as filter socks, rubber mats, fiber cord, etc., will be aliquotted into a large ceramic crucible and ashed overnight at 550°C. The remaining solids are transferred to a PTFE beaker and completely dissolved using nitric, hydrochloric, and hydrofluoric acids. Remnant hydrochloric and hydrofluoric acids will be eliminated through repeated sample evaporation with nitric acid. The final residue will be dissolved in a measured volume of 0.1 N HNO_3, and subjected to gross alpha and gross beta determination as an aqueous solution.

4. Interferences

4.1 High hygroscopic salt content in evaporated samples can cause the sample mass to fluctuate due to moisture absorption. To minimize this interference, the salts are converted to oxides by heating the sample until it glows with a characteristic dull-red color.

4.2 Excessive dissolved solids in any aqueous sample or extract will affect the detection limit for gross alpha and gross beta, as the residual mass cannot practically exceed 100 mg for gross alpha analysis and 200 mg for gross beta analysis. In such cases, sample count times can be extended to, 1000 minutes, or the maximum practical count time not to exceed 2000 minutes.

4.3 Non-uniformity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

4.4 Radionuclides that are volatile under the sample preparation conditions of this method will not be measured. They may include, but are not limited to nuclides of: polonium, cesium, carbon, iodine and hydrogen.

4.5 Low energy beta emitters that are unable to be measured by this method due to limitations of the instrumentation are Pu-241, Fe-55, C-14 and H-3.

4.6 When counting alpha and beta particle activity by a gas flow proportional counting system, counting at the alpha plateau

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discriminates against beta particle activity, whereas counting at the beta plateau is sensitive to alpha particle activity present in the sample. This latter effect should be determined and compensated for during the calibration of the specific instrument being used.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel. Since this analysis is for a radioactive constituent, the sample must be treated as radioactive. .

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves will be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of the department manager or the department manager's specified designee. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 Hydrofluoric acid (HF) is particularly hazardous because a serious skin exposure may cause no immediate sensation of pain. The acid penetrates the skin and spreads internally, causing tissue damage deep under the skin. The resulting burn is painful, difficult to treat, and easily infected. Gloves must be checked for pinhole leaks before use. They must be rinsed before they are removed and must be discarded after use. HF burn gel shall be put on suspected HF burns after flushing (except the eyes) until medical help can be obtained. Medical attention shall be sought even if suspicions arise after working hours. Contact the group leader immediately for further information if a HF burn is suspected.

5.8 In addition, HF vapors are also hazardous. Exposure can cause permanent damage. Breathing HF vapors even for a short time and at a low temperature can be injurious to the respiratory system and even fatal. All such direct contact must be avoided.

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5.9 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory.

6. Definitions

6.1 Batch: For all analysis, an analytical batch contains 20 or fewer samples of similar matrix prepared at the same time, by the same analyst, using the same reagents. For batches containing AZ drinking water samples, a sample duplicate must be analyzed at a frequency of 10%.

6.2 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to mass exceeding 1gor volumes in excess of 1mL. Measurements of mass and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below one gram or one milliliter, the word "approximately" must be used prior to the described weight or volume.

6.3 Throughout this procedure, exact or critical mass and volumes will be designated by the use of one or more decimal places. Measurements of mass and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc.

6.4 When aliquotting samples on a balance, the observed weight on the balance must be recorded in preparation logbooks to the lowest weight indicated on the balance. Sample aliquot weights must not be targeted. Once sample is removed from the sample container and transferred to a beaker, it must not be removed from the beaker.

6.5 The method utilized for obtaining the sample aliquot, whether on a balance, in a graduated cylinder, or by pipette, must be clearly annotated in the preparation logbook.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

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7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Aqueous samples

8.1.1 Containers used for sample collection must never be re-used. Either plastic or glass containers may be used for sample collection.

8.1.2 Aqueous samples must be preserved at the time of collection by adding enough concentrated (16M) HNO_3 to the sample to make the sample pH <2. Typically, 2mL 16M HNO_3 per liter of sample is sufficient to obtain the desired pH. Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 24 hours, and the pH must be rechecked by laboratory personnel prior to removing sample for analysis. The pH recheck date and time, the initials of the analyst verifying the pH, as well as any adjustments or notes regarding the preservation must be recorded in the pH Verification Logbook.

8.1.2.1 For dissolved analysis, samples must be filtered through a 0.45μm membrane filter and preserved to a pH <2.

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8.1.2.2 For total analysis, the sample has not been filtered, but has been preserved.

8.1.3 Refrigeration is not required for aqueous samples.

8.2 Soil, sludge, air filter or organic samples

8.2.1 Con[t]ainers used for sample collection must never be re-used. Either plastic or glass bottles or plastic bags may be used for sample collection.

8.2.2 Preservation is not required for soil, sludge, air filter, or organic matrices.

8.2.3 Refrigeration is not required for soil, sludge, air filter, or organic matrices.

8.3 The maximum hold time for samples analyzed by this procedure is 180 days between sample collection and sample analysis.

9. Equipment and Supplies

9.1 Gas Flow Proportional Counting System. (Low background beta <3 cpm). Refer to SOP PGH-R-002, current revision "Gas Flow Proportional Counter Operation" for instructions on GFPC system operation.

9.2 Electric hot plate or electric griddle.

9.3 Centrifuge.

9.4 Membrane filters, 0.45μm, 47mm Metricel or equivalent.

9.5 Heat lamp or drying oven set at 105°C.

9.6 Glassware, various sizes.

9.7 Stainless steel counting planchets. 2 inch diameter by either 1/8 inch or ¼ inch deep.

9.8 Analytical balance.

9.9 Top loader balance capable of weighing 1.00g to 500.00g.

9.10 Plastic petri dishes for storage of sample planchets.

9.11 Desiccator.

9.12 Software supplied with the instrument to control instrument operation. Refer to SOP PGH-R-002, current revision "Gas Flow Proportional Counter Operation" for applicable software details.

9.13 Computer capable of running the Gas Flow Proportional Counter System software, monitor, mouse, keyboard, and printer. Refer to SOP PGH-R-002, current revision "Gas Flow Proportional Counter Operation" for computer hardware specifications.

9.14 TSW Wide Twill Smears, FRHAM Safety Products, item FS812.

10. Reagents and Standards

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10.1 Reagents should be prepared from reagent grade chemicals, unless otherwise specified below. Reagent water must be at least ASTM Type II quality or better. NOTE: Consult the Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Distilled or deionized (DI) water. ASTM Type II as produced using the specifications documented in SOP PGH-C-027, current revision.

10.3 Acetone, ACS reagent, $(CH_3)_2CO$, anhydrous.

10.4 Aluminum Chloride Hexahydrate, 4 mg/mL: Dissolve 1.0 g of $AlCl_3 \cdot 6H_2O$ ASTM Type II DI water, and dilute to 250 mL with ASTM Type II DI water.

10.5 Ammonium hydroxide, 6N: Dilute 400mL conc. NH_4OH to 1L DI water (ASTM Type II).

10.6 ATP (Alternate Test Procedure) Solution: Combine 13 mL each of the following solutions; 4 mg/mL Aluminum Chloride Hexahydrate, 4 mg/mL Barium Chloride Dihydrate, 40 mg/mL Calcium Nitrate Tetrahydrate, 4 mg/mL Iron Chloride, 100 mg/mL Magnesium Sulfate Hepathydrate, 80 mg/mL Sodium Bicarbonate, 14 mg/mL Sodium Phospahte Dibasic, and 60 mg/mL Sodium Sulfate, in 600 mL of ASTM Type II DI water. Acidify the solution with 2 mL of concentrated nitric acid, and dilute the solution to 1.0L with ASTM Type II DI water.

10.7 Barium carrier, 10mg/mL: Dissolve 8.8g $BaCl_2 \cdot 2H_2O$ in 500mL DI water (ASTM Type II).

10.8 Barium Chloride Dihydrate, 4 mg/mL: Dissolve 1.0 g of $BaCl_2 \cdot 2H_2O$ in ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.9 Bromocresol purple, 0.1%: dissolve 100mg of the water-soluble reagent in 100 mL DI water (ASTM Type II).

10.10 Calcium hydrate 4-hydrate: ACS grade.

10.11 Calcium Nitrate Tetrahydrate, 40 mg/mL: Dissolve 10.0 g of $Ca(NO_3)_2 \cdot 4H_2O$ ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.12 Cellulose powder/paper pulp/water mixture. Add 1.0g cellulose powder or paper pulp to 1.0L of DI water (ASTM Type II) plus 10 drops of diluted (1:4) detergent. Shake and stir vigorously prior to use.

10.13 Detergent, diluted 1 to 4 with DI water (ASTM Type II). Alconox® or equivalent.

10.14 Ludox, SM-30® colloidal silica solution. Dilute with DI to obtain a dissolved concentration of 2mg/mL-evaporated solids.

10.15 Ferric nitrate nona-hydrate: $(Fe(NO_3)_3 \cdot 9H_2O)$, ACS grade.

10.16 Hydrochloric acid, 12N, concentrated, ACS reagent grade.

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10.17 Hydrofluoric acid, 29N, concentrated, Sp. Gr. 1.18, 49%. Must be stored in a plastic container.

10.18 Hydrogen peroxide, 30%.

10.19 Iron carrier, 10mg/mL: Dissolve 35.0g $Fe(NO_3)_3 \bullet 9H_2O$ in 300mL of DI water (ASTM Type II), add 2mL 16N HNO_3, and dilute to 500mL with DI water (ASTM Type II).

10.20 Iron (III) Chloride, 4 mg/mL: Dissolve 1.0 g of $FeCL_3$ in ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.21 Magnesium nitrate hexa-hydrate, $(Mg(NO_3)_2 \cdot 6H_2O)$, ACS grade.

10.22 Manganese (II) nitrate $(Mn(NO_3)_2)$, ACS grade.

10.23 Magnesium Sulfate Heptahydrate, 100 mg/mL: Dissolve 25 g of $MgSO_4 \bullet 7H_2O$ in ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.24 Nitric Acid, 16N, concentrated, Sp. Gr. 1.42, 70.4%.

10.25 Nitric Acid 12N: Carefully add 750mL concentrated 16N HNO_3 to 200mL DI water (ASTM Type II), cool thoroughly and dilute to 1L with DI water (ASTM Type II).

10.26 Nitric Acid 8N: Carefully add 500mL concentrated 16N HNO_3 to 300mL ASTM Type II DI water, cool and dilute to 1L with ASTM Type II DI water.

10.27 Nitric Acid 1N: Carefully add 62.5mL of concentrated HNO_3 (16N) to 800mL of deionized water. Cool and dilute to 1Lwith deionized water.

10.28 Nitric Acid, 6N: Carefully add 375mL concentrated 16N HNO_3 to 500mL DI water (ASTM Type II), cool and dilute to 1L with DI water (ASTM Type II).

10.29 Nitric Acid, 0.1N: Add 100mL of 1N HNO_3 to 500mL DI water (ASTM Type II) and dilute to 1L with DI water (ASTM Type II).

10.30 Polymethylmethacrylate (PMMA, Acrylic), 0.2%: Dissolve 1g of PMMA in 500mL acetone.

10.31 Sodium Bicarbonate, 80 mg/mL: Dissolve 20 g of sodium bicarbonate in ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.32 Sodium nitrate:$(NaNO_3)$, ACS grade.

10.33 Sodium Phosphate Dibasic Anhydrous, 14 mg/mL: Dissolve 3.5 g of Na_2HPO_4 in ASTM Type II DI water and dilute to 250 mL with ASTM Type II DI water.

10.34 Sodium Sulfate Anhydrous, 60 mg/mL: Dissolve 15 g of $NaSO_4$ in ASTM Type II DI water, and dilute to 250 mL with ASTM Type II DI water.

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10.35 Sulfuric acid, 1N: Dilute 55mL concentrated H_2SO_4 to 1L with DI water (ASTM Type II).

10.36 Radioactivity standard solutions, various. Alpha solutions of Th-230 or Natural Uranium, and beta solutions of Sr-90 and Cs-137 may be utilized for batch control spike samples or instrument calibrations. All radioactive standards must be NIST traceable. Sources used for calibration purposes must be diluted in HNO_3 solution. Alternate radioactive solutions may be used for calibration purposes if specifically requested by the client (i.e. Am-241 for gross alpha), however alternate calibration solutions may not be used in the analysis of drinking water.

11. Calibration

Alpha and beta radioactivity emissions are inhibited by the medium through which they must travel. For this reason, when counting radioactivity emissions by gas flow proportional counting, system efficiency decreases as sample residue thickness increases. Because sample analysis using this SOP generates sources with significantly variable residue mass, an appropriate system calibration that corrects for this “self-absorption” characteristic must be performed.

Note: Calibration sources for gross alpha and gross beta measurement by Gas Flow Proportional Counting (GFPC) are prepared to match prepared sample sources of various matrices to the best extent possible. Calibration sources are prepared by mimicking the sample preparation technique for each matrix.

There are four sample source preparation techniques covered by this SOP: Evaporation Technique, direct count of contamination smears and air filters, affixation of pulverized solid sources to a counting planchet, and the specified co-precipitation technique.

Due to the lack of recognized solid standard reference materials for gross alpha and gross beta content, the laboratory utilizes the calibration curves generated for the evaporation technique for the analysis of prepared solid samples.

Additionally, the calibration curves generated for the evaporation technique are used as the base calibration for all matrices for which the specified sample preparation technique is an evaporative technique including; drinking waters, non-potable water (dissolved and total), evaporation of digestates (dissolved filters, etc.), digested organic matrices, and dissolved solids such as soils.

11.1 Gross Alpha and Gross Beta Efficiency Calibration (Evaporation Technique)

11.1.1 Tap water, concentrated through prolonged evaporation with nitric acid, is the suggested calibration solution specified in the EPA drinking water method EPA 900.0. However, because samples may be received from various parts of the country, it is recognized that the mineral make-up of the laboratory’s tap water may not be consistent with samples received. Preparation of a salt solution as described in Section 11.1.2.1 or 11.1.2.2 is allowed. If the

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concentrated tap water solution is utilized, it must be assessed for alpha and beta contributions which would affect the calibration calculations.

11.1.2 The salt matrix used for calibration purposes must be reasonably consistent with matrix components that are routinely encountered with the sample matrices to be analyzed. The salt matrix must be chloride-free and preserved in HNO_3. The salt matrix must be analyzed for radioactive components and be determined to be radioactivity free. There are two options available. The recipe for the ATP solution is from the EPA Protocol for the Evaluation of Alternate Test Procedures. The other salt matrix recipe is utilized by standards providers. Both solutions exclude the use of any form of potassium due to the presence of K-40.

11.1.2.1 ATP solution is prepared by combining 13 mL each of the following solutions; 4 mg/mL Aluminum Chloride Hexahydrate, 4 mg/mL Barium Chloride Dihydrate, 40 mg/mL Calcium Nitrate Tetrahydrate, 4 mg/mL Iron Chloride, 100 mg/mL Magnesium Sulfate Hepathydrate, 80 mg/mL Sodium Bicarbonate, 14 mg/mL Sodium Phospahte Dibasic, and 60 mg/mL Sodium Sulfate, in 600 mL of ASTM Type II DI water. Acidify the solution with 2 mL of concentrated nitric acid, and dilute the solution to 1.0L with ASTM Type II DI water.

11.1.2.2 Salt Matrix Recipe- The salt matrix recipe given below is designed to achieve a target cation ratio of 8 for magnesium, 20 for calcium, 50 for sodium, 1 for iron, and 4 for manganese. Dissolve 2.65g of magnesium nitrate hexa-hydrate, 6.10g calcium nitrate tetra-hydrate, 5.49g sodium nitrate, 0.522g ferric nitrate nona-hydrate, and 1.30g manganese (II) nitrate in 500mL of ASTM Type II water. Dilute to 1.0L using ASTM Type II water.

11.1.3 Calibration curves generated by this method are applicable to samples that generate residues within the range of the calibration curve. For drinking water analysis, sample residues must be within the calibration range. Analysis results for non-DW matrices that generate residue content outside of the calibration range may be reportable with the approval of the client. Results of this type must be reported with appropriate qualification.

11.1.4 For calibration curves for gross alpha by the evaporative method, Pace requires a minimum of eight calibration sources with approximately equivalent distribution of solid mass over the expected range of samples to be analyzed. For example, an appropriate alpha calibration curve with an expected range of 0mg up to 100 mg may include eight or more sources (with masses

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distributed over the range) of approximately 0 mg, 15 mg, 30 mg, 40 mg, 55 mg, 70 mg, 90 mg, and 100 mg. Actual masses will vary from calibration to calibration due to the matrix used to create the sources. Calibration curves for gross beta by the EPA 900.0 method require a minimum of five calibration sources with approximately equivalent distribution of solid mass over the expected range of samples to be analyzed.

11.1.5 Determine the dissolved solids content of the calibration salt solution by adding 5mL of salt solution to a tared stainless steel counting planchet. Evaporate the solution to dryness under a heat lamp, flame the source to a dull red heat, cool completely, and transfer to a desiccator, and re-weigh to determine the residue salt concentration of the salt solution.

11.1.6 Determine the appropriate volumes of salt solution to be used to generate sources that meet the criteria established in Section 11.1.4 of this SOP. Transfer appropriate volumes of salt solution to labeled glass beakers. Add 5mL of concentrated HNO_3 to each sample. Add 1 mL of Ludox solution to each calibration sample.

11.1.7 Evaporate each calibration source to near dryness. Perform step 12.1.8 of this SOP as for samples. Upon completion of sample transfer and drying, reweigh each source to determine whether the calibration source mass meets the criteria established in section 11.1.4 of this SOP.

11.1.8 Place each calibration source under a heat lamp and add 3 mL 8N HNO_3 solution to redissolve source residue. Evaporate each source to dryness. For alpha calibration, transfer between 500 and 5000 dpm (by mass) of an appropriate alpha standard solution to each calibration planchet. For beta calibration, transfer between 500 and 2000 dpm (by mass) of an appropriate beta standard solution to each calibration planchet.

Note 1: The standard solutions used for calibration purposes are Th-230 or natural uranium for gross alpha and Sr/Y-90 or Cs-137 for gross beta. Alternate radioactive solutions may be used for calibration purposes if specifically requested by the client (i.e. Am-241 for gross alpha), however, alternate calibration solutions may not be used in the analysis of drinking water.

Note 2: Alpha calibration efficiencies for the specified mass range typically fall between 5% for the high mass standard and 25% for the zero mass standard. Standard activity for each calibration planchet should be optimized to allow consistent count times to acquire the required 10,000 net counts. Low mass standards typically require approximately 1000 dpm and high mass standards may utilize as much as 5000 dpm to allow for consistent count times.

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Note 3: Beta calibration efficiencies for the specified mass range typically do not deviate more than 10% between the low and high mass standards. Equivalent amounts of beta standard should be used for all beta calibration sources.

Note 4: The specified calibration source activities have been optimized to allow manageable count times for individual sources. Maximum calibration source activities have been set to minimize the potential impact of any cross contamination within the detector system.

11.1.9 Add 5 mL 8N HNO_3 solution to each calibration source and evaporate under a heat lamp to redistribute calibration source residue. Repeat this step until source residue is evenly distributed across the planchet.

11.1.10 Flame the calibration sources to a dull red heat. Cool the sources completely.

11.1.11 Transfer dried sources to a desiccator to cool. Reweigh sources to determine source mass.

11.1.12 Count each source in the detector being calibrated until a minimum of 10,000 net counts has been acquired for the radioactivity type being determined.

11.1.13 Perform efficiency calculations as detailed in Attachment 1 of this SOP.

11.1.14 Plot the system efficiency (as cpm/dpm) versus source mass (in mg) using MS Excel. Utilize the MS Excel least squares curve-fitting program to determine the best curve to fit against the measured data.

11.2 Gross Alpha Cross talk Calibration (Evaporation Technique)

11.2.1 When counting alpha and beta particle activity by a gas flow proportional counting system, counting at the alpha plateau discriminates against beta particle activity, whereas counting at the beta plateau is sensitive to alpha particle activity present in the sample. This latter effect should be determined and compensated for during the calibration of the specific instrument being used.

11.2.2 Alpha cross talk calibration must be performed concurrent to the gross alpha efficiency calibration process using the data generated during alpha efficiency source counting. Cross talk calibration factors are determined by calculating the percent-observed beta count rate (as cpm) versus observed alpha count rate (as cpm) for each calibration source. Refer to Attachment 1 of this SOP for cross talk calibration calculations.

11.2.3 Plot the system alpha cross talk factor versus source mass (in mg) using MS Excel. Utilize the MS Excel least squares curve-fitting program to determine the best curve to fit against the measured data.

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11.3 Gross Alpha Calibration (Co-precipitation Technique)

11.3.1 Calibration curves for gross alpha by the co-precipitation technique require a minimum of four calibration sources with approximately equivalent distribution of solid mass over the expected range of samples to be analyzed. For example, an appropriate alpha co-precipitation calibration curve with an expected range of 20 mg up to 70 mg may include four or more sources (with masses distributed over the range) of approximately 20 mg, 35 mg, 50 mg, and 70 mg. Actual weights will vary from calibration to calibration due to the matrix used to create the sources.

11.3.2 To disposable centrifuge tubes add the following variable volumes of iron carrier solution (10 mg/mL) and barium carrier solution (10 mg/mL):

Calibration Source Number	Iron carrier (10 mg/mL)	Ba carrier (10 mg/mL)
1	0.3	0.3
2	0.5	0.5
3	0.75	0.75
4	1.0	1.0

11.3.3 Add 2 mL of 6N HNO_3 and 20 mL DI water (ASTM Type II) to each calibration source.

11.3.4 Add approximately 500 dpm of alpha solution to each calibration source. Add 5 drops of diluted detergent solution to each calibration solution.

11.3.5 Perform steps 12.5.4 through 12.5.10 of this SOP as for sample analysis.

11.3.6 Count each source in the detector being calibrated until a minimum of 10,000 net counts has been acquired for the radioactivity type being determined.

11.3.7 Perform efficiency calculations as detailed in Attachment 1 of this SOP.

11.3.8 Plot the system efficiency (as cpm/dpm) versus source mass (in mg) using MS Excel. Utilize the MS Excel least squares curve-fitting program to determine the best curve to fit against the measured data.

11.4 Gross Alpha Efficiency Calibration (Direct Count of Contamination Wipes or Air Filters)

11.4.1 Affix a TSW Wide Twill Smear onto a 2-inch 1/8-inch deep stainless steel planchet using the sticky back siding of the smear. Label the planchet with the appropriate information documenting the calibration source ID.

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11.4.2 Place the Planchet/Smear on an analytical balance and tare the balance. Transfer a quantity of Th-230 aqueous Standard Reference Material (SRM) onto the surface of the smear. Transfer the SRM around the entire interior region of the smear to obtain a surface contamination distribution that represents the contamination survey process. Record the mass of the SRM added.

11.4.3 Carefully remove the planchet/smear from the balance and transfer to a location for open-air drying of the SRM material.

11.4.4 Count each source in the detector being calibrated until a minimum of 10,000 net counts has been acquired for the radioactivity type being determined.

11.4.5 Perform efficiency calculations as detailed in Attachment 1 of this SOP.

11.5 Gross Alpha Cross talk Calibration (Direct Count Technique)

11.5.1 Alpha cross talk calibration must be performed concurrent to the gross alpha efficiency calibration process using the data generated during alpha efficiency source counting. Cross talk calibration factors are determined by calculating the percent-observed beta count rate (as cpm) versus observed alpha count rate (as cpm) for each calibration source. Refer to Attachment 1 of this SOP for cross talk calibration calculations.

11.5.2 Plot the system alpha cross talk factor versus source mass (in mg) using MS Excel. Calculate the detector cross-talk factor using the single source.

11.6 Gross Beta Efficiency Calibration (Direct Count of Contamination Wipes or Air Filters)

11.6.1 Affix a TSW Wide Twill Smear onto a 2-inch 1/8-inch deep stainless steel planchet using the sticky back siding of the smear. Label the planchet with the appropriate information documenting the calibration source ID.

11.6.2 Place the Planchet/Smear on an analytical balance and tare the balance. Transfer a quantity of Sr-90 aqueous SRM onto the surface of the smear. Transfer the SRM around the entire interior region of the smear to obtain a surface contamination distribution that represents the contamination survey process. Record the mass of the SRM added.

11.6.3 Carefully remove the planchet/smear from the balance and transfer to a location for open-air drying of the SRM material.

11.6.4 Count each source in the detector being calibrated until a minimum of 10,000 net counts has been acquired for the radioactivity type being determined.

11.6.5 Perform efficiency calculations as detailed in Attachment 1 of this SOP.

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11.7 Calibration curve development process

11.7.1 Following regression analysis, measured pCi values for each calibration source must be calculated using the calibration curve. Each measured source should be within 10% of known. If the value is not within 10%, assess the point using a z-score. If the z-score for the point is greater than 2.56, the point must be removed from the calibration curve. It is not acceptable to remove the low-mass or high-mass point from use for calibration. If calculated z-scores indicate that removal of these points is required, the sources must be replaced with a newly-prepared source of similar mass. Calibration points may not be removed from the calibration curve without approval of the Department Manager/Supervisor. Following removal of individual points, the regression process must be repeated until the criteria established in this SOP have been met. The minimum number of calibration points for each calibration parameter is specified in Section 11.8 of this SOP. If the regression process culminates with fewer than the required number of data points specified in Section 11.8 of this SOP, formal corrective action must be applied and new sources must be prepared. A narrative discussing technical justification for modifications or exclusions of any calibration point must be created and kept with the calibration data.

11.7.1.1 The z-score for calibration point assessment is calculated as follows:

11.7.1.1.1 $$Z_{CalPt} = \frac{ABS(Y_1 - Y_2)}{J}$$

Where:

Y1 = The average difference of the calibration points from the calculation curve used

Y2 = The difference of the calibration point being assessed from the calculation curve used

J = The standard deviation of the calibration points from the calculation curve used

11.7.2 Curve data points must be removed from the population of points used for calibration if the calculated z-score is greater than 2.56.

11.7.3 Curve data points may be removed from the population of points used for calibration if the calculated difference from the curve for the point is greater than 10%.

11.7.4 Curve data points may be removed from the population of points used for calibration if visual inspection of the source indicates that the distribution of calibration material on the source is inconsistent. When such points are removed, the calibration narrative must

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include a photograph of the affected source. Additionally, concurrence between the calibration analyst and Department Manager/Supervisor must be documented in the calibration narrative.

11.8 Calibration curve acceptance criteria

Calibration curves generated by the process detailed in this SOP must meet the following minimum criteria to be used for sample analysis. It is required that calibration curves that do not meet the documented acceptance criteria not be placed into service. In this instance, calibration curve values must not be implemented into active spreadsheets used for the calculation of results. This approach prevents the un-intentional generation of results for samples counted on non-compliant detectors.

11.8.1 Instrument calibration must include the minimum number of calibration points specified for each alpha determining method.

11.8.1.1 For gross alpha evaporation technique efficiency calibrations, a minimum of 8 calibration points must be utilized in the final calibration curve.

11.8.1.2 For gross alpha crosstalk calibrations, a minimum of 6 calibration points must be used in the final calibration curve.

11.8.1.3 For gross alpha by the co-precipitation technique efficiency calibrations, a minimum of 4 calibration points must be utilized in the final calibration curve.

11.8.1.4 For gross beta evaporation technique efficiency calibrations, a minimum of 5 calibration points must be utilized in the final calibration curve.

11.8.1.5 For gross alpha efficiency, gross beta efficiency, and alpha crosstalk factor calibrations for the direct count method (swipes and air filters), a single prepared calibration source is used. One source is prepared for gross alpha efficiency and alpha cross-talk and a separate single source is used for gross beta calibration.

11.8.2 The default assessment device for acceptability of calibration curves generated by this SOP is the curve regression coefficient. The default acceptance criteria for curve acceptability is to achieve a regression co-efficient of 0.80 or higher.

11.8.3 For gross alpha crosstalk calibrations, source count uncertainties are relatively high due to the collection of fewer source counts. Sources are counted to acquire the minimum 10,000 net counts for alpha efficiency determination however, there are no requirements for the number of source beta counts that must be collected for determination of alpha crosstalk factors. For this reason, alpha

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crosstalk curves achieving a regression coefficient of less than 0.80 may be re-assessed to determine curve usability.

11.8.3.1 For gross alpha crosstalk calibrations that do not satisfy the default regression coefficient of 0.8, calibration curves are acceptable when: Each data point within the final calibration data set has an acceptable z-score as calculated as specified at 11.7.1.1 of this SOP.

11.8.3.2 Each data point within the final calibration data set is within 10% of the curve-calculated result.

11.8.3.3 The average of the absolute values of the calibration point difference from curve values is less than 7.5%.

11.8.4 Some calibration types may yield factors that are very close across the calibration range. This is often the case for gross beta efficiency versus mass. Gross beta calibration curves that do not meet the regression co-efficient requirement may be re-assessed using the following technique.

11.8.5 Calculate the relative standard deviation (RSD) between all of the points used for the calibration curve. If the calculated (RSD)of the population is less than 5%, the regression co-efficient requirement is waived. Narrate this occurrence in the applicable calibration narrative. The applicable curve generated from the calibration process is utilized to determine sample-specific efficiency factors. Calibration efficiencies and cross-talk factors determined using a single source must be verified by second-source comparison.

11.8.5.1 Prepare calibration verification sources in the same fashion as calibration sources were prepared. The calibration verification source may be a mixture of alpha and beta-emitting radionuclides.

11.8.5.2 Count the sources for a time sufficient to obtain a minimum of 500 net alpha and net beta counts.

11.8.5.3 Calculate the gross alpha and gross beta activity of each verification source using the detector alpha efficiency, beta efficiency, and alpha cross-talk factors.

11.8.5.4 For acceptable calibration verification, the calculated gross alpha and gross beta activities must be within 10% of known values.

11.8.6 Calibrations and all associated documentation must be reviewed and approved by either the department supervisor or QA prior to implementation.

11.9 Calibration Frequency

11.9.1 Calibrations or calibration reverification for tests associated with drinking water analyses must be performed on an annual basis.

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Because the laboratory uses drinking water calibration curves (Evaporative Technique) for other matrices including non-potable water (dissolved and total), evaporation of digestates (dissolved filters, etc.), digested organic matrices, and dissolved solids such as soils, the matrix calibrations are inherently performed or verified annually by association. Additionally, the annual calibration or calibration verification is required for sources associated with direct counting (contamination wipes/air filters) and the specified co-precipitation technique. As allowed by specifications within the Manual for the Certification of Laboratories Analyzing Drinking Water, calibration sources may be retained for calibration re-verification purposes. Calibration sources used for reverification purposes must be stored in a desiccator continuously when not in use by the laboratory.

NOTE: PASI has adopted this reference as the basis for all gross alpha and gross beta-related calibration verifications.

11.9.2 For calibration reverification, only the sources used for the initial calibration may be used. Prior to using calibration mass sources for reverification of calibration curves, sources must be reweighed to calculate the net mass of calibration material in the calibration sources. Newly measured source masses must be used for subsequent reverification calculations.

11.9.3 Calibration verification requires the counting of no fewer than three of the original calibration sources for the evaporative technique. For the co-precipitation technique, no fewer than two of the original calibration sources should be used for reverification purposes. For the direct count technique for contamination wipes and air filters, the single original calibration source is used for reverification purposes. When reverifying calibration curves generated using multiple sources, the sources counted for calibration reverification must span the range of the weights used for the initial calibration.

11.9.4 In order for the reverification counts to be acceptable, the calculated result +/- the 3-sigma count uncertainty must overlap the target activity.

11.9.5 Detectors for which reverification counts do not meet this criteria must be recalibrated following the requirements listed in this SOP.

12. Procedure

Unless specified otherwise, the documented analysis process must be followed, as written, including the order of analytical process and the addition of chemicals.

12.1 Analysis – Drinking Water and other aqueous samples (Dissolved or Total)

12.1.1 For each sample to be counted on a ~2-inch diameter planchet (20 cm^2) select a sample aliquot that contains no more than 150 mg

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residue for beta analysis and not more than 100mg residue for alpha analysis.

12.1.1.1 To determine the solids content of each sample, weigh a clean labeled stainless steel planchet. Record this tare weight in the logbook.

12.1.1.2 Pipette 5.0mL of the sample onto the planchet. Add 3mL 1N HNO_3 to each planchet and heat to dryness on a hotplate or under a heat lamp.

12.1.1.3 If the samples will require flaming (such as for groundwaters or visibly hygroscopic samples), flame the planchet.

12.1.1.4 Reweigh the planchet to determine the mass of solids in 5.0mL of sample. Calculate the quantity of sample to be used for analysis such that residual solids will be less than 100mg for alpha and less than 150mg for beta determination. For aqueous samples, do not use more than approximately 200 grams of sample for analysis.

12.1.1.5 Planchets are not to be reused for sample analysis and must be discarded in the appropriate solid waste container.

12.1.1.6 Alternatively, specific conductance on an unpreserved sample may be used as an indicator of the dissolved solid content of the sample. (Samples must be preserved prior to analysis in accordance with Section 8)

12.1.2 Weigh the appropriate aliquot of sample (based on the 5mL test) in an appropriately sized glass beaker. Record the observed mass of the sample added to the beaker (do not remove any sample from the beaker).

12.1.3 Prepare QC samples by weighing approximately 200g of ASTM Type II DI water into an appropriately sized beaker. Add the appropriate amounts of alpha and beta spiking solutions to each of the LCS/LCSD and MS/MSD in accordance with the requirements in Sections 14.7.3 and 14.10.3 of this SOP.

12.1.4 Add 1mL of diluted Ludox® solution to all samples and all QC samples. Add 10mL of the ATP solution to the LCS/LCSD only. Note: Chloride salts are significantly hygroscopic and will cause fluctuations in residue mass during sample counting. Additionally, chloride salts will cause the stainless steel planchet to corrode and cause erroneous sample residue results. For this reason, chloride salts are converted to nitrate salts that are less hygroscopic by adding concentrated nitric acid to the sample and evaporating to dryness.

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12.1.5 Add about 5mL of 16N HNO_3 to each sample and QC sample and allow them to evaporate to near dryness on a hotplate. Do NOT allow samples to go dry to avoid baking solids onto the beaker.

12.1.6 Add 5mL of concentrated HNO_3 to the evaporated sample. Evaporate to near dryness on a hotplate.

12.1.7 For samples known to contain high chloride content, repeat step 12.1.6 a total of three additional times.

12.1.8 Transfer the sample to a tared planchet with the aid of a rubber policeman or transfer pipette and 1N HNO_3 from a wash bottle. Thoroughly wet the beaker walls with a few drops of 1N HNO_3 and transfer the washings to the planchet using a rubber policeman or a transfer pipette. Rinse the beaker a minimum of two additional times with 1N HNO_3 and transfer the rinses to the planchet. Additional rinses may be utilized as necessary if visible residue remains in the beaker, until all residue is removed from the beaker. All subsequent transfers should be made after the initial transfers have dried in the planchet. It is critical to not overfill the sample planchets to eliminate the potential for sample loss during evaporation. All transfers to the planchet should be limited to an approximate 5mL per transfer between evaporations. Samples observed to spill during evaporation must be removed from the analytical batch and re-prepared in a new batch.

12.1.9 Evaporate the final solution on the planchet to dryness under heat lamps. Drinking water samples prepared in accordance with EPA method 900.0 must be dried in an oven set at 105°C for a minimum of 2 hours. Record the time when the samples are placed in the oven. Counting may begin 72 hours after this time. Allow samples to cool in a desiccator and record the final mass. Store in a desiccator until analysis.

12.1.10 Note: Some types of dissolved water solids, when converted to nitrate salts, are quite hygroscopic (water absorbing) even after being dried under a heat lamp. When such salts are present the sample may gain mass as a result of environmental conditions while waiting counting in an automatic counting system.

12.1.11 When there is evidence of hygroscopic salts in sample planchets, it is recommended that they be flamed to a dull red heat with an open flame burner to convert the nitrate salts to oxides before weighing and counting. Allow samples to cool and record the final mass. Store in a desiccator until instrument analysis. Record which samples were flamed in the preparation logbook.

12.1.12 Count prepared samples in a low-background gas flow proportional counting system as detailed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. Samples may be counted for gross alpha and gross beta radioactivity immediately after drying.

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12.1.12.1 Drinking water samples analyzed in accordance with EPA method 900.0 require a delay of 72 hours between sample evaporation and counting to allow time for equilibrium of radium-226 and daughters to occur. Pace uses the time the samples are placed in the drying oven as the start time when determining the end of 72 hour wait period.

12.1.13 Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.2 Analysis of Organic Matrices (Oils):

This section is applicable to all organic samples that can be evaporated to dryness by simple means of convection heating. Other samples, such as high boiling point organic matrices should be analyzed with clearly detailed instruction from the Department Manager/ Supervisor or the specified designee.

12.2.1 Transfer 0.1-10mL of sample directly into a tared planchet. Be sure to select a volume of sample that will generate a residue mass that is within the calibration range.

12.2.2 Place each sample under a heat lamp and evaporate slowly to dryness. If hygroscopic salts are suspected, heat the sample until it glows with a characteristic dull red color to stabilize the mass.

12.2.3 Weigh the dried planchet and determine the net residue mass. Store the samples in a desiccator until instrumental analysis is required.

12.2.4 Count samples in a low-background gas flow proportional counter as instructed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.3 Analysis of Air Filters or Contamination Smears by direct counting

12.3.1 Mount air filters or contamination smears directly on a planchet with the loaded face of the filter exposed. Chemical separations are not required for filters.

12.3.2 Count samples in a low-background gas flow proportional counter as instructed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.4 Analysis of Soil, Vegetation, Animal Matter, Coal or Coal Ash, and Other non-soil Solid samples:

12.4.1 Direct solid plating method:

12.4.1.1 The calibration curve for gross alpha and gross beta analysis brackets the range of approximately 0mg to

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100mg. The minimum quantity of solid sample that should be analyzed is 30 mg. For this method, the reaction between the nitric acid and solid sample may cause an increase in the final evaporated sample source mass on the analysis planchet. For this reason, the maximum allowable sample mass aliquotted should be 80 mg. Weigh 30-80mg of dried and homogenized sample onto a tared planchet (Enough to achieve the desired MDC, Add 5mL 1N HNO_3.

12.4.1.2 Evaporate to dryness under a hot lamp.

12.4.1.3 Add 1mL of 0.2% PMMA in acetone reagent, and evaporate to dryness under the infrared lamp two times. NOTE: It is important that the residue be evenly distributed over the inner surface of the planchet so that it may be counted accurately.

12.4.1.4 Transfer samples to a desiccator to cool. When samples have cooled, re-weigh to determine residue mass.

12.4.1.5 Count samples in a low-background gas flow proportional counter as instructed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.4.2 Complete dissolution method for solid samples and filters

12.4.2.1 Aliquot a representative portion of the sample into a large ceramic crucible. Typically, 1-5g of sample is used.

12.4.2.2 Cover the crucible with a ceramic crucible cover and place the sample in a muffle furnace set to ramp to a final temperature of 550°C. Allow the sample to ash overnight.

12.4.2.3 Turn off the muffle furnace, allow the crucible to cool completely, remove from the muffle oven, and transfer the sample solids to a clean, labeled PTFE beaker.

12.4.2.4 To each sample add 10mL each of nitric, hydrochloric, and hydrofluoric acids. Cover the Teflon beaker with a Teflon cover and reflux the samples for thirty minutes on an electric griddle set at 300C.

12.4.2.5 Remove the PTFE cover and allow the samples to evaporate to dryness on the hot plate.

12.4.2.6 Repeat steps 12.4.2.4 and 12.4.2.5 one additional time.

12.4.2.7 Add 10mL concentrated nitric acid to each sample and evaporate the sample to dryness on a hotplate.

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12.4.2.8 Repeat step 12.4.2.7 two additional times to remove residual HCl and HF.

12.4.2.9 Dissolve the sample residue in a measured volume of 0.1N HNO_3.

12.4.2.10 Proceed with the sample as if it were an aqueous sample, beginning with Step 12.1.1.1 of this SOP.

12.5 Analysis of aqueous samples for gross alpha by co-precipitation

The quantity of sample that can be utilized for gross alpha analysis using evaporation techniques is limited by the total dissolved solids (TDS) concentration of the sample. For samples with high TDS, extending count times to the practical limit of 1000 minutes may not significantly improve results. For this reason, an alternate co-precipitation procedure should be utilized for the analysis of high TDS samples (>500 mg/L) for gross alpha content.

Not all samples can be successfully analyzed by this alternate method. Samples with suspended solids must be filtered prior to commencing analysis by SM7110C. Samples with minerals which readily precipitate as either a hydroxide or a sulfate may result in final precipitate masses greater than the highest mass used in the calibration curve. Samples with a high precipitate residue (typically greater than 60mg) must be re-prepped using less sample. Consideration must be given to whether analyzing a sample for gross alpha by this method will result in statistically better data than that obtained using the EPA 900.0 method.

12.5.1 The default analysis mass by this method is 200 g for drinking water samples and 50 g for non-DW water sources. Weigh the pre-defined amount of sampleinto a labeled glass beaker. Record the observed mass of the sample added to the beaker (do not remove any sample from the beaker). If the sample quantity utilized for analysis is less than 200g, dilute to approximately 200g using DI water (ASTM Type II). Never remove sample after dilutions are performed. Acidify QC samples with 2mL concentrated nitric acid. Add the applicable amount of Th-230 spiking solution to the LCS/LCSD and MS/MSD samples as required in sections 14.7.3 and 14.10.3 respectively. Fortify all diluted samples with 2mL concentrated nitric acid to ensure a starting pH of <2.

12.5.2 Perform all chemical additions in the order specified in the following steps. Deviations from the steps may result in incomplete precipitation of the desired elements and may compromise the integrity of the final counting source.

12.5.3 Add 5 drops of diluted detergent and place the sample on a hot plate set to high.

12.5.4 While stirring, add 20mL of 1N sulfuric acid to the sample. Heat to a boil for 10 minutes. Reduce heat to simmer, continue stirring and add 0.5mL of barium carrier solution. Continue heating with periodic stirring for 30 minutes.

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12.5.5 Add 1mL of bromocresol purple indicator solution, 0.5mL of iron carrier and 5mL of the cellulose powder solution. Continue stirring and add 6N NH_4OH drop wise to the sample until there is a distinct color change (yellow to purple). Continue heating with periodic stirring for 30 minutes longer. Allow to cool completely

12.5.6 Filter the sample through a tared 47mm, 0.45 μm membrane filter, rinsing all the precipitate from the beaker to the filter using DI water (ASTM Type II). Wash the precipitate with 25mL of DI water.

12.5.7 Remove the filter and place it in its planchet. Place the planchet containing the filter into a drying oven set at 105°C. Leave the door ajar and periodically monitor the filters to ensure they do not heat to the point of possible ignition.

12.5.8 Remove the planchet containing the filter from the oven, cool slightly, and place each in a labeled petri dish to ensure the filters cannot get mixed up prior to being reweighed and glued to the planchet.

12.5.9 Reweigh each filter and record the masses in the preparation logbook. Carefully remove the filter from the planchet, apply stick glue to the planchet, and center the filter on the planchet. Ensure there are no raised edges on the filter.

12.5.10 Once dry and mounted, store the samples in a desiccator and allow 3 hours for the collected radon progeny to decay before counting.

12.5.11 Following the initial 3 hour decay requirement, count the samples as soon as possible to minimize the effect of radium-226 daughter ingrowths. Count samples in a low-background gas flow proportional counter as instructed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. . Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

13. Calculations

13.1 Refer to Attachment I of this SOP for gross alpha and/or gross beta associated calculations.

13.2 Any verified result for drinking water that exceeds the maximum contaminant level (MCL) established for Gross Alpha and/or Gross Beta (photon emitters) must be reported to the appropriate personnel and agencies according the specific requirements of the state where the water was sampled. The directions for reporting any results that exceed the MCL limits are documented in the State Drinking Water Emergency Reporting Requirements Binder and Pace SOP PGH-C-025, current revision.

13.2.1 Gross Alpha MCL>= 15pCi/L

13.2.2 Gross Beta/photon emitter MCL>= 4mrem/year

14. Quality Control

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14.1 General guidelines for drinking water samples with results that exceed the Maximum Contaminant Level include the following: (All steps are to be conducted as soon as the exceedence has been identified.)

14.1.1 Verify the result(s) to ensure that there were no transcription or calculation errors and that all QC results are within the acceptable limits. Correct any problems and determine the new result. If there were no errors or the result still exceeds the MCL, continue with the reporting process.

14.1.2 Immediately notify the Department Manager/Supervisor, and QA Department that a reportable result has been identified. Use telephone notifications to inform the contact people if the variance is identified after hours along with an e-mail follow up to document the event.

14.1.3 Refer to the State Drinking Water Emergency Reporting Requirements Binder for the state specific information regarding the proper course of action to take. Time is of the essence during this process with some of the state reporting requirements as short as 1 hour from the verification of an exceedence.

14.2 Each analyst who performs this test must satisfactorily complete a Demonstration of Capability Study as documented in Section 3.4 of the most recent revision of the Quality Assurance Manual.

14.2.1 The DOC study results are evaluated against the LCS acceptance limits.

14.3 Daily instrument Quality Control checks for Gas Flow Proportional Counting Systems must be completed following the instructions detailed in the gas flow proportional counter operations SOP PGH-R-002.

14.4 The LCS and matrix spike solutions must consist of the same nuclides as those used for calibration. The LCS and MS spiking solutions must come from a source other than that used for the calibration.

14.5 See Appendix III for performance indicator evaluation calculations and criteria. Numerical performance indicators may be used to assess QC for non-drinking water samples when the default assessment indicates a QC failure. The numerical performance indicator must be within +/- 3 for all other matrices. The z-score for precision assessment may be used for drinking waters with the approval of the Department Manager/Supervisor using the +/- 2 specification.

14.6 Method Blank (MB)

14.6.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, DI water (ASTM Type II) (Reagent Blank) must be carried through the procedure. A reagent blank may be used for sample correction purposes following

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approval of the Department Manager or the department manager/Supervisor's specified designee and affected clients.

14.6.2 The results of the method blank must be less than the reporting limit. The National Primary Interim Drinking Water Regulations (NIPDWR) require a gross alpha detection limit of 1.0 pCi/L for compliance with Part 141.15(a) and 3.0 pCi/L for compliance with Part 141.15(b) and a gross beta detection limit of 4.0 pCi/L.

14.6.2.1 If the method blank is out of control, individual sample results may still be reportable if results are less than the CRDL (contract required detection limit) or greater than 10 times the blank result. Relative sizes of the sample and blank aliquots must be factored when making this determination (raw counts).

14.6.2.2 Projects analyzed under the DOD QSM must evaluate the method blank to ½ the detection limit. Corrective Action must be performed for any MB that has a positive value greater than ½ the detection limit.

14.7 Laboratory Control Sample (LCS)

14.7.1 One LCS must be prepared for each analytical batch.

14.7.2 Typical detection limits are 3 pCi/L for alpha and 4 pCi/L for beta.

14.7.3 Both the alpha and beta spike solution activities must be between 2 and 10 times the detection limit.

14.7.4 A reference material containing a known concentration of alpha (e.g. Th-230, natural uranium, Am-241) or beta (e.g. Sr/Y-90 or Cs-137) radioactivity in the same matrix as the batch is analyzed with the batch.

14.7.4.1 If this material is not available, a well-characterized material (WCM) may be used.

14.7.4.2 If neither of these are available, DI water (ASTM Type II) may be spiked with the appropriate gross alpha and/or gross beta standard.

14.7.4.3 For drinking water analysis, the alpha standard must be either Th-230 or natural uranium, and the beta standard must be Sr/Y-90 in accordance with EPA method 900.0.

14.7.5 Percent Recovery Calculation

$$\%REC = \frac{(LCSConc)}{TrueValue} * 100$$

Where:

LCSConc = Analytical result of the LCS
TrueValue= Known concentration of the LCS

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14.7.6 LCS %REC acceptance limits are 69-121% for gross alpha and 79-130% for gross beta for the evaporative technique. LCS recovery limits are 46-100% for gross alpha and 75-132% for gross beta by direct plating technique. LCS recovery limits are 75-125% for gross alpha by SM7110C.

14.8 Laboratory Control Sample Duplicate (LCSD)

14.8.1 An LCSD must be analyzed for samples with low gross alpha and/or gross beta activity concentrations in order to comply with NELAC standards. A LCSD is not required for other gross alpha and/or gross beta analyses; however analysis of an LCSD must be utilized to measure batch precision whenever adequate sample quantity is not available for sample DUP analysis. The LCSD must be prepared in an identical fashion as the LCS and processed identically as for other samples.

14.8.2 The LCSD must pass the acceptance criteria established for the LCS recovery and the criteria established for duplicate precision.

14.9 Sample Duplicate (DUP)

14.9.1 One Duplicate Sample (DUP) must be randomly assigned within each batch. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at the time of sample collection. The sample DUP is a duplicate quantity of sample processed identically as other samples in the analytical batch.

14.9.1.1 Drinking water samples from the state of Arizona must be batched at a frequency of 1 duplicate for every 10 samples or fewer.

14.9.2 Relative Percent Difference Calculation

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

Where:

R1 = Result Sample 1
R2 = Result Sample 2

14.9.3 Duplicate sample RPD acceptance limits are <34% for gross alpha and <29% for gross beta for the evaporative and direct plating techniques. Duplicate RPD acceptance is <25 % for Gross Alpha by SM 7110C.

14.9.3.1 The DUP evaluation criteria for batches that include drinking water samples from the state of Arizona have an acceptance

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limit of <20% RPD or <2 RER for gross alpha and gross beta.

14.10 Sample Matrix Spikes (MS)

14.10.1 This analytical method does not require the use of carriers or radiotracers for yield determination therefore, a sample matrix spike (MS) is required for gross alpha and gross beta analysis.

14.10.2 Typical detection limits for gross alpha and gross beta are 3 and 4 pCi/L respectively.

14.10.3 The spike amount must be greater than 10 times the detection limit.

14.10.4 A matrix spike is prepared by spiking a portion of an appropriate alpha (Th-230, natural uranium, or Am-241) and beta (Sr/Y-90 or Cs-137) standard solution into one sample within the batch.

14.10.4.1 For drinking water analysis, the alpha standard must be either Th-230 or natural uranium and the beta standard must be Sr/Y-90 in accordance with EPA method 900.0.

14.10.5 Process the matrix spike sample identically with the other samples.

14.10.6 The purpose of the MS is to assess the effect of sample components on the analytical process.

14.10.7 The quantity of sample used for the MS must be equivalent to the quantity used for sample analysis.

14.10.8 Percent Recovery Calculation

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations.

14.10.9 MS acceptance limits are 55-135% for gross alpha and 79-130% for gross beta by the evaporative technique. MS acceptance limits are 75-125% for gross Alpha by SM 7110C.

14.10.9.1 Projects analyzed under the DOD QSM must use the LCS acceptance limits to evaluate the MS and MSD samples.

14.11 Sample Matrix Spike Duplicates (MSD)

14.11.1 A sample Matrix Spike Duplicate (MSD) is not required for this analysis. When required by the customer/contract, a MSD must be prepared for each analytical batch. The MSD must be prepared as a duplicate of the MS.

14.11.2 The MSD must pass the acceptance criteria established for the MS recovery and the criteria established for duplicate precision.

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14.11.3 An MS/MSD sample analysis may be performed instead of a sample duplicate analysis. If MS/MSD are prepared instead of a sample duplicate, and the batch includes drinking water samples from the state of Arizona, the duplicate analysis criteria for frequency in section 14.9.1.1 of this SOP must be met.

14.12 Summary of QC related Activities:

Method Blank	One per Batch
Reagent Blank	One per Batch (as required by client)
Duplicate Sample	One per Batch or a frequency of 10% for batches containing drinking water samples from Arizona.
Matrix Spike	One per Batch
Matrix Spike Duplicate	One per Batch or a frequency of 10% for batches containing drinking water samples from Arizona. (as required by client)
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch for samples with low alpha and/or beta concentration or in absence of Duplicate sample.

14.13 Corrective Actions for Out-Of-Control Data

14.13.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis and evaluate the usefulness of the data in the final report.

14.13.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.13.3 Matrix Spike Recovery (MS) – MS recoveries that fail high and outside of control criteria with a sample result that is less than the reporting limit may be reported with narration. Additionally, MS recoveries that fail low and outside of control criteria for Drinking Water samples with a sample result that is greater than the MCL must be reported with comment as potentially biased low due to matrix interference. Otherwise, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Matrix effect must be determined by re-analysis of the MS/Sample pair or demonstration of acceptable precision between a MS/MSD

14.13.3.1 The analyst must evaluate the MS results to attempt to determine the cause of the failure and the appropriate

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action to take based on that evaluation. All decisions made must be documented.

14.13.4 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.13.5 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

14.13.5.1 The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.13.6 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

14.13.6.1 The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit, and duplicate precision meets the acceptance criteria.

14.13.7 If there is no additional sample available for reanalysis and evaluate the usefulness of the data in the final report.

14.14 Contingencies for handling Out-of-Control or Unacceptable Data

14.14.1 Method Blank (Reagent Blank): If the sample is exhausted evaluate the usefulness of the data in the final report.

14.14.2 Duplicates: If the sample is exhausted then evaluate the usefulness of the data in the final report.

14.14.3 Matrix Spike Recovery: If a Matrix Spike Duplicate is analyzed and the spike recoveries are not comparable, and the sample is exhausted, evaluate the usefulness in the final report.

14.14.4 Matrix Spike Duplicate: If a Matrix Spike Duplicate is analyzed and the spike recovery is not comparable to the Matrix Spike and the sample is exhausted and evaluate data usefulness in the final report.

15. Method Performance

15.1 Laboratory control samples are analyzed with each batch; the results are charted to monitor control limits and trending.

15.2 Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

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15.3 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.4 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into appropriate receptacles.

16.2 Discard acidified samples and unusable standards into proper waste drains.

16.3 Dispose of waste materials in accordance to type: Non-hazardous, hazardous, non-radioactive, radioactive or mixed.

17. References

17.1 Krieger, H. L. and Whittaker, E. L., Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, "Gross Alpha and Gross Beta Radioactivity," Method 900.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.2 Eaton, A. D., et. al., editors, Standard Methods for the Examination of Water and Wastewater, 19th Edition, "Gross Alpha and Gross Beta Radioactivity (Total, Suspended and Dissolved)," Method 7110, American Public Health Association, Baltimore, MD, 1995.

17.3 Eaton, A. D., et. al., editors, Standard Methods for the Examination of Water and Wastewater, 20th Edition, "Gross Alpha and Gross Beta Radioactivity (Total, Suspended and Dissolved)," Method 7110, American Public Health Association, Baltimore, MD, 1998.

17.4 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW846), Volume 1C, Third Edition, "Gross Alpha and Gross Beta," Method 9310, U. S. Government Printing Office, Washington, D.C., September 1986.

17.5 “Protocol for the Evaluation of Alternate Test Procedures for Analyzing Radioactive Contaminants in Drinking Water”, EPA, Office of Water (MS-140), EPA 815-R-14-002, Feb 2014.

17.6 “Standard Test Method for Beta Particle Radioactivity of Water”, ASTM D 1890-90, ASTM Standards Volume 11.02.

17.7 “Standard Test Method for Alpha Particle Radioactivity of Water”, ASTM D 1943-90, ASTM Standards Volume 11.02.

17.8 “Radiochemical Determination of Gross Alpha and Gross Beta Particle Activity in Water”. Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA 520/5-84-006, Page 00-01-1, 1984.

17.9 “Radiochemical Determination of Gross Alpha Activity in Drinking Water by Co-precipitation”, Eastern Environmental Radiation Facility

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Radiochemistry Procedures Manual, EPA 520/5-84-006, Page 00-02-1, 1984.

17.10 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.11 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.12 Currie, L., Limits for Quantitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40. No. 3, Pg 586-593, 1968.

17.13 Currie, L., Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, NUREG/CR - 4007, USNRC, 1984.

17.14 “American National Standard Calibration and Usage of Alpha/Beta Proportional Counters”, ANSI N42.25-1997.

17.15 “Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)”, July 2004, Final.

17.16 “American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories”, ANSI N42.23-1996.

17.17 Department of Defense Quality System Manual for Environmental Laboratories (DoD QSM), current version.

17.18 National Primary Interim Drinking Water Regulations (NIPDWR), Part 141.15.

17.19 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.20 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.21 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

17.22 Pace SOP PGH-R-002, current revision (Gas Flow Proportional Counter Operation).

17.23 Pace SOP PGH-R-005, current revision (Analysis of Samples for Strontium-90(89) Content).

17.24 Pace SOP PGH-R-006, current revision (Analysis of Samples for Total Uranium Content).

17.25 Pace SOP PGH-C-025, current revision (MCL Violation Reporting).

17.26 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Attachment I: Gross Alpha and Gross Beta Calculations.

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18.2 Attachment II: Tables depicting typical gross alpha and gross beta detection limits with typical sample quantity/system efficiency/system background/count time values.

18.3 Attachment III: Evaluation procedures for QC Samples using Numerical Indicators.

19. Method Modifications

19.1 EPA 900.0 method indicates the use of 1N HNO_3 for initial sample preservation in the amount of 15mL of 1N HNO_3 per 1L of sample. Sample bottles purchased and provided by Pace Analytical are pre-packaged containing 2mL of concentrated nitric acid in a 1L bottle. The final acid concentration is increased from 0.015 N to 0.032 N when comparing the above techniques and has no adverse impact on analytical results.

19.2 A 5mL test is performed to determine solid content. This is used to determine the maximum amount of sample that can be processed and still remain under the 100mg solid limit.

19.3 Ludox reagent is added to aqueous samples to ensure even distribution of sample residue in the counting planchet when drying. The use of Ludox significantly improves mass distribution for low mass samples, such as drinking waters, and QC samples using DI as the sample matrix. Because the material is used for drinking water and DI QC samples, the reagent is added to all aqueous samples for consistency. Ludox reagent does not interfere with accurate gross alpha and/or gross beta analysis.

19.4 ATP test solution is not listed in the EPA procedures. It is added to ensure evaluation of the Laboratory Control sample is not limited to only the low mass end of the attenuation curves, since individual sample residues may vary from the lowest to highest mass range of the attenuation curves.

19.5 The aqueous procedure has been modified to handle other matrix types (i.e. solids, vegetation, etc.).

19.6 For routine analysis of aqueous samples, PASI's default process for measuring the quantity of sample to be analyzed is to measure the mass of sample transferred and the mass of sample used is documented in the appropriate logbook. Subsequent calculations for analysis of aqueous samples assume the density of aqueous samples to be 1.0g/mL. For these samples, analysis results are reported in volume units without density correction.

19.7 Method SM7110C indicates use of 1.0mL of barium carrier solution (5mg Ba/mL) for the co-precipitation technique. Pace utilizes 0.5mL of a 10mg Ba/mL solution. This modification was made in order to minimize the footprint of chemical solutions used in the lab. The 10mg Ba/mL solution is used for EPA 905.0 (PGH-R-005 current revision) and is shared between tests. The quantity of Ba added for the co-precipitation technique is equivalent, 5mg Ba.

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19.8 Method SM7110C indicates use of 1.0mL of Iron carrier solution (5mg Fe/mL) for the co-precipitation technique. Pace utilizes 0.5mL of a 10mg Fe/mL solution. This modification was made in order to minimize the footprint of chemical solutions used in the lab. The 10mg Fe/mL solution is used for EPA 908.0 (PGH-R-006 current revision) and is shared between tests. The quantity of Fe added for the co-precipitation technique is equivalent, 5mg Fe.

19.9 The calibration process for the evaporation technique in this SOP requires concentration of a salt matrix onto a planchet, re-suspension of the salt residue in acid followed by addition of calibration standard to the dissolved salt solution in the planchet. EPA method 900.0 specifies combining the salt matrix and calibration standard in a glass beaker followed by evaporation. The evaporated calibration liquid is transferred to a planchet for evaporation under a heat lamp. This modification was incorporated by PASI in order to ensure complete capture of the calibration material for absolute efficiency determination, eliminating transfer losses as a source of error.

19.10 For the co-precipitation technique calibration procedure, SM7110C specifies the use of six replicate source analyses for the determination of a single-point detector efficiency. The approach does not incorporate compensation for losses due to mass attenuation. PASI utilizes a minimum of four calibration sources of varying mass in order to generate an attenuation curve.

19.11 For the co-precipitation technique calibration procedure, the method is designed to selectively precipitate calibration elements by pH adjustment and utilizes a pH indicator in the process. PASI's preparation of calibration sources for the co-precipitation technique varies from method SM7110 due to application of a smaller-scale process for calibration source generation. This scale adjustment limits un-recoverable losses while adhering to the intent of method SM7110C. In the defined calibration process, Pace limits the quantity of acid used to match the process scale.

19.12 For the evaporative method, EPA Method 900.0 does not require addition of nitric acid to samples prior to the initial evaporation step. PASI requires addition of nitric acid to samples prior to evaporation to aid in the removal of chlorides assumed to be present in samples.

19.13 EPA Method 900.0 limits transfer of evaporated samples to planchets to no more than 5mL at a time. This volume is not measured and so PASI has incorporated a qualification of approximately 5mL at the appropriate step.

20. Revisions

Document Number	Reason for Change	Date

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Document Number	Reason for Change	Date
PGH-R-001-15	1. Removed an extraneous line at 5.2. Removal subsequently updated all following numbering within Section 5. 2. Added new material used in procedure at 9.14, wide twill smears. 3. Updated beginning of Section 11 to document the types and general calibration development of SOP-specified calibrations. 4. Modified header at 11.2 to specify the calibration process documented in 11.2 was for the evaporative technique of analysis. 5. At 11.4 added the process for calibrating air filters and contamination smears for gross alpha efficiency. 6. At 11.5 added the process for calibrating air filters and contamination smears for gross alpha crosstalk. 7. At 11.6 added the process for calibrating air filters and contamination smears for gross beta efficiency. 8. Section 11.7 updated to include specific criteria for calibration curve development. 9. Section 11.8 updated to include specific criteria for calibration curve acceptability. 10. Section 11.9 updated to include the required frequency of calibrations as well as an allowed calibration-verification process that may be used in lieu of performing a new calibration. 11. Added revision table for modifications.	25Mar2014
PGH-R-001-16	1. Section 8.1 – Added requirement and instruction for pH verification and recording verification information. 2. Section 8.3 – Added maximum hold time of 180 days. 3. Section 9 – Referenced the current revision of the instrument SOP, PGH-R-002. 4. Section 10 – Added Hydrofluoric acid, changed nitric acid concentrations as N instead of M, fixed ferric nitrate chemical formula. 5. Section 11 – Included provisions for non-drinking water sample residue greater than the highest residue on the calibration curve, and instruction s to add ludox to calibration sources generated for the evaporative techniques. 6. Section 12.1 – Changed max beta residue to 150 mg to be within calibration. 7. Section 12.3 – Added to ensure QC samples are prepared, acidified and spiked, prior to adding any chemicals. 8. Section 12.1.4 – Added note regarding chlorides and the addition of extra nitric acid. 9. Section 12.1.8 – Added instructions to limit transfer volumes to planchets to eliminate overflow and loss of sample. 10. Section 12 – Included instrument SOP PGH-R-002	13Jul2014

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Document Number	Reason for Change	Date
	reference where applicable. 11. Section 12.6 – Included information regarding reprep of 7110C samples with high residue, and evaluate data usefulness of method against EPA 900.0. 12. Section 12.6.1 – Inserted the preparation and addition of QC samples to ensure spiking occurs prior to other chemical additions. 13. Section 14 – Updated LCS/LCSD, DUP RPD, and MS/MSD limits for SM7110C and direct plate techniques. Section 19 – Updated to include method deviations, including initial sample preservation acid concentration and amount, number of points for calibration for method SM 7110C and reasoning, use of Ludox®, concentrations of iron and barium carriers in SM7110C, use of additional acid during evapo	
PGH-R-001-17	Removed from section 5.1: Analysts must be trained as radiation workers and personal dosimeter worn.	20Feb2015
PGH-R-001-18	1. Section 12- Updated references to using 8N HNO_3 rinses to using 1N HNO_3 rinses to comply with EPA 900.0. 2. Section 12.1.8 – Updated SOP to state minimum number of rinses needed as 2, but additional rinses may be used as necessary. 3. Section 12.5.1.1 – Clarified solids aliquot guidelines. 4. Section 12.6.1 – Included typical sample aliquot amount.	13Feb2017
S-PGH-R-001-rev.19	1. Section 2.7, 2.8, 2.9 were added to include non-aqueous samples and unusual matrices. 2. Section 3.4 removed, urine analysis is not performed. 3. Section 8 and 12.2 were removed. Pace does not analyze urine samples. 4. Section 8.1.2 samples must be held minimum of 24 hours. 5. Section 10: Added reagents and preparation instructions associated with ATP solution. 6. Section 12.2 “Analysis of Urine samples” removed as the lab does not analyze this matrix. 7. New Section 12.2 “Analysis of Organic Matrices” modified to specify oils as organic materials. 8. Section 14.13.3 updated to standardize required actions for MS failures. 9. Section 17.5 includes reference for the ATP solution 10. Section 19.4 discusses addition of ATP solution to LCS and LCSD samples.	08Feb2018

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Attachment I (Calculations)

The gross alpha or gross beta radioactivity concentration of a sample is calculated according to the following equations:

Eq. 1 $$\alpha Act = \frac{(S_A - B_A)}{(Denom)}$$

Eq. 2 $$\beta Act = \frac{(S_B - B_B - X * (S_A - B_A))}{(Denom)}$$

Eq. 3 $$Denom = E * V * 2.22$$

Where:

αAct	=	Gross Alpha sample concentration in pCi/unit (L, g, F, etc.)
βAct	=	Gross Beta sample concentration in pCi/unit (L, g, F, etc.)
S_A	=	Gross alpha count rate for the sample (in cpm)
S_B	=	Gross beta count rate for the sample (in cpm)
B_A	=	Alpha count rate for the detector background (in cpm)
B_B	=	Beta count rate for the detector background (in cpm)
2.22	=	Conversion factor from dpm to pCi.
E	=	Detector alpha or beta efficiency (as cpm/dpm, obtained from the respective alpha or beta efficiency calibration curve).
V	=	Sample quantity analyzed, (volume, mass, or fraction in L, g, or %filter, etc).
X	=	Alpha cross talk factor (as fraction, obtained from the alpha cross talk calibration curve)

The sample specific counting uncertainty (C.U.) is calculated as follows.

Eq. 4 $$\alpha C.U. = \frac{1.96 * \sqrt{((S_A/T_S) + ((B_A/T_B)}}{\text{Denom}}$$

Eq. 5 $$\beta C.U. = \frac{1.96 * \sqrt{((S_B/T_S) + ((B_B/T_B)}}{\text{Denom}}$$

Where:

T_S	=	Count time for the sample (in minutes)
T_B	=	Count time for the background count (in minutes)

S, B, and Denom as previously defined.

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As summed background and analyte count rates approach zero, assumptions underlying the uncertainty calculation are violated and it will return an unrealistic value of zero (0) uncertainty when zero summed counts are observed. The following equation provides a more accurate estimate of count uncertainty at zero and near-zero count rates.

Eq. 6 ZeroUnc=ZeroActFact/SmplTime/Denom

Note 1: Depending on sample type and contract requirements the zero activity factor may be either 3.0 or 2.71. PASI's default ZeroActFact is 2.71 consistent with the current version of ANSI N42.23. Bioassay samples must be calculated using 3.0 to be consistent with ANSI N13.30

Note 2: The Zero Count Uncertainty is compared to the count uncertainty above. The larger of the two is used as the counting uncertainty in subsequent total error calculations.

The error term is further evaluated to provide an estimate of total error hereafter referred to as the *Combined Standard Uncertainty* (CSU a.k.a. TPU). The CSU is calculated as follows:

Eq.7 $$\alpha\text{CSU (pCi/U)} = \sqrt{(\alpha\text{C.U.})^2 + (UE1 * \alpha Act)^2 + (UE2 * \alpha Act)^2 + (UE3 * \alpha Act)^2 + (UE4 * \alpha Act)^2}$$

Eq.8 $$\beta\text{CSU (pCi/U)} = \sqrt{(\beta\text{C.U.})^2 + (UE1 * \beta Act)^2 + (UE2 * \beta Act)^2 + (UE3 * \beta Act)^2 + (UE4 * \beta Act)^2}$$

Where:

UE1, UE2, UE3, and UE4 represent partial derivatives estimating the relative uncertainty at the *95% confidence interval* for various factors in the activity calculation as follows:

UE1 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with preparation (e.g., sample aliquot or transfers and splits prior to addition and equilibration of tracer).

UE2 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with analysis (e.g., peak integration, peak overlap, tracer contaminants).

UE3 represents combined factors estimating relative uncertainty (fractional) associated with yield correction (e.g., count uncertainty for tracer peak, SRM known value, tracer volume or mass aliquot, tracer equilibration efficiency).

UE4 represents the factor estimating additional uncertainty (activity) associated with an individual sample -- to be used in exceptional circumstances with approval of the Department Supervisor and appropriate documentation and narration only.

The Minimum Detectable Concentration (MDC) is calculated per guidance of ANSI N42.23 and N13.30 as:

Eq. 9 $$\alpha\text{ MDC} = \frac{4.65 * \sqrt{(B_A/T_B) * T_S} + ZeroActFact}{T_S * \text{Denom}}$$

Eq. 10 $$\beta\text{ MDC} = \frac{4.65 * \sqrt{(B_B/T_B) * T_S} + ZeroActFact}{T_S * \text{Denom}}$$

Where:

B_A, B_B, T_B, T_S, ZeroActFact, and Denom are as previously defined.

Perform instrument calibration calculations as follows:

Efficiency Calculation:

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Eq. 11 $E_A = \frac{C_A}{D_A}$

Eq. 12 $E_B = \frac{C_B}{D_B}$

Eq. 13 $X_A = \frac{A_B}{A_A}$

Where:

EA	=	System alpha efficiency (as cpm/dpm).
EB	=	System beta efficiency (as cpm/dpm).
C_A	=	Net alpha count rate of the alpha calibration source (in cpm).
D_A	=	Total dpm of alpha standard added to the calibration source (in dpm).
C_B	=	Net beta count rate of the alpha calibration source (in cpm).
D_B	=	Total dpm of beta standard added to the calibration source (in cpm).
X_A	=	Alpha to beta crosstalk factor for the calibration source (as decimal).
A_B	=	Beta count rate for the alpha calibration source used for cross talk calibration (as cpm).
A_A	=	Alpha count rate for the alpha calibration source used for cross talk calibration.

The critical level (Lc) is calculated per guidance of ANSI N42.23 as:

Eq. 14 $$Lc = \frac{1.65 * \sqrt{(B) * (1/Ts + 1/Tb)}}{Denom}$$

Where:

B, T_s, T_b, *ZeroActFact*, and Denom are as previously defined.

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Attachment II (Method Performance)

Method performance is determined by the background and efficiency of the specific detector used, sample quantity analyzed, and the length of time the sample is counted. In general, the larger the sample, the lower the background, and the longer the count, the lower the detection limits.

The following table (Table 1) depicts typical gross alpha detection limits with typical sample quantity/system efficiency/system background/count time values:

Table 1: Sample Variables versus MDA for Gross Alpha Analysis

Det. Bkg (cpm)	Sys. Eff. (cpm/dpm)	C.T. (min)	Samp. Quantity (g,mL)	MDA (pCi/L)
0.05	0.30	240	50	2.4
0.05	0.30	100	150	1.3
0.05	0.30	60	200	1.3
0.05	0.25	500	50	1.9
0.05	0.25	100	200	1.2
0.05	0.25	60	500	0.65
0.10	0.20	500	100	1.6
0.10	0.20	240	150	1.6
0.10	0.20	60	500	1.1
0.10	0.10	1000	50	4.4
0.10	0.10	500	100	3.2
0.10	0.10	100	200	3.9

The following table (Table 2) depicts typical gross beta detection limits with typical sample quantity/system efficiency/system background/count time values:

Table 2: Sample Variables versus MDA for Gross Beta Analysis

Det. Bkg (cpm)	Sys. Eff. (cpm/dpm)	C.T. (min)	Samp. Quantity (g,mL)	MDA (pCi/L)
0.75	0.45	240	50	5.4
0.75	0.45	100	150	2.9
0.75	0.45	60	200	2.8
0.75	0.40	500	50	4.2
0.75	0.40	100	200	2.4
0.75	0.40	60	500	1.3
1.2	0.35	500	100	3.0
1.2	0.35	240	150	2.9
1.2	0.35	60	500	1.8
1.2	0.30	1000	50	4.9
1.2	0.30	500	100	3.5
1.2	0.30	100	200	4.0

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Attachment III - (Numerical Performance Indicators)

1. Method Blank (MB)

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:

x = Measured blank activity
u(x) = Standard uncertainty (1 sigma) in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. Laboratory Control Sample (LCS)

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = Combined standard uncertainty (1 sigma) of the result squared.
$u^2(c)$ = Combined standard uncertainty (1 sigma) of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. Duplicates (DUP)

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:

x_1, x_2 = two measured activity concentrations
$u^2(x_1)$, $u^2(x_2)$ =the combined standard uncertainty (1 sigma) of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:

x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective combined standard uncertainties (1 sigma) of these values.

4.2 MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

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STANDARD OPERATING PROCEDURE

Mechanical Hydrometer/Grain Size Analysis

Reference Methods: ASTM-D422

Local SOP Number:	S-MT-ME-043-Rev.01
Effective Date:	Date of Final Signature
Supersedes:	S-MT-ME-043-Rev.00

APPROVALS

Laboratory Manager	Date: 2/2/2018
Laboratory General Manager	Date: 1/31/2018
Laboratory Quality Manager	Date: 1/25/2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure has been written to describe the Grain Size/Hydrometer Analysis process conducted by Method ASTM D422.

2. Summary of Method

2.1. The distribution of particle sizes retained on the #200 sieve is determined by sieving. Particle sizes smaller than the #200 sieve are determined by a sedimentation process using a hydrometer.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP is applicable to the quantitative determination of the distribution of particle sizes in soils.

4. Applicable Matrices

4.1. This SOP is applicable to soils.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	500g or more in zip-lock bags	N/A	Ambient	Not applicable to this method

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Stirring device	electric motor turns a vertical shaft at a speed of 10,000 rpm	Hamilton Beach Commercial Mixer
ASTM hydrometer	graduated to read specific gravity of the suspension and grams per liter of suspension	Model 152 H.
sedimentation cylinder	glass 18 inches high and 2 ½ inches in diameter. Volume of 1000 mL.	Fisher p/n NC0105384, 08-568-2A or equivalent

series of sieves	3", 2", 1½", 1", 3/4", 3/8", #4, #10, #20, #40, #60, #140, #200.	Gilson
beaker or jar	250 mL capacity.	250 mL beaker-Fisher WM Pint Mason Jar-local store
Trays for drying soils	Various sizes	Local
Clock	Clock with a second hand	
RoTap or shaker	Tyler Ro-Tap or shaker	Tyler Rx-29, Gilson SS-15 or equivalent
Drying oven	Drying oven capable of maintaining 110 +/- 5 ^{o}C	Thelco 130DM, Isotemp 630F or equivalent
Analytical Balance	Capable of weighing >.1 gram	Mettler Toledo New Classic MF, Ohaus Adventurer, Sartorius LC 620S or equivalent

10. Reagents and Standards

10.1. Table 10.1. Reagents and Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
solution of sodium hexametaphosphate(HMP)	Dissolve 2000 g HMP in ~3 L of warm Deionized water. Dilute to 4L	NWS p/n GFS/67552

11. Calibration and Standardization

11.1. Analytical Balance

11.2. Daily Calibration Check

11.2.1. Balance must be calibrated daily. Refer to SOP S-MN-Q-264, Daily Calibration Verification.

12. Procedure

12.1. Separate the sample in two portions, particles retained on the # 10 sieve and particles passing the # 10 sieve. Do this by separating large rocks and aggregates using # 10 sieve. Place remaining solids into a clean mortar and break apart with rubber coated pestle or onto freezer paper covered tray and break with wooden rolling pin. Sieve again through the # 10 sieve. Repeat until only rocks (solids that cannot be broken apart further) remain. The portion passing through the #10 sieve (hydrometer portion) shall be approximately 100 g. for sandy soils and approximately 50 grams for silt or clay soils. The portion retained on the #10 sieve shall be approximately 400-500 g. (depending on initial sample weight).

12.2. Wash the material which is retained on the #10 sieve with tap water. Rinse with Deionized water. Dry the soil that is retained on #10 sieve in 110 ± 5^{o} C. oven until dry (about an hour).

12.3. Weigh dried sample and record in appropriate section of the worksheet (Attachment I).

12.4. Sieve dried samples through a stack of 3", 2", 1½, 1", 3/4", 3/8", #4, and #10, sieves for 7 minutes on the RoTap. Record weights in appropriate columns on 'Hydrometer/Grain Size Analysis Form' (Attachment I) Note: Use the larger sieves only if needed.

12.5. Select a representative sample of the material that passes #10 sieve as determined in section 11.2 above (hydrometer portion). Record amount on Hydrometer Grain Size Analysis Form (Attachment

I). NOTE: For sandy soils the sample should be approximately 100 grams. For silt or clay soils the sample should be approximately 50 grams. For samples that are a blend, use between 50 and 100 grams depending on makeup.

12.6. Place the selected sample in a 250 mL beaker and cover with 125 mL of the Sodium Hexametaphosphate (HMP) solution. Stir with metal spatula until the soil is thoroughly wetted. Allow to soak at least 16 hours. Set up a blank at this time with only the HMP solution. At this time fill sedimentation cylinders 1/3 to 1/2 full with deionized water. Also fill a couple of gallon jugs at this time. This will allow for all of the samples to be same ambient temperature when analyzing.

12.7. At the end of the soaking period, disperse the sample by placing it in the stirring apparatus. If needed add deionized water to the stirring apparatus so that the cup is at least ½ full. Stir for 1 minute. Start with the blank. NOTE: for steps 12.7-12.9 a tag team approach may be used, with one person prepping the sample and the other taking readings.

12.8. Immediately after stirring for 1 minute, transfer the soil/water slurry to the glass sedimentation cylinder, and add deionized water until the total volume is 1000 mL. Place stopper on top of the cylinder and turn the cylinder upside down and back for a period of 1 minute to complete the agitation of the slurry.

12.9. After 1 minute of agitation, set the cylinder on counter and place the hydrometer tube into the cylinder. For the blank, also insert the thermometer**.** Take the first reading on the hydrometer after 2 minutes. When taking readings, obtain the reading at the bottom of the meniscus formed by the suspension around the stem. Record the reading in the appropriate 'Hydrometer Reading' column on the 'Hydrometer/Grain Size Analysis Form' (Attachment I). ***Also record the corrected temperature on the form.*** Additional readings will be taken at 5, 15, 30, 60, 250, 1440 minutes or until the reading has reached the blank reading ±2. Record readings in the appropriate columns on the 'Hydrometer/Grain Size Form' (Attachment I).

NOTE: When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20-25 seconds before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled water.

12.10 After taking the final hydrometer reading, transfer the suspension slurry to a #200 wet sieve and wash with tap water until the water passing through the sieve is clear. Rinse with deionized water. Transfer the material left on the #200 sieve into a small pan and dry in the oven at ?110 ± 5º C.

12.10. After the material has been dried to a constant weight, let it cool, weigh the material, and place it in a nest of sieves including #20, #40, #60, #140, and #200. Sieve for 7 minutes on RoTap and record each retained weight on the 'Hydrometer/Grain Size Form (Attachment I).

12.11. Calculations: All data collected during the sieving and hydrometer reading process is then transferred into the Geosystems Soils Testing computer program which performs all calculations and plots the graph. The report form produced by the computer program is titled 'ASTM D422 Report'.

12.12. Other data that is entered on the 'Hydrometer/Grain Size Analysis' form shall include project name, Job#, Date of Sample Collection, Date Test is Performed, analyst and Sample number.

13. Quality Control

13.1. Not applicable to this SOP.

14. Data Analysis and Calculations

14.1. All data collected during the sieving and hydrometer reading process is then transferred into the Geosystems Soils Testing computer program which performs all calculations and plots the graph.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

13.2. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: Is not applicable to this method.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Training Procedures.

19. Method Modifications

19.1. Not performing hygroscopic moisture correction on air dried sample for hydrometer test.

19.2. 10.1 modified HMP to be 50g/L instead of 40g/L per modification of ASA, Methods of Soil Analysis, Part 1, 15-5 Hydrometer Method.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. For further information on waste management, see SOP S-MN-S-003, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute; Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Standard Test Methods for Particle-Size Analysis of Soils, ASTM-D422-63, ASTM (2002) ASTM International.

25.5. Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants, ASTM-D421-85 (2002) ASTM International.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I - Hydrometer Grain Size Analysis Spreadsheet.

27. Revisions

Document Number	Reason for Change	Date
S-MT-ME-043	First Issue	23Oct2015
S-MT-ME-043-Rev.01	Update to LLC. Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. In Section 12.5, removed "in blank" from second sentence, split up NOTE into additional sentences. In Section 12.6, replaced "tongue depressor" with "metal spatula" Removed #10 from Section 12.10.	25Jan2018

Attachment I - Hydrometer Grain Size Analysis Spreadsheet

Document Name: Hydrometer Grain Size Analysis	Document Revised: 26Oct2015 Page 1 of 1
Document No: F-MT-I-287-Rev.00	Issuing Authority: Pace Motana Quality Office

HYDROMETER/GRAINSIZE ANALYSIS (ASTM D422)

Air Drying

Analyst:_____ Date:_____ Humidity:_____

Therm ID:_____ Temp.:_____ Temp Corr.: _____

	Sample ID# :	
	Pan ID# :	
	Balance ID:	
A.	Paper + Pan Tare	
B.	Mass Dry Sample w/Pan	
C.	+10 Weight:	
D.	+10 weight (after washing):	
E.	Loss by washing: (D-E)	

Analyst:_____

Sieve	**Weight Retained**
3in.	
2in.	
1 1/2in.	
1in.	
3/4in.	
3/8in.	
#4	
#10	

Grams of -10 used for Hydrometer:_____

50 Grams Clay ← → **100 Grams Sand**

Start Time: _____ **Analyst:**_____ **Blank Reading:**_____

Therm ID:_____ **Temp:**_____ **Temp Corr.:**_____

Time (Min)	**Time (Military)**	**Hydrometer Reading**
2.0 Min.		
5.0 Min.		
15 Min.		
30 Min.		
60 Min.		
250 Min.		
1440 Min.		

Sieve*	**Weight Retained**
#20	
#40	
#60	
#140	
#200	

Hydrometer is washed

Reviewed By/Date:_____

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF FREE CYANIDE
REFERENCE METHODS: EPA SW-846 METHODS 9013A AND 9014

SOP NUMBER:	S-IN-I-129-rev.06
EFFECTIVE DATE:	July 5, 2017
SUPERSEDES:	S-IN-I-129-rev.05

APPROVAL

General Manager — June 22, 2017 Date

Quality Manager — June 21, 2017 Date

Department Manager — June 22, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

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Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining free cyanide in aqueous samples and non-aqueous sample extracts while meeting the requirements specified in EPA methods 9013A and 9014.

2. Summary of Method

2.1. In the colorimetric procedure, cyanide is converted to cyanogen chloride by reaction of the cyanide with chloramine-T at a pH below 8. After the reaction is complete, the color is formed when pyridine-barbituric acid reagent is added. Absorbance is read at 570nm for the complex that is formed. To obtain colors of comparable intensity, it is essential that the salt content be the same in both samples and standards.

3. Scope and Application

3.1. This method is applicable for the measurement of free (non-complexed) cyanide.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of free cyanide analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable matrices

4.1. This method is applicable to drinking water, natural surface waters, domestic and industrial wastewaters and soil extracts.

5. Limits of Detection and Quantitation

5.1. The default reporting limit is 0.10mg/L for aqueous samples and 0.5mg/kg for solids. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Oxidizing agents such as chlorine decompose most cyanides. Chlorine interferences can be removed by adding an excess of sodium arsenite to solid sample extracts or an excess of ascorbic acid to aqueous samples to reduce the chlorine to chloride, which does not interfere with this test.

6.2. Sulfides affect the colorimetric process. If a drop of sample on lead acetate paper indicates the presence of sulfide, treat a portion of the sample (25mL more than the cyanide determination) with powdered lead carbonate. This will precipitate out any sulfides as grey lead sulfide. After the sample no longer shows a positive result on the lead acetate paper, filter the sample through filter paper and measure out the sample aliquot to be used for the cyanide test.

6.3. Thiocyanate is reported to be an interferent when present at very high levels.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	250mL in plastic or glass container	pH >12 using 50% NaOH	Cool to ≤6°C	Analysis must be completed within 14 days of collection date.
Solid	50g in a glass widemouth container	None required	Cool to ≤6°C	Extraction must be completed within 14 days of collection date. Analysis must be completed within 14 days of extraction date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation/Equipment

Equipment	Vendor	Description / Comments
Spectrophotometer	Westco SmartChem 200	Or equivalent equipment for use at 570nm.
pH meter	Accumet AR25 or equivalent	

9.2. General Supplies

Item	Vendor	Description
Auto-pipettes	Eppendorf or equivalent	Various sizes
Volumetric flasks	Fisher or equivalent	Class A, various sizes
Beakers	Fisher or equivalent	
Plastic bottles	C&G or equivalent	500mL for soil preparation
Filter paper	Whatman #1 or equivalent	
Syringe filter	Environmental Express or equivalent	0.45um
Potassium-Iodide Starch paper	Fisher or equivalent	Used to test for oxidizing interferences
Lead Acetate paper	Fisher or equivalent	Used to test for sulfide interferences
Ottawa Sand	Fisher or equivalent	Used as a clean non-aqueous matrix

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Ascorbic Acid	Reagent grade crystals or equivalent
Lead Carbonate	Reagent grade powder or equivalent
Sodium Hydroxide (50%)	Reagent grade solution or equivalent.
Sodium Hydroxide Pellets	Reagent grade or equivalent.
Sodium phosphate monobasic monohydrate	Reagent grade, crystalline, or equivalent
Phosphate buffer, 1M	Dissolve 69g of sodium phosphate monobasic monohydrate in reagent water in a 500mL volumetric flask. Add 2mL concentrated probe rinse solution and dilute to volume with reagent water. Refrigerate when not in use. Expires 6 months from date of preparation.
Barbituric acid	Reagent grade or equivalent
Pyridine	Reagent grade liquid or equivalent
Hydrochloric Acid	Concentrated
Pyridine-barbituric acid reagent	Under a fume hood, place 15g of barbituric acid into a 1L beaker and wash down the sides of the beaker with 100mL of reagent water. Add 75mL of pyridine and mix. Add 15mL of concentrated hydrochloric acid and mix. Cool to room temperature and then dilute to about 900mL with distilled water and mix until all barbituric acid has dissolved. Add 4mL concentrated probe rinse solution then transfer to a 1L volumetric flask and dilute to volume with reagent water. Store in an opaque container and refrigerate when not in use. Expires 6 months from date of preparation.
Chloramine-T	Reagent grade powder or equivalent
Chloramine-T solution	Dissolve 0.4g of chloramine-T in 100mL of reagent water. Refrigerate until ready to use. Must be prepared fresh daily.
Concentrated Probe Rinse Solution	Westco part #3AS-RN00-21, or equivalent
Probe Rinse Solution	Dilute 0.5mL of the concentrated probe rinse solution to 1L with reagent water. Store ambient.
Concentrated Cuvette Wash Solution	Westco part #3AS-RN00-20, or equivalent
Cuvette Wash Solution	Dilute 50mL of the concentrated cuvette wash solution to 1L with reagent water. Store ambient.
Sodium Hydroxide diluent solution (0.25N)	Used as diluent and receptacle wash water. Dissolve 10g of sodium hydroxide pellets in 500mL of reagent water. Dilute to 1L. Expires 6 months from date of preparation.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This standard is used for spiking MS/MSD sets.	Used for the LCS and MS

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Cyanide Calibration standard	Ricca; catalog #2543; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Refrigerate continuously upon receipt only taking an aliquot to reach room temperature for use.
Intermediate Cyanide Calibration standard	Refer to Section 10.2.3.1	Must be prepared fresh daily	Not applicable
Working Cyanide Calibration Standards	Refer to Section 10.2.3.2	Must be prepared fresh daily	Not applicable
Stock Cyanide ICV standard	SPEX (anion standard); catalog # RSCN9-2Y; 1000mg/L in 2% KOH solution or equivalent	Manufacturer's recommended expiration date	Refrigerate continuously upon receipt only taking an aliquot to reach room temperature for use.
Intermediate Cyanide ICV standard	Refer to Section 10.2.3.3	Must be prepared fresh daily	Not applicable
Working Cyanide ICV standard	Refer to Section 10.2.3.4	Must be prepared fresh daily	Not applicable

10.2.3. Standard Preparation Procedures

10.2.3.1. Intermediate Cyanide Calibration Standard Preparation

Dilute 2.5mL of the stock cyanide standard (1000mg/L) to 50mL with diluent for a final concentration of 50mg/L.

10.2.3.2. Working Cyanide Auto-dilution Calibration Standard Preparation

Dilute 0.5mL of the Intermediate Cyanide Calibration Standard (50mg/L) to 50mL in diluent for a final concentration of 0.5mg/L. This standard must be prepared fresh daily and will be auto-diluted by the SmartChem autosampler to prepare the other calibration standards as detailed below:

Standard ID	Percentage of 0.5mg/L Calibration Std. Used	Final Concentration
CAL0	0%	0 mg/L
CAL1	1%	0.005 mg/L
CAL2	2%	0.01 mg/L
CAL3	4%	0.02 mg/L
CAL4 (CCV)	10%	0.05 mg/L
CAL5	20%	0.10 mg/L
CAL6	40%	0.2 mg/L
CAL7	100%	0.5 mg/L

10.2.3.3. Intermediate Cyanide ICV Standard Preparation

Dilute 1.25mL of stock ICV standard (1000mg/L) to 50mL with diluent to give a standard concentration of 25mg/L. This standard is also used to prepare the LCS and MS.

10.2.3.4. Working Cyanide ICV Standard Preparation

Dilute 5mL of Intermediate Cyanide ICV Standard (25mg/L) to 25mL with diluent to give a standard concentration of 5mg/L.

11. Calibration

11.1. Initial Calibration: Initial calibration standards are analyzed in increasing order of concentration. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve is run on each working day.

11.2. Linear Calibration: Using the Westco SmartChem software, prepare a standard curve by plotting absorbance of standard versus the cyanide concentration. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately after an initial calibration curve. The acceptable range for this standard is +/-10% Difference, which is equivalent to 90-110% Recovery.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.6. Initial Calibration Blank (ICB): An ICB must be analyzed after each ICV. If any ICB result is above the reporting limit, sample analysis must not proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable range for this standard is +/-10% Difference, which is equivalent to 90-110% Recovery.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by

acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.9. Continuing Calibration Blank (CCB): A CCB must be analyzed after each CCV. If any CCB result is above the reporting limit, sample analysis must be stopped. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedure

12.1. Aqueous Sample Preparation

12.1.1. Oxidizing agents such as chlorine decompose most cyanide. To determine if oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.5g portions of ascorbic acid until a drop of sample produces no color on the indicator paper. Add an additional 0.5g portion of ascorbic acid.

12.1.2. Prescreen aqueous samples for sulfide. Acidify a piece of lead acetate paper with a drop of acetic acid. Add a drop of sample to the paper. If a brown or black precipitate appears, sulfides are present and must be removed. Add 5g of lead carbonate to a 50mL aliquot of the sample and shake. A precipitate should form. After 2 minutes, filter the sample through filter paper and recheck for sulfides. Repeat if necessary.

12.1.3. Dilute 1mL of sample to 10mL with diluent and analyze per Section 12.3.

12.1.4. Method Blank Preparation: Method Blank consists of 20mL diluent solution.

12.1.5. LCS Preparation: Dilute 0.4mL of the Working Cyanide ICV Standard (5mg/L) to 10mL with diluent solution for a concentration of 0.2mg/L.

12.1.6. MS/MSD Preparation: Dilute 0.4mL of the Working Cyanide ICV Standard (5mg/L) to 10mL with sample for a concentration of 0.2mg/L.

12.2. Soil/Solid Sample Preparation

12.2.1. Weigh 4.0g of sample into a plastic bottle, add 0.4mL of 50% NaOH and dilute to 40mL with reagent water and mix. A smaller sample size may be used if ratio of sample to NaOH to reagent water is maintained.

12.2.2. Shake sample for 10-15 seconds and allow sample to settle for 1 minute. Check sample pH. Sample pH should be >10. If not, add 50% NaOH to pH>10. Repeat 50% NaOH addition until pH does not drop.

12.2.3. Tumble sample for 16-20 hours then filter.

12.2.4. To determine if oxidizing agents are present, test a drop of the filtrate with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.5g portions of ascorbic acid until a drop of sample produces no color on the indicator paper. Add an additional 0.5g portion of ascorbic acid.

12.2.5. Prescreen the filtrate for sulfide. Acidify a piece of lead acetate paper with a drop of acetic acid. Add a drop of sample to the paper. If a brown or black precipitate appears, sulfides are present and must be removed. Add 5g of lead carbonate to a 50mL aliquot of the sample and shake. A

precipitate should form. After 2 minutes, filter the sample through filter paper and recheck for sulfides. Repeat if necessary**.**

12.2.6. Dilute 1.0mL of filtrate to 5mL with diluent and analyze per Section 12.3.

12.2.7. **Method Blank Preparation:** A Non-Aqueous Method Blank is prepared using Ottawa Sand and treating and tumbling in the same manner as non-aqueous samples per Sections 12.2.1 through 12.2.6.

12.2.8. **LCS Preparation:** Dilute 0.4mL of the Working Cyanide ICV Standard (5mg/L) to 10mL with the prepared Non-Aqueous Method Blank filtrate for a concentration of 10mg/kg.

12.2.9. **MS/MSD Preparation:** Dilute 0.4mL of the Working Cyanide ICV Standard (5mg/L) to 10mL with prepared non-aqueous sample filtrate for a concentration of 10mg/kg.

12.3. Determination of Cyanide

12.3.1. Configure instrument according to manufacturer's instructions. Establish initial calibration as described in Sections 11.1 through 11.6.

12.3.2. Fill disposable sample cups with samples and load them into the autosampler in the desired order. Fill clean reagent bottles with the appropriate reagents for this method as noted in Section 10.1.

12.3.3. Select the appropriate method in the software with the following parameters:

Type	End Point
Direction	Up
Decimals	4
Model	Linear
Filter 1	570 nm
Sample Blanking	No after Reagent 1
Calibration Code	CYN4

Method Code: WCYN **Range: 0.005 to 0.5 mg/L CN**	**Volume uL**	**Delay Time sec.**	**Read Time sec.**	**Rinse uL**	**Code**
Sample Volume	150				
Reagent 1: Sodium Phosphate	63	36	0	0	CNSP
Reagent 2: Chloramine-T	15	72	0	0	CNCL
Reagent 3: Color Reagent	150	0	504	0	CNPY

12.3.4. Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial Calibration Standards
CCB
CCV
ICV
ICB
CCV
CCB
Method Blank
LCS
Client Samples, MS, MSD
CCV
CCB
Client Samples, MS
CCV
CCB

12.3.5. Samples that exceed the linear range must be reanalyzed at a dilution or over range concentration must be qualified as estimated. Dilutions are made with 0.25N NaOH Diluent Solution.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limits	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (mg/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = Cyanide concentration from instrument in mg/L
V_f = Final volume in Liters
D = Dilution factor of prepared aqueous sample or of solid sample extract
V_i = Initial volume in Liters
W_s = Weight of solid sample extracted in kilograms

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Standard cyanide solutions are purchased as certified standards.

19.2. Determination of cyanide is adapted for the automated spectrophotometric method instead of titrimetric or manual spectrophotometric specified in Method 9014.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846 Methods 9013A and 9014.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-129-rev.04	1. Table 9.1: recipe for Potassium Phosphate buffer revised.	29Oct2012
S-IN-I-129-rev.05	1. Converted to 27-section format. 2. Cover page: changed phone number, revised effective date format and revised document control format. 3. Section 6.1: updated treatment for chlorine in samples. 4. Section 9.1: updated instrument details changing Lachat to SmartChem. 5. Section 10.1: updated reagents for SmartChem method. 6. Table 10.3: updated standards used for SmartChem method. 7. Section 10.2.3: updated standard preparation. 8. Section 11: added ICB. 9. Section 12: added preparation of batch QC per matrix, changed instrument set-up details for SmartChem method and removed calculations to Section 14. 10. Table 13.1: updated MS frequency and removed calculations to Section 14.	11Jan2016
S-IN-I-129-rev.06	1. Table 7.1: revised storage temperature format. 2. Section 9.2: added syringe filters. 3. Section 10.1: updated reagent details. 4. Table 10.3: updated stock calibration standard details. 5. Section 12: updated volumes and weights to match current practices. 6. Table 13.1: updated LCS corrective action. 7. Section 14: updated equations to be in like terms. 8. Section 25.3: added years 2003 and 2009 to TNI reference.	20Jun2017

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

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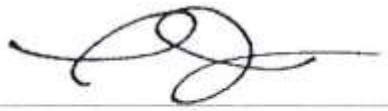

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Table of Contents

1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1 Introduction to Pace

1.1.1. Pace Analytical Services, LLC is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the 2003 NELAC Standard, 2009 TNI Standard, ISO/IEC 17025 Standard and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**

- **Flexible Response To Demand**
- **Pursue Opportunities Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environmental where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The lab is required to appoint deputies for key managerial personnel. These deputies must be documented for auditing purposes. The deputies, by position, are the following:

1.7.6.1. Deputy for Senior General Manager – Chief Operating Officer

1.7.6.2. Deputy for General Manager – Senior General Manager

1.7.6.3. Deputy for Organics Technical Director – Organics Laboratory Manager

1.7.6.4. Deputy for Inorganics Technical Director – Inorganics Laboratory Manager

1.7.6.5. Deputy for Senior Quality Manager – Senior General Manager

1.7.6.6. Deputy for Quality Manager – Senior Quality Manager

1.7.6.7. Deputy for Client Services Manager – Client Services Supervisor

1.7.6.8. Deputy for Administrative Business Manager – Administrative Assistant

1.7.6.9. Deputy for Project Managers – Client Services Supervisor

1.7.7. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Sample Preparation
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Microbiological Analysis
- Bioassay Analysis

1.7.8. The organizational structure for Pace – Minneapolis, Billings, Virginia, & Duluth, are listed in Attachments IIA-IID. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8 Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.3. Assistant General Manager / Operations Manager

- In the absence of the SGM/GM, performs all duties as listed above for the SGM or GM;
- Oversees the daily production and quality activities of all departments;
- Manages all departments and works with staff to ensure department objectives are met;
- Works with all departments to ensure capacity and customer expectations are accurately understood and met;
- Works with SGM/GM to prepare appropriate budget and staffing plans for all departments;

- Responsible for prioritizing personnel and production activities within all departments;
- In the absence of a General Manager, presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Performs formal and informal performance reviews of departmental staff.

1.8.4. Senior Quality Manager

- Provides quality oversight for multiple laboratories where there is not a local quality manager or for labs where there are multiple and separately distinct quality systems in the same facility;
- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The SQM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains the currency of the Quality Manual.

1.8.5. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality. They may also report to a Senior Quality Manager (SQM);
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The QM is responsible for

reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;

- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains the currency of the Quality Manual.

1.8.6. Technical Director

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.7. Administrative Business Manager

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.8. Client Services Manager

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;
- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.9. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody.

1.8.10. Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.11. Additional job descriptions are available upon request from the laboratory ABM.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based training system. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;

- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);
- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;
- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in SOP S-MN-Q-279 **Training and Employee Orientation** or its equivalent revision or replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff.

1.11.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees.

1.11.3. Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out an associated internal chain of custody.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.1.2. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-MN-Q-270 **Review of Analytical Requests** or its equivalent revision or replacement.

2.2. Sampling Materials and Support

2.2.1. Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed

2.2.2. Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are

recorded on the chain of custody in the "relinquished" and "received by" sections. All information except signatures is printed.

2.3.4. Additional information can be found in SOP S-MN-C-001 **Sample Management** or its equivalent revision or replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.2.1. For Ohio VAP samples, the narrative for any report that includes qualified data must also include a discussion of any bias in the results when requirements outlined in the SOP cannot be performed, for example: insufficient volume for re-extraction/re-analysis or incorrect preservative.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2^{o}C of the required temperature, or within the method-specified range. For samples with a required temperature of 4^{o}C, samples with a temperature ranging from just above freezing to 6^{o}C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice.

Any samples that are not received at the required temperature will not be processed without prior client approval.

- Samples for **drinking water** analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).
- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to ± 0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ± 0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤ 6°C limit. Please reference the Support Equipment SOP for more information.

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

Note 3: Biological Tissue Samples must be received at the following temperature based on program and contract: cooled to ≤ 6°C during the first 24 hours after collection; then samples must be kept frozen at ≤ - 10°C. TNI rules also apply if the samples are brought straight from the field; they are acceptable if evidence of cooling is present (i.e., received on ice).

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.4.5. Additional information can be found in SOP S-MN-C-001 **Sample Management** or its equivalent revision or replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions

- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 12:00 am as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.5.3. For DoD work, if the time of the sample collection is not provided, the laboratory must assume the most conservative time of day. This is defined as 12:01am.

2.5.4. The LIMS automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of Work Order Number-Sample Number, for example 12345678-001, where the Work Order Number is 12345678 and 001 is the sample number. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.5.5. Sample labels are printed from the LIMS and affixed to each sample container.

2.5.6. Samples with holding times that are near expiration date/time may be brought directly to the laboratory for analysis at the discretion of the Project Manager and/or SGM/GM/AGM/OM.

2.5.7. Additional information can be found in SOP S-MN-C-001 **Sample Management** or its equivalent revision or replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in SOP S-MN-C-001 **Sample Management** or its equivalent revision or replacement and in SOP S-MN-S-003 **Waste Handling and Management** or its equivalent revision or replacement.

2.6.2. **Storage Conditions**

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Storage blanks, consisting of two 40mL aliquots of reagent water, are stored with volatile samples and are used to measure cross-contamination acquired during storage.

2.6.2.3. Additional information including procedures and criteria for evaluating storage blanks where applicable can be found in SOP S-MN-Q-263 **Monitoring Temperature Controlled Units.**

2.6.3. **Temperature Monitoring**

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

2.6.3.2. The temperature of each refrigerated storage area is maintained at ≤ 6°C (but above freezing) unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at ≤ -10°C unless state, method or program requirements differ. The temperature of each storage area is checked and documented each day of use (each calendar day). Additional information, including corrective actions for temperatures outside of acceptance limits, can be found in SOP S-MN-Q-263 **Monitoring Temperature Controlled Units.**

2.6.4. **Hazardous Materials**

2.6.4.1. Samples designated by clients upon receipt as pure product, potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.6.4.2. Additional information regarding hazardous waste handling can be found in the laboratory's SOP for **Waste Handling and Management** S-MN-S-003, or its equivalent replacement.

2.6.5. **Foreign/Quarantined Soils**

2.6.5.1. Foreign soils and soils from USDA regulated areas must be adequately segregated to enable proper sample disposal. The USDA requires these samples to be treated by an approved procedure. Additional information regarding USDA regulations and sample handling can be found in the laboratory's SOP for **USDA Regulated Soil Handling** S-MN-Q-253, or its equivalent replacement.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-MN-C-004, **Subcontracting Samples** or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace Analytical work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in SOP S-MN-C-004 **Subcontracting Samples** or its equivalent revision or replacement.

2.7.5. Subcontracted labs used for DoD work must be accredited by DoD or its designated representatives. Subcontracted labs must receive project specific approval from the DoD client

before any samples are analyzed. These requirements also apply to the use of any laboratory under the same corporate umbrella, but at a different facility or location.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

- Air canisters are submitted for cleaning upon data validation. Due to media capacity, air canister samples are not retained as standard environmental samples.

2.8.2. Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be stored at ambient temperature when the hold time is expired; the report has been delivered, and/or allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of **hazardous** samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in SOP's S-MN-S-003 **Waste Handling and Management** and S-MN-C-001 **Sample Management** or their equivalent replacements.

3.0. QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or final reports must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.1.5. For Ohio VAP projects, the laboratory must minimize the use of qualified data. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies prior to reporting. When requirements outlined in the SOP cannot be performed, the narrative for any report that includes qualified data must also include a discussion of any bias in the results.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.2.3. For DoD samples, the method blank will be considered to be contaminated if: 1) The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater; 2) The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater or 3) The blank result otherwise affects the sample results as per the test method requirements or the project-specific objectives. If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

3.2.4. For Ohio VAP projects, the laboratory must minimize the use of qualified data. In the case of method blank having any reportable contamination, the laboratory is required to reanalyze the associated samples with an acceptable method blank if there is sufficient sample remaining. Acceptable method blanks are those that are free of contamination below the reporting limit. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding method blanks for Ohio VAP projects. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains **all** analytes required by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. The lab must ensure that all target components are included in the spike mixture for the LCS over a two (2) year period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
    - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
    - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;
    - For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

Note: the use of marginal exceedances is not approved for work from the state of South Carolina.

3.3.5. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria (this is a TNI allowance). When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS (if possible) or reported with appropriate data qualifiers.

Note: This is not approved for South Carolina. South Carolina samples must be re-extracted and re-analyzed to report data with no recoveries exceeding limits.

3.3.6. For Ohio VAP projects, the laboratory must minimize the use of qualified data. In the case of LCS failures, the laboratory is required to reanalyze the associated samples with an acceptable LCS for all target compounds if there is sufficient sample remaining. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for Ohio VAP projects. The narrative for any report that includes qualified data must also include a discussion of any bias in the results. The MS may not be used in place of passing LCS for Ohio-VAP projects.

3.3.7. For DoD projects, the laboratory is not allowed to have any target analytes that exceed DoD LCS control limits. In the case of LCS failures, the laboratory is required to reanalyze the associated samples with an acceptable LCS for all target compounds if there is sufficient sample remaining. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for DoD projects. All LCS failures must be accounted for in project case narratives. See applicable method SOPs for further corrective action.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section. However, the lab must ensure that all targeted components are included in the spike mixture for the MS/MSD over a two (2) year period.

3.4.3. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

3.4.4. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.4.5. For Ohio VAP projects, MS/MSD's are optional and will be directed by the Certified Professional. The laboratory must minimize the use of qualified data. In the case of MS/MSD failures, the laboratory is required to reanalyze the associated samples only when the associated LCS also fails acceptance criteria and if there is sufficient sample remaining. When an LCS is acceptable and the MS results are outside of criteria, and no system anomaly is detected, the samples will be reported with appropriate data qualifiers indicating matrix interference. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for Ohio VAP projects.

3.4.6. For DoD work, each preparation batch of samples must contain an associated MS and MSD (or sample duplicate) using the same matrix collected for the specific DoD project. If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative. Additional MS/MSDs may be required on a project-specific basis. The MS/MSD must be spiked with all target analytes with the exception of PCB analysis, which is spiked per the method. The

concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate concentration of concern. Multiple spiked samples may need to be prepared to avoid interferences.

3.4.7. For DoD work, the results of all MS/MSD must be evaluated using the same acceptance criteria used for the LCS.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.5.3. For Ohio VAP projects a sample duplicate is optional and will be directed by the Certified Professional. The laboratory must minimize the use of qualified data. In the case of duplicate samples exceeding the RPD criteria found in applicable analytical SOPs, the laboratory is required to reanalyze the associated sample and duplicate as long as no sampling error was detected if there is sufficient sample remaining. If the sample and duplicate still do not agree, a comment would be made stating there may be sample non-homogeneity. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding sample duplicates for Ohio VAP projects. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to measure the extraction or purge efficiency and to monitor the effect of the sample matrix on compound recovery.

3.6.1.1. For Ohio VAP samples, the narrative for any report that includes qualified data must also include a discussion of any bias in the results.

3.6.1.2. For the TO-15 method, surrogates are not evaluated for Ohio VAP samples.

3.6.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.7.3. For Ohio VAP projects, samples with internal standard that are outside of method criteria must be reanalyzed to confirm sample matrix effect. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding internal standards for Ohio VAP projects. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories uses a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

3.8.3. The LOD is determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity.

3.8.3.1. Where specifically stated in the published method, LODs or MDLs will be performed at the listed frequency. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

3.8.4. For Ohio VAP projects, a valid MDL must be in place prior to sample analysis. MDLs must be spiked at or below the reporting limit and will not be accepted if it was spike higher than the reporting limit.

3.8.5. DoD definition for LOD - The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate is 1%.

3.8.6. Additional information can be found in SOP S-MN-Q-269 **Determination of LOD and LOQ** or its equivalent revision or replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. Results below the reporting limit are not allowed to be reported without qualification. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. For DoD approved methods, the LOQ and LOD shall be verified quarterly and valid LOQ must be in place prior to sample analysis.

3.9.3. Additional information can be found in SOP S-MN-Q-269 **Determination of LOD and LOQ** or its equivalent revision or replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-MN-Q-255 **Estimation of Measurement Uncertainty** or its equivalent revision or replacement.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

3.11.2. Additional information can be found in SOP S-MN-Q-258 **Proficiency Testing Program** or its equivalent revision or replacement.

3.12. Rounding and Significant Figures

3.12.1. In general, the Pace laboratories report data to no more than three significant figures. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace - Mpls, MT, & VM-Dul follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

3.12.3.1. Pace - Mpls, MT, & VM-Dul follow the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant figures
Values ≤ 10 – Reported to 2 significant figures

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within the retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute retention time windows must be calculated, unless instrument maintenance fixes the problem. When a new column is installed, a new retention time window study must be performed.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, or when the laboratory develops or modifies a method, the laboratory validates the method prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.14.2. Additional information can be found in SOP S-ALL-Q-047 **Method Validation and Instrument Verification**, or equivalent replacement.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in SOP S-MN-Q-268 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. Copies of all quality systems documentation provided to DoD for review must be in English.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 **Document Numbering**.

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and then all applicable lab staff sign the Quality Assurance Manual. Once approved, all applicable lab staff sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in SOP S-MN-Q-273 **Preparation of SOP's** or its equivalent revision or replacement.

4.1.6.4. For Ohio VAP certification, it is required by the Ohio Administrative Code that the laboratory must seek Ohio VAP review and approval of all SOPs and Quality Manual subsequent modifications prior to implementation.

4.1.6.5. For DoD approval, all technical SOPs are reviewed for accuracy and adequacy annually and whenever method procedures change and updated as appropriate. All such reviews are documented and made available for assessment. Non-technical SOPs that are not required elements of the quality system are considered administrative SOPs and are not required elements

of the quality system are considered administrative SOPs and are not required to be reviewed annually.

4.2. Document Change Control

4.2.1. Additional information can be found in SOP S-MN-Q-268 **Document Control and Management** or its equivalent revision or replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. All prepared standard or reagent containers include the Pace identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials, unless the container is too small to hold all of this information. This ensures traceability back to the standard preparation logbook or database.

5.1.5. All initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Additional information concerning the procurement of standards and reagent and their traceability can be found in the SOP S-MN-Q-275 **Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. All applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative.

5.2.2.1 For Ohio VAP projects, samples must be reanalyzed to obtain results within the linear range unless there is insufficient sample volume for reanalysis

5.2.3. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

5.2.4. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.5. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in SOP S-MN-Q-264 **Support Equipment** or its equivalent revision or replacement.

5.3.2. On each day the support equipment is used, it is verified, as applicable, in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately. This applies mainly to thermometers within temperature-controlled units and balances.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified every 5 years at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the local Quality department.

5.3.4. **Thermometers**

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified, at a minimum, every 3 years with equipment directly traceable to NIST.

5.3.4.2. Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures (working digital thermometers are calibrated quarterly). Each thermometer is individually numbered and assigned a correction factor based on the NIST reference

source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. **pH/Electrometers**

5.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions. See the analytical method SOP's for more specific information.

5.3.6. **Spectrophotometers**

5.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

5.3.7. **Mechanical Volumetric Dispensing Devices**

5.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-MN-Q-264 **Support Equipment** or its equivalent revision or replacement.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

5.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)

- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical, field, or product testing data is generated, it is documented appropriately. These logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. Data review checklists, either hardcopy or electronic, are used to document the primary data review process. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in SOP S-MN-L-132 **Data Review Process** or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 **Manual Integration** or its equivalent revision or replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer (this cannot be the primary analyst). The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations. Data review checklists, either hardcopy or electronic, are used to document the secondary data review process.

6.2.3. Some reports and/or data packages may be reviewed by the QM or SQM or designee based on program requirements (e.g., DoD) or client requirements. In this case a thorough review for completeness and accuracy may include a compilation of raw data and QC summaries in addition to the final report to produce a full deliverable package. In the case of DoD, 100% of all packages must have a final administrative review (to confirm that primary and secondary reviews were completed and documented and that data packages are complete) and 10% of all data packages must be reviewed by the Quality Manager for technical completeness/accuracy. This 10% review can be done after the data packages have been submitted to the clients.

6.2.4. Additional information regarding data review procedures can be found in SOP S-MN-L-132 **Data Review** or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 **Manual Integration** or its equivalent revision or replacement.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. Please reference SOP S-MN-C-007 **Final Reports and Deliverables**, or its equivalent revision or replacement.

6.3.3. Additional items may be required per client QAPPs or different state regulations.

6.3.3.1. Ohio VAP requires an Affidavit that must summarize if there are any exceptions to what has been reported, this includes, but is not limited to, itemizing any analytes that the laboratory is not approved for under the VAP program. Any analytes reported that are not part of a scope of accreditation or approval program must be clearly indicated on the final report and associated paperwork such as an Affidavit.

6.3.4. For DoD labs both date and time of preparation and analysis are considered essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report.

6.3.5. Any changes made to a final report shall be designated as “Revised” or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.6. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.7. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager

- Client Services Manager
- Project Manager
- Project Coordinator

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations, and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis, and personnel involved. TNI-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.5.4. Additional information regarding archiving procedures can be found in SOP S-MN-L-106 **Data Archiving and Retrieval** or its equivalent revision or replacement.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports that are disposed.

6.6.2. For Ohio VAP labs, all documents and data prepared or acquired in connection to VAP work must be retained for a period of 10 years after the data of reporting. After 10 years, if the laboratory wishes to dispose of the records, the laboratory must notify the VAP agency by certified mail of such intent and provide the agency an opportunity to request the materials from Pace. The documents must not be disposed of until notification has been received in response to the Pace request for disposal.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in SOP S-MN-Q-271 **Internal and External Audits** or its equivalent revision or replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections: 1) Raw Data Reviews, 2) traditional Quality Systems internal audits (including SOP and method compliance), 3) Final Report Reviews, and 4) Corrective Action Effectiveness Follow-up.

7.1.2.2. Internal systems audits are conducted yearly at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.3. Internal Audit Reports and Corrective Action Plans

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the

performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

7.1.3.3. Additional information can be found in SOP S-MN-Q-271 **Internal and External Audits** or its equivalent revision or replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in SOP S-ALL-Q-015 **Review of Laboratory Management System** or its equivalent revision or replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, and staffing.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.0. CORRECTIVE ACTION

Additional information can be found in SOP S-MN-Q-262 **Corrective and Preventive Actions** or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using Pace's LabTrack system or other system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The following items are examples of sources of laboratory deviations or non-conformances that may warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- Proficiency Testing Sample Results
- Internal and External Audits
- Data or Records Review
- Client Complaints
- Client Inquiries
- Holding Time violations

8.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g., matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

8.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within the lab's corrective action system. The documentation must include (as applicable): the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.4. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within the lab's corrective action system.

8.1.5. Based on the root cause(s) determined the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any ramifications to data quality if impacted (with the possibility of work being recalled).

8.2. Corrective Action Completion

8.2.1. **Internal Laboratory Non-Conformance Trends**

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

8.2.2. **PE/PT Sample Results**

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.2.2. Additional information, such as requirements regarding time frames for reporting failures to states, makeup PTs, and notifications of investigations, can be found in SOP S-MN-Q-258 **Proficiency Testing Program** or its equivalent revision or replacement.

8.2.3. **Internal and External Audits**

8.2.3.1. The SQM/QM is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. **Data Review**

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. **Client Complaints**

8.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

8.2.6. **Client Inquiries**

8.2.6.1. When an error on the customer report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

8.2.7. **Holding Time Violations**

8.2.7.1. In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the SQM/QM must be made aware of all holding time violations.

8.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.

American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. For South Carolina – batch is defined the same as TNI with the exception of an 8 hour start to finish of processing samples rather than 24 hours.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.

Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. Time of Analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g. extractions and incubations). For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).

Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.

Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).

Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.

Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.

Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.

Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.

Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP)). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.

Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

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10.21. UCMR Laboratory Approval Requirements and Information Document, most current version.

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11.0. REVISIONS

The Pace Corporate Environmental Quality Office files an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to previous versions of the Quality Assurance Manual. This document is available upon request. All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 19.0	General: made administrative edits that do not affect the policies or procedures within the document (including revising company name to Pace Analytical Services, LLC). Cover page: removed corporate approval signature lines. Implemented QAM 19.0 SOT.	25May2017
Quality Assurance Manual 19.1	Removed all references and attachments for Davis laboratory. Updated Approval personnel for all labs per current org charts. 1.9.2 – updated SOP number to new local SOP number. 2.5.4 – updated "XXXX (insert LIMS sample numbering convention)" to "Work Order Number-sample……sample number." 2.6.1 – updated from SOT numbers to local SOP numbers. Added section 3.1.5 for Ohio VAP. 3.14.2 & 6.1.3 – updated to new corporate SOP number instead of retired local SOP. 6.2.4 – updated from SOT number to local SOP number, added S for SOP title Section 9.0, Holding Time row – added "Time of Analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g. extractions and incubations). Updated all attachments to most current versions.	16May2018

ATTACHMENT I - QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I - QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

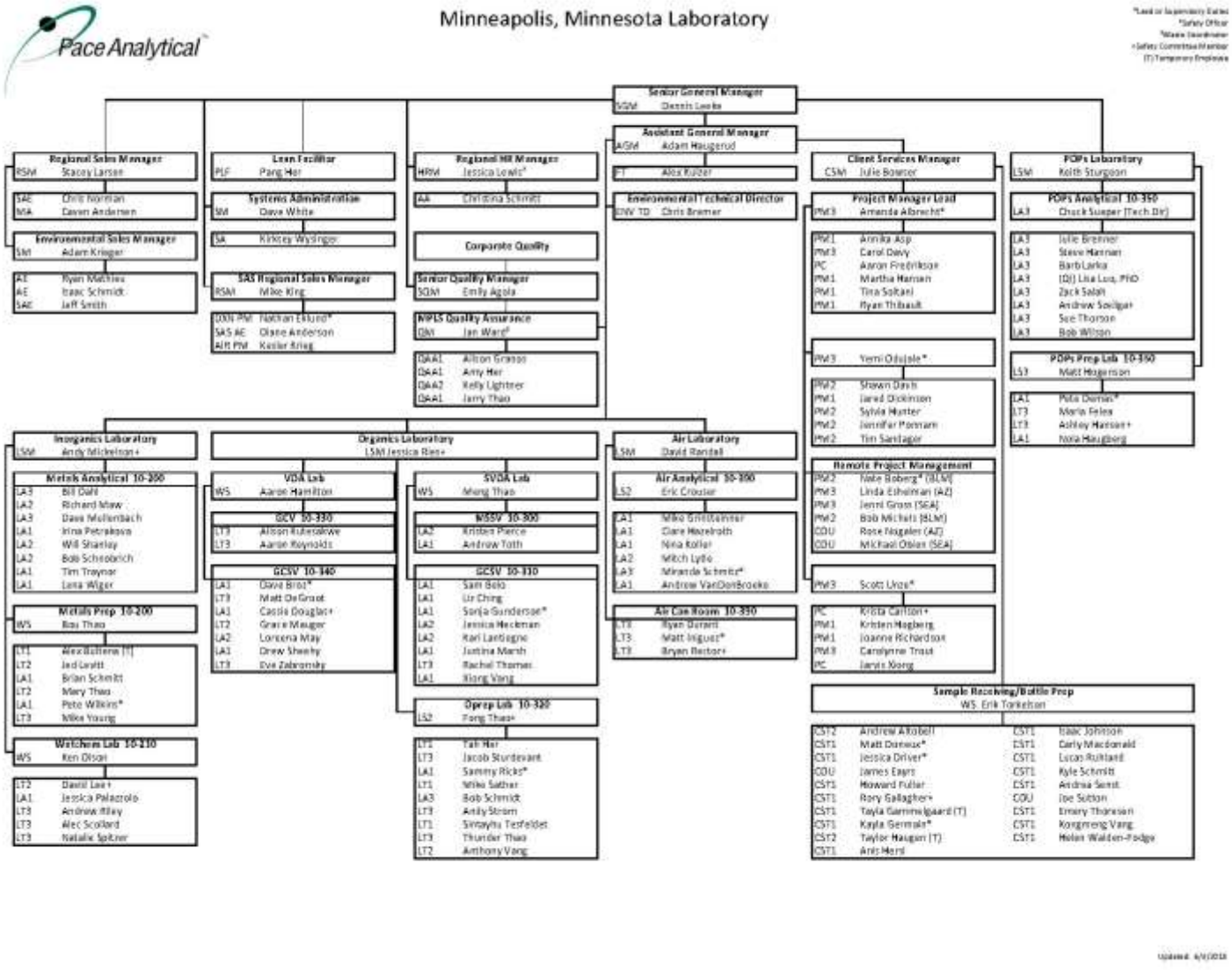
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

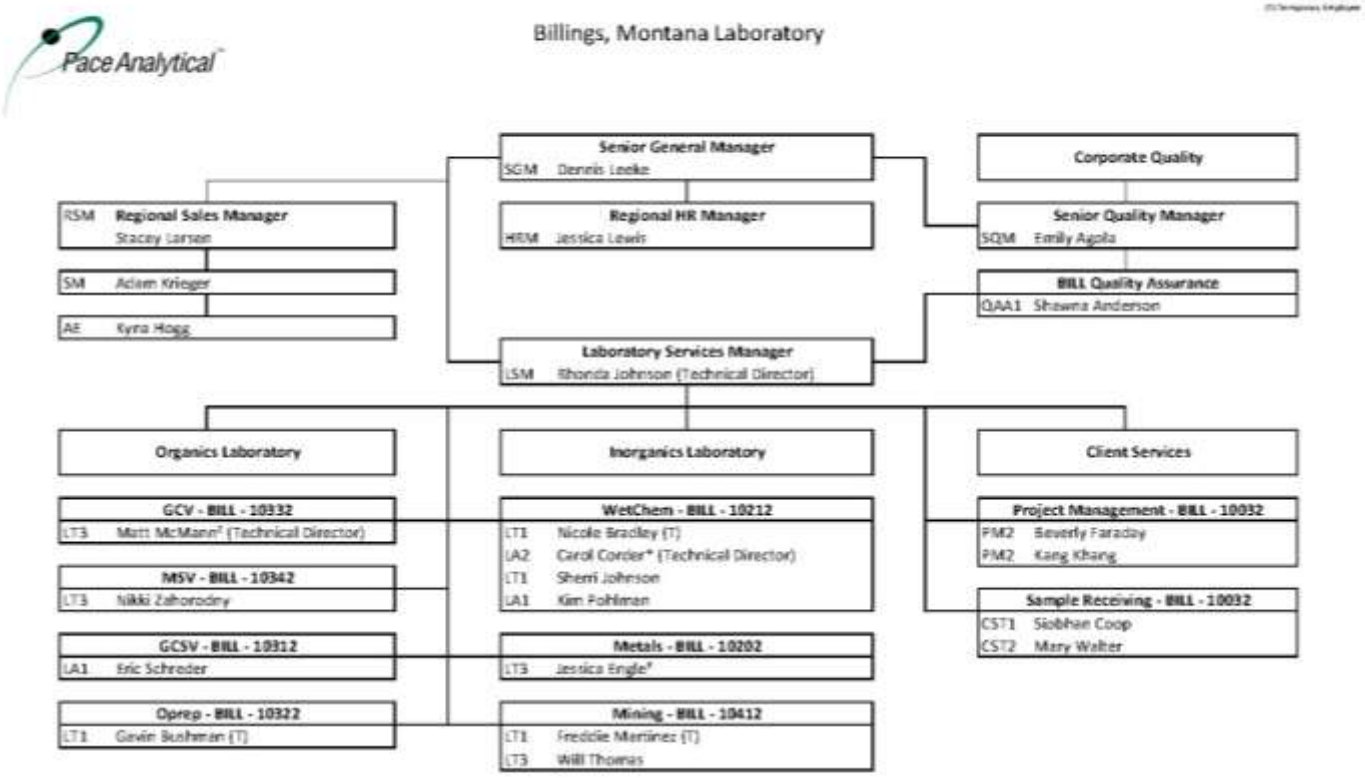
$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

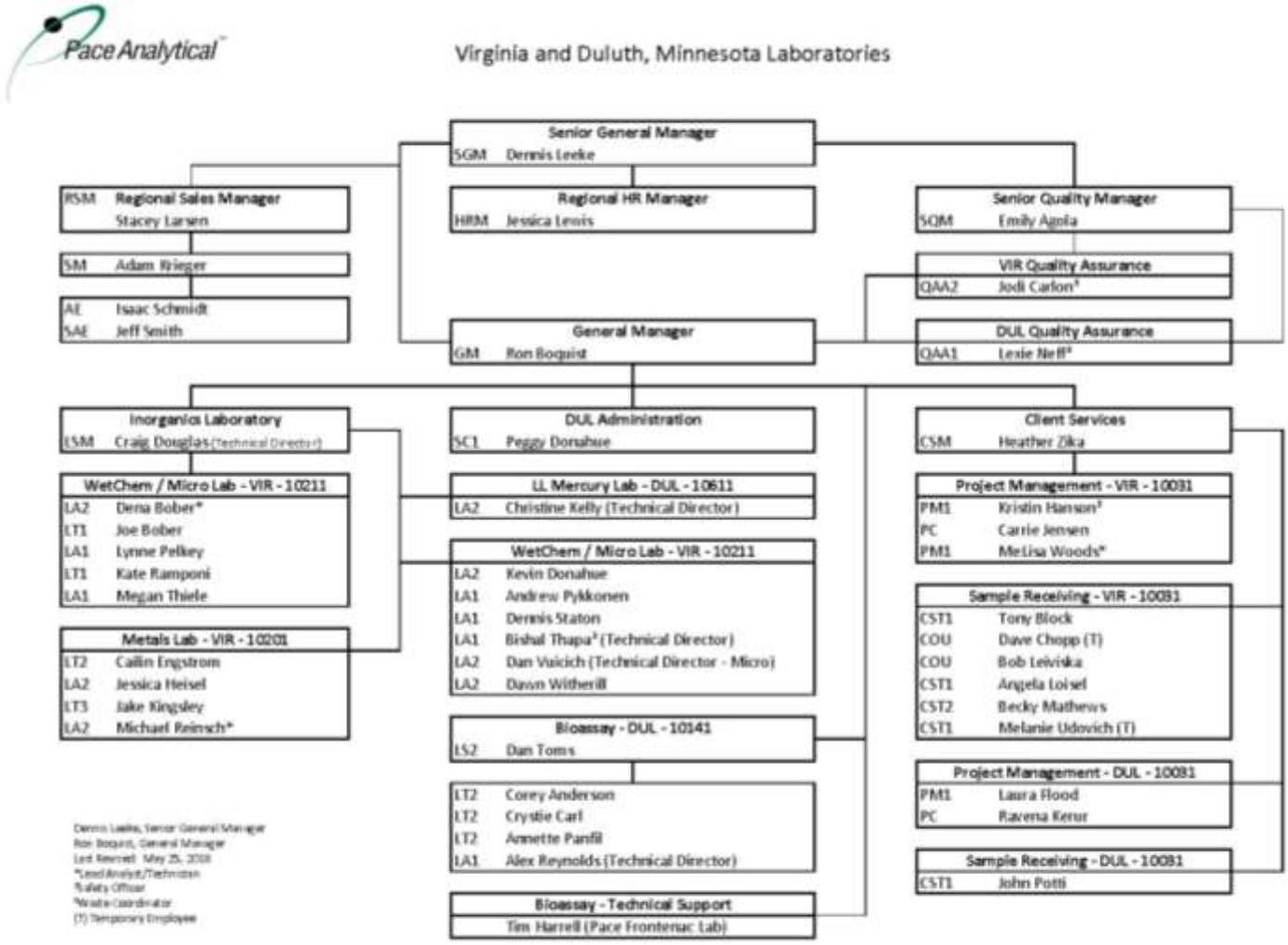
ATTACHMENT IIA - MINNEAPOLIS LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



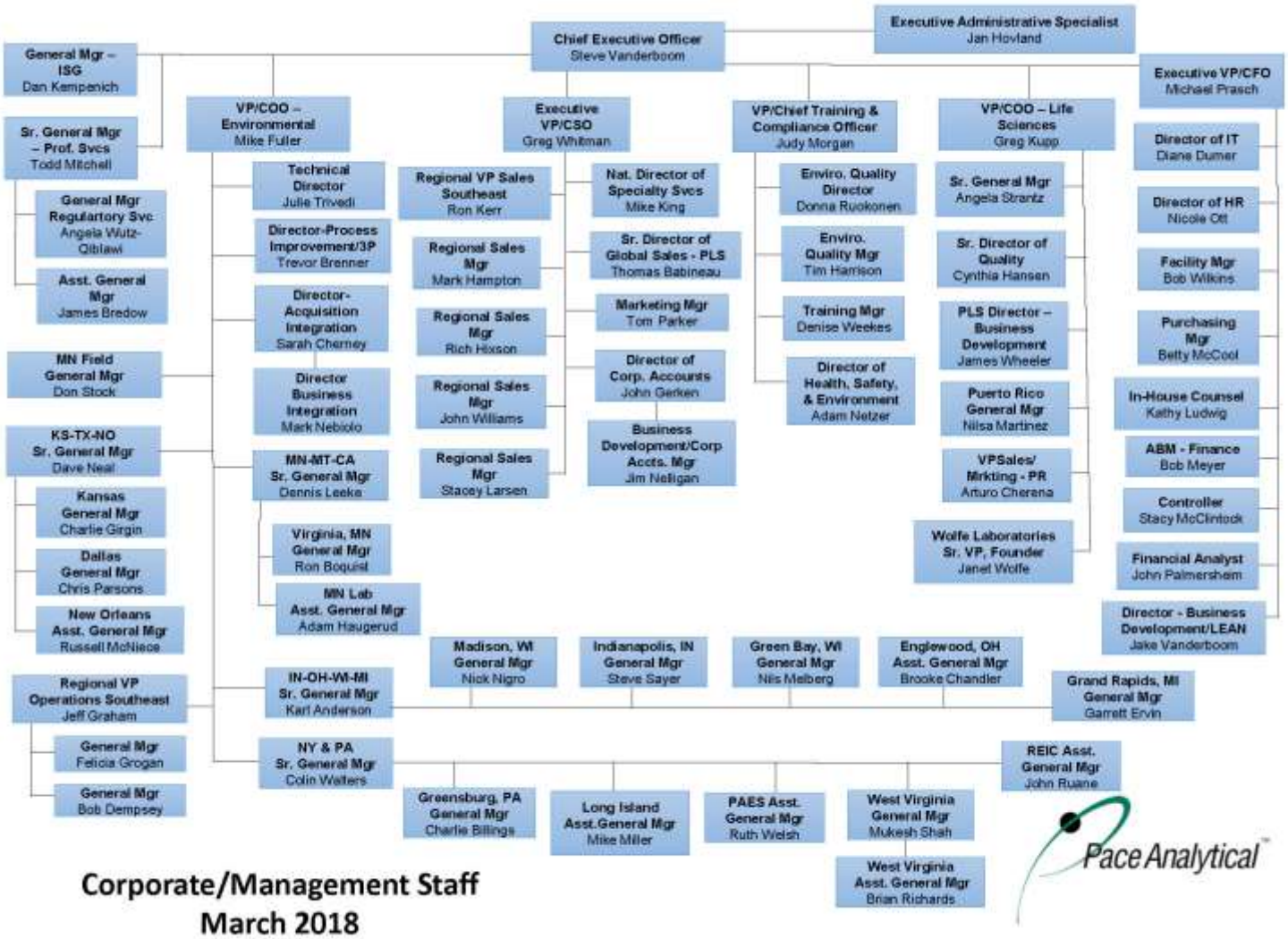
ATTACHMENT IIB - MONTANA LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT IIC - VIRGINIA AND DULUTH LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

INSTRUMENT	Pace ID	SERIAL #	MANUFACTURER	MODEL
GC	10AIR0	CN10429060	Agilent Technologies	6890N
MS	10AIR0	US43146819	Agilent Technologies	5973 Network
Concentrator	10AIR0	1343	Entech Instruments, Inc.	7100A
GC	10AIR5	2843A20766	HP	5890
GC	10AIR7	CN10429056	Agilent Technologies	6890N
MS	10AIR7	US43146821	Agilent Technologies	5973 Network
Concentrator	10AIR7	1298	Entech Instruments, Inc.	7100A
GC	10AIR9	US00002531	Agilent Technologies	G1530A
Headspace Sampler	10AIR9	IT00507022	Agilent Technologies	G1888
GC	10AIRA	US00034289	ALS Ready	6890A
Concentrator	10AIRA	1150	Entech Instruments, Inc.	7100A
MS	10AIRB	US44621387	Agilent Technologies	5973 inert
GC	10AIRB	CN10517058	Agilent Technologies	6890
Concentrator	10AIRB	U22038	Markes	Unity2
Autosampler	10AIRB	GB00g-10131/GBOOH-70106	Markes	CIA Advantage/CIA Satellite
GC	10AIRD	CN10742037	Agilent Technologies	7890A
MS	10AIRD	US73317788	Agilent Technologies	5975C
Concentrator	10AIRD	1563	Entech Instruments, Inc.	7100A
Autosampler	10AIRE	CN10020012	Agilent Technologies	7693
MS	10AIRE	US10407503	Agilent Technologies	5975C
GC	10AIRE	CN10241030	Agilent Technologies	7890A
Thermal Desorber	10AIRE	L1009271	Perkin Elmer	Turbomatrix 650
Canister Autosampler	AIR7T1	1240	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIR7T2	1069	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIRBT1	1239	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIRBT2	1158	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIR0T1	1068	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIR0T3	1141	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIRD	1284	Entech Instruments, Inc.	7016 CA
Canister Autosampler	AIRD	1283	Entech Instruments, Inc.	7016 CA
Can Cleaning Rack	Rack 1	na	Pace	na
Can Cleaning Rack	Rack 2	na	Pace	na
Can Cleaning Rack	Rack 3	na	Pace	na
Refrigerator/Freezer	A4	DK25BZ	Keystone	KSTRC312AW
Oven	10AIR10	149432	Despatch	LDB Series

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Tube Conditioner/Dry Purger	10AIR24	820R4051501	Perkin Elmer	Turbomatrix TC220
GCMS	10AIRF	648N1031001	PerkinElmer	Clarus SQ 8 C
GCMS	10AIRG	US00040933	NA	6890A
GC	10AIRH	CN10803059	Agilent Technologies	7890A
MS	10AIRH	US80848612	Agilent Technologies	5975C
Preconcentrator	10AIRH	1450	Entech Instruments, Inc.	7200
Autosampler	10AIRH	1586	Entech Instruments, Inc.	7016D
Autosampler	10AIRH	1579	Entech Instruments, Inc.	7016D
GCMS	10MSHR14	CN10705008	Waters/Micromass	Autospec
Autosampler	10MSHR14	CN21920651	Waters/Micromass	Autospec
GCMS	10MSHR14	M590	Waters/Micromass	Autospec
Freezer	H2	080200474	Kenmore	
Freezer	H1	01206544	NA	NA
GCMS	10MSHR09	US10544001	Agilent	6890N
GCMS	10MSHR09	P669	Waters/Micromass	Autospec Premier
GCMS	10MSHR06	US00033386	Agilent	6890A
GCMS	10MSHR06	M496	Waters/Micromass	Autospec Ultima
GCMS	10MSHR12	P808	Waters/Micromass	Autospec Premier
Autosampler - Y	10MSHR12	280399	Waters/Micromass	Autospec
GCMS	10MSHR12	CN10471195	Agilent	NA
GCMS	10MSHR12	CN11301038	Agilent	NA
GCMS	10MSHR05	US00036565	Agilent	6890A
GCMS	10MSHR05	M488	Waters/Micromass	Autospec Ultima
Autosampler F	10MSHR05	280398	Waters/Micromass	Autospec
LC-MS/MS	10LCMS01	V23210806	Sciex	4000
LC-MS/MS	10LCMS02	V1390304	Sciex	4000
Refrigerator	DP1	950804979	Kenmore	564.9932910
Freezer	DP1	950804979	Kenmore	564.9932910
Freezer	DP2	W834049450	Kenmore Elite	NA
Micro 100 Turbidimeter	10HR14	201309191	Scientific Inc.	Micro 100 Turbidimeter
Microwave extraction	10HR13	M09903	CEM	MarsXpress
Accelerated Solvent Extractor	10HR12	1020363	ACE	200
N-EVAP	DW1	57966	Organomation	8125
N-EVAP	DW2	57529	Organomation	8125
N-EVAP	N-EVAP 4	57964	Organomation	8125
N-EVAP	N-EVAP 5	57410	Organomation	8125
N-EVAP	N-EVAP 6	57527	Organomation	8125
N-EVAP	N-EVAP 7	57528	Organomation	112
Hypersep Vaccuum Manifold	10HR15	1632	Thermo Scientific	60104233
Hypersep Vaccuum Manifold	10HR16	1552	Thermo Scientific	60104233
Oven	DP4	O06M-568117-RM	Lindberg Blue	GO1340A-1
Freezer	DP5	EWR223703	Kenmore	NA
freezer	DP6	AS0115A228W20498	SPT	UF-214W

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

ICPMS	10ICM3	SN-01298-C	Thermo Scientific	Xseries 2
ICPMS - autosampler	10ICM3	071778A560	Teledyne Cetac	ASX560
ICPMS - chiller	10ICM3	110140001120717	Thermo	NA
ICPMS - pump	10ICM3	31001424325	SOGEVAC pump	NA
ICPMS	10ICM8	5P13142395	Aglient 7700	G3281A
ICPMS - autosampler	10ICM8	US011191A520	Teledyne Cetac	ASX520
ICPMS - chiller	10ICM8	2U1551028	Agilent	NA
ICPMS - pump	10ICM8	129449393	Edwards	NA
ICPMS	10ICM9	JP12412084	Aglient 7700	G3281A
ICPMS - autosampler	10ICM9	US0312120AS520	Teledyne Cetac	ASX520
ICPMS - chiller	10ICM9	3U1621341	Agilent	NA
ICPMS - pump	10ICM9	169436540	Edwards	NA
ICPMS	10ICMB	JP16120262	Agilent ICPM	7800
ICP	10ICP4	MY14160002	Agilent Technologies	700 Series-ICP-OES
ICP - autosampler	10ICP4	12140A520	Teledyne Cetac	ASX520
ICP - chiller	10ICP4	1B13C1081	Agilent	NA
ICP	10ICP5	MY15180003	Agilent Technologies	5100 -ICP-OES
ICP - autosampler	10ICP5	AU15140009	Agilent	SPS4
ICP - chiller	10ICP5	1A1550426	Agilent	NA
Mercury Analyzer - being repaired	10HG3	111003QTA	Cetac Quick Trace	M-7500
Mercury Autosampler	10HG3	061005A520	ASX-520	MAS Ver w/Diluter
Mercury Analyzer	10HG4	06201Q76	Cetac	M7600
Mercury Autosampler	10HG4	061289A520	Cetac	AX-520
Mercury Analyzer	10HG08	US15254007	Cetac	M7600
Mercury Autosampler	10HG08	0315134A520	Cetac	ASX-520
Hot Block	10MET02	6266CECW2910	Environmental Express	SC154
Hot Block	10MET04	6083CECW2815	Environmental Express	na
Hot Block	10MET08	8031CECW3358	Environmental Express	NA
Hot Block	10MET10	8031CECW3346	Environmental Express	NA
Hot Block	10MET22	5388CEC2469	Environmental Express	SC154
Hot Block	10MET23	8708CECW3720	Environmental Express	SC154
Hot Block	10MET26	8793CECW3764	Environmental Express	SC154
Hot Block	10MET09	8031CECW3342	Environmental Express	NA
Hot Plate	10MP02	n/a	Cole Parmer	n/a
Hot Plate	10MP03	n/a	Cole Parmer	n/a
TCLP agitator/tumbler	10MET34	0685RKME0010	Analytical Testing Corp	DC-20
Hot Plate/hot block	10MET35	1073970926967	Thermolyne	HP47135
Turbidity Meter	10MP04	08040C029534	Hach	2100P
pH meter	10MP05	10240	Scientific Instruments	IQ180GLP
pH meter	10MP06	1343	Orion Research	Expandable Ion Analyzer EA 940
Tumbler	10MET36	0685SAMH002	Analytical Testing Corp	42R5BFC1-E3
Water Bath	10MET37	302N0020	Fisher Scientific	128
Tumbler	10MET38	0685SGMP0002	Analytical Testing Corp	42R5BFC1-E3

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Fridge	MP1	4316063619504	Danby Designer	DBC120BLS
Tumbler	10MET39	0685SGMQ0006	Analytical Testing Corp	42R5BFC1-E3
Turbidity Meter	10MP08	201101226	HS Scientific	MicroTPW
pH meter	10MP07	2404439	Oakton	pH700
Oven/Desiccator	10MET40	903N0075	Fisher Isotemp	NA
Tumbler	10MET43	NA	MilliporeSigma	YT310RAHW
Oven - moved 07.07.15	10WET20	510N0239	Fisher Scientific	Isotemp Oven
Oven	10WET49	1589080190130	Fisher Scientific	851F
Stir plate	10MET44	C272000401175991	Fisher Scientific	S88857200
Oven/Desiccator	10MET41	903N0078	Fisher Isotemp	NA
Centrifuge	10MET45	42243876	ThermoScientfic	Legend XT
UltraSonicator	10OP17	RPC10096911F	Branson	8510
Sonicator	10OP01	G3914	Misonix	XL 2020
Sonicator	10OP02	G4180	Misonix	XL 2015
Sonicator	10OP04	R1638	Misonix	Sonicator 3000
Soxtherm	10OP06	8465 08 0003	Gerhardt	na
Soxtherm	10OP07	1/8465 08 0005	Gerhardt	na
Soxtherm	10OP08	1/8465 08 0002	Gerhardt	na
Soxtherm	10OP09	1/8465 08 0003	Gerhardt	na
N-EVAP	10OP10	8169	Organomation	112
N-EVAP	10OP11	7537	Organomation	112
Refrigerator	OP1	T34931C10	Traulsen	NA
Centrifuge	10OP13	31210390	IEC	Centra GP8
Centrifuge	10OP14	9304	Damon/IEC Division	na
Centrifuge	10OP15	28899M	International Clinical Centrifuge	CL28899M
N-EVAP	10OP18	4185	Organomation	II2
Turbo Vap	10OP20	TV0910015115	Caliper Life Sciences	Turbo Vap II
Buchi Concentrator-vacuum controller	10OP21	10000162387	Buchi Labortenchik Ag	V-855
Buchi Concentrator-vacuum pump	10OP21	1000166230	Buchi Labortenchik Ag	V-700
Buchi Concentrator-Recirculating Chiller	10OP21	1019513	Buchi Labortenchik Ag	F-108
Buchi Concentrator System	10OP21	1000167481	Buchi Labortenchik Ag	Q101
Microwave extraction	10OP19	MD3483	CEM	MarsXpress 230/60
Smart System 5 Intella-tempcalibrator	10OP19	B15683	Smart System	4014
Line Conditioner TSI Power VRp series	10OP19	13060111	Tsi Power	VRp-3000-0238
Sonicator	10OP23	NA	Bransonic	B8200R-3
Sonicator	10OP22	G1879	Heat Systems	XL2020
Buchi Concentrator-vacuum controller	10OP24	1000171188	Buchi Labortenchik Ag	V-855
Buchi Concentrator-vacuum pump	10OP24	1000176128	Buchi Labortenchik Ag	V-700
Buchi Concentrator-Recirculating Chiller	10OP24	1000174259	Buchi Labortenchik Ag	F-108
Buchi Concentrator System	10OP24	1000176659	Buchi Labortenchik Ag	Q101

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Buchi Concentrator-vacuum controller	10OP25	1000174543	Buchi Labortenchik Ag	V-855
Buchi Concentrator-vacuum pump	10OP25	1000176882	Buchi Labortenchik Ag	V-700
Buchi Concentrator-Recirculating Chiller	10OP25	1000172490	Buchi Labortenchik Ag	F-108
Buchi Concentrator System	10OP25	1000176601	Buchi Labortenchik Ag	Q101
Buchi Concentrator-vacuum controller	10OP26	1000171253	Buchi Labortenchik Ag	V-855
Buchi Concentrator-vacuum pump	10OP26	1000174270	Buchi Labortenchik Ag	V-700
Buchi Concentrator-Recirculating Chiller	10OP26	1000174257	Buchi Labortenchik Ag	F-108
Buchi Concentrator System	10OP26	1000176658	Buchi Labortenchik Ag	Q101
Refrigerator	OP4	T127161605028	Whirlpool	WH43S1E
Refrigerator/freezer	C13	NA	NA	NA
Refrigerator	C10	63278-01	NA	Walk-in
Refrigerator	C1	NA	NA	Walk-in
Freezer	C3	WB12555570	Frigidaire	FFU21F5HWK
Refrigerator	C17	KR48-1AS 9029136	Beverage Air	KR48-1AS
Refrigerator	C18	30692	U.S. Cooler	Walk-in/FCL3476GL1
Refrigerator	C16	34365	NA	NA
Refrigerator	C22	9199842	TRUE	GDM-47-HC-LD
Freezer	C23	MBF800307916061700C40007	ATOSA	MBF8003
Freezer	C21	WB65148072	Kenmore	22042410
Refrigerator	C24	R49S-18010046	Volition	R49-S
GC System	10MSSA	CN10021030	Agilent	7890A
Autosampler Tower	10MSSA	CN95203168	Agilent/HP	7693 Series
Autosampler Tray	10MSSA	CN10020004	Agilent/HP	7693 Series
MS Detector	10MSSA	US10030005	Agilent/HP	5975C
Peltier Cooling System	10MSSA	782005285	Gersel	CIS 4
AutoSampler Tower	10MSSB	CN75045773	Agilent	7863B
GC/Oven	10MSSB	CN10842006	Agilent	7890
MS Detector	10MSSB	US73317796	Agilent	5975C
AutoSampler Tray	10MSSB	CN00654640	Agilent	7683
Peltier Cooling System	10MSSB	782005793	Gersel	CIS 4
GC	10MSSD	CN10550045	Agilent	6890N
MS	10MSSD	US53931370	Agilent	5975
Autosampler	10MSSD	CN54337193	Agilent	G2614 A
Tower 7683B	10MSSD	CN54829558	Agilent	62915A
GC	10MSS6	US10245155	Agilent	6890N
Autosampler Tower	10MSS6	US10417469	Agilent/HP	7683
MS	10MSS6	US21854348	Agilent/HP	5973N
Autosampler Tray	10MSS6	US81100461	Agilent/HP	7683
GC	10MSS7	CN10319023	Agilent	6890N
Tower 7683	10MSS7	CN24728345	Agilent	62613A

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Turret 7683	10MSS7	US90403281	Hewlet Packard	62614A
Mass Spec 5973	10MSS7	US21864477	Agilent	62579A
AutoSampler Tower	10MSS8	US11818906	Agilent/HP	7683
GC/Oven	10MSS8	US10123035	Agilent	6890 N
MS Detector	10MSS8	US10440794	Agilent	5973 N
AutoSampler Tray	10MSS8	US10610754	Agilent/HP	7683
GC/Oven	10MSS9	US00033558	Agilent	6890 A
AutoSampler Tower	10MSS9	3519A42616	Agilent	18593B
MS Detector	10MSS9	US90440006	Agilent	5973 N
AutoSampler Tray	10MSS9	3518A38650	Agilent	18596C
AutoSampler Tray	10MSSE	3643A43317	Agilent	18596M
Injector Tower	10MSSE	US10512270	Agilent	G1513A
GC/Oven	10MSSE	US00006288	Agilent	G1530A
MS Detector	10MSSE	US63810194	Agilent	G1098A
Autosampler Tray	10MSSF	CN91252935	Agilent	7683B Series
Injector Tower	10MSSF	CN91756454	Agilent	7683
MS Detector	10MSSF	US91732455	Agilent	5975C
GC	10MSSF	CN10920003	Agilent	7890A
GC	10MSSG	US00025032	Agilent	G1530A
MS	10MSSG	US82311330	Agilent	G1098A
Autosampler Tray	10MSSG	3446A37132	HP	18596M
Injector Tower	10MSSG	US64500134	HP	G1513A
MS	10MSSH	US1703R003	HP	NA
GC	10MSSH	CN17013216	HP	NA
Autosampler Tray	10MSSH	CN16480039	Agilent/HP	N A
Injector Tower	10MSSH	CN16480250	Agilent/HP	NA
GC	10GCSA	CN10549055	Agilent	6890N
Autosampler Tray	10GCSA	CN54237066	Agilent	G2614A
Tower	10GCSA	CN54929639	Agilent	G2613A
ECD 1	10GCSA	U8977	Agilent	G2397A
ECD 2	10GCSA	U8978	Agilent	G2397A
GC	10GCSB	CN11201069	Agilent	7890A
Autosampler Tray	10GCSB	CN11130097	Agilent	64514A
Tower	10GCSB	CN91200383	Agilent	64513A
ECD 1	10GCSB	U19081	Agilent	G2397A
ECD 2	10GCSB	U19082	Agilent	G2397A
GC Oven	10GCS4	2750A16953	HP	5890
AutoSampler /Tower	10GCS4	2704A09552	HP	7673A
AutoSampler Tray	10GCS4	2718A06429	HP	7673A
GC	10GCS7	US10126008	Agilent	6890 N
AutoSampler Tray	10GCS7	US13612659	Agilent/HP	G2614A
Tower	10GCS7	US93809196	Agilent/HP	G2613A
ECD 1	10GCS7	U10055	Agilent	G2397A
ECD 2	10GCS7	U2932	Agilent	G2397A

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

GC	10GCS9	CN10915106	Agilent	7890A
Tower	10GCS9	CN10020012	Agilent	64513A
Autosampler Tray	10GCS9	CN91100084	Agilent	64514A
GC Oven	10GCSC	US10349021	Agilent	6890 N
AutoSampler	10GCSC	CN54237163	Agilent/HP	62614A
Tower	10GCSC	US00411307	Agilent/HP	62613A
Freezer	SV3	BA14703423	Frigidaire	FFTR1814LW7
Refrigerator	SV3	BA14703423	Frigidaire	FFTR1814LW7
10GCS7	10GCSD	NA	NA	NA
Freezer	SV4	BB01H1E0100BHD7S0358	Haier	HUM013EA
10GCSA	10GCSE	NA	NA	NA
GC	10GCSF	CN10848062	Agilent	7890A
Tower	10GCSF	CN44659505	Agilent	G2913
Tower	10GCSF	CN91756454	Agilent	G2913A
Autosampler	10GCSF	CN00654640	Agilent	G2614A
GC	10GCSG	US00035764	Agilent	6890A
Autosampler Tray	10GCSG	CN43530410	Agilent	G2614A
Tower	10GCSG	CN44659505	Agilent	G2613A
ECD 1	10GCSG	U26804	Agilent	G2397A
ECD 2	10GCSG	U26805	Agilent	G2397A
Agilent	10GCSH	US10238103	Agilent	6890A
GC	10GCSI	CN11141025	Agilent	7890A
Autosampler Tray	10GCSI	CN84951713	Agilent	G2614A
Tower	10GCSI	CN84951713	Agilent	G2613A
ECD 1	10GCSI	U128247	Agilent	G2397A
ECD 2	10GCSI	U30564	Agilent	G2397A
GC	10GCSJ	CN10906059	Agilent	7890A
Autosampler	10GCSJ	CN85252214	Agilent	G2614A
Tower	10GCSJ	CN85154864	Agilent	G2313A
ECD 1	10GCSJ	U27008	Agilent	G2397A
ECD 2	10GCSJ	U30558	Agilent	G2397A
GC	10GCSK	CN10906049	Agilent	7890A
Autosampler Tray	10GCSK	CN11080020	Agilent	G4514A
Tower	10GCSK	CN16480250	Agilent	G4513A
ECD 1	10GCSK	U27007	Agilent	G2397A
ECD 2	10GCSK	U16942	Agilent	G2397A
AutoSampler	10MSV1	13719	Environmental Sample Tech, Inc.	na
Concentrator	10MSV1	93081004	Tekmar	3000
GC	10MSV1	US00005556	HP	6890
MS	10MSV1	US63810130	HP	5973
AutoSampler	10MSV5	cents211121510	EST Analytical	Centurion
Concentrator	10MSV5	EV331120210	Encon Evolution	na
GC	10MSV5	DE00020316	HP	6890
MS	10MSV5	US81221500	HP MS	5973
Concentrator	10MSV6	173001	Tekmar	3000
AutoSampler	10MSV6/10MSV9	13352	Varian Archon	na

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

GC	10MSV6/10MSV9	US00036184	Agilent	6890A
MS	10MSV6/10MSV9	US01140180	Agilent	5973
AutoSampler	10MSV7	cents207121110	Environmental Sample Tech, Inc.	na
GC	10MSV7	CN107520005	Agilent Technologies	6850
Concentrator	10MSV7	(94251012) US02060004	Tekmar	3000
MS	10MSV7	US74818132	Agilent Technologies	5975C
GC	10MSV8	(CN10742012) US73337433	5975C	5975C
AutoSampler	10MSV8	cents205112310	EST Analytical	Centurion
Concentrator	10MSV8	EV333120210	Encon Evolution	na
MS	10MSV8	US73337433	Agilent	5975C
Concentrator	10MSV9	1064004	Tekmar	14-3100-OEL
GC	10MSVA	US10215113	Agilent	6890
MS	10MSVA	US10442746	Agilent	5973
autosampler/concentrator	10MSVA	US11203002	Tekmar	Atomx 15-0000-100
GC	10MSVE	US40620426	HP	6890
Concentrator	10MSVE	CN10427049	Teledyne Tekmar	14-9800-100
AutoSampler	10MSVE	US12058001	Teledyne Tekmar	15-0500-000
MS	10MSVE	US40620426	HP	5973
GC	10MSVF	CN16433144	Agilent	7890B
AutoSampler	10MSVF	CENTS205112310	EST Analytical	Centurion
Concentrator	10MSVF	EV332120210	EST Analytical	Encon Evolution
MS	10MSVF	US1701R009	Agilent	5977B
AutoSampler	10GCV3	cent132042304	EST Analytical	Centurion
Concentrator	10GCV3	94189002	Tekmar Dohrmann	3000
GC	10GCV3	3133A37290	HP	5890 Series II
AutoSampler	10GCV5	13713	Environmental Sample Tech, Inc.	na
Concentrator	10GCV5	99343009	Tekmar	3100
GC	10GCV5	US00020223	HP	G1530A
AutoSampler	10GCV6	13719	EST Analytical	Archon 8100
Concentrator	10GCV6	US020600004	Tekmar	14-3100-EOL
GC	10GCV6	US00042909	Agilent/HP	HP 6890
AutoSampler	10GCV9	CENT244112907	EST Analytical	Centurion
Concentrator	10GCV9	580013108P	EST Analytical	Encon
GC	10GCV9	CN12071022	Agilent Technologies	7890A
Oven	10VOA03	6520-6528	Thermo Scientific	NA
Refrigerator	C-2	NA	Walk-in	NA
Refrigerator	C-7	6331221	Beverage Air	KR74-1AS
Sonicator	10VOA04	RWA040963796A	Fisher Scientific	FS220
Freezer	V5	WB94954367	Frigidaire	LFFH21F7HWG
Refrigerator	V6	96020404	Norlake Scientific	NSLF482WAW/1
Oven	10WT56	U19R-507936-UR	Lindberg/Blue M	MO1450PSA-1
Refrigerator/Freezer	V8	NA	Frigidaire	NA
Refrigerator	V7	K13809596	Amana	ABB2221WEB1

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Incubator	10WET16	115770704-57744	Fisher Scientific	Isotemp Incubator
Incubator	10WET22	30100031/WB24501232	Fisher Scientific	307
Incubator	10WET35	2018090423462	Fisher Scientific	307C
Incubator	10WET60	300789-1711	Thermo Forma	3940
Autotitrator	10WET6	1888001004148	Metrohm	888 Titrando Titrator
Autosampler	10WET6	1778001003123	Metrohm	778 Sample Processor
probe	10WET6	263664	Metrohm	778 Sample Processor
Diss. Oxy Meter	10WET51	00K0500	YSI	5000
Oven	10WET17	9410-305	Precision Scientific	130 DM
AutoClave	10WET29	12770804/02244	Harvey	na
pH Meter	10WET7	001577	Orion	na
pH Meter	10WET31	10044	IQ Scientific Instruments	na
Thermoreactor	10WET26	89543	Neutec Group Inc.	ECO 25
COD Reactor	10WET11	COD-B0140	Bioscience, Inc.	na
KoneLab Discrete Analyzer	10WET3	P0419693	Thermo Fisher Scientific	Konelab 20
Conductivity meter	10WET9	206454	Oaktom	Con 110 Series
Conductivity meter - probe	10WET9	204/02	Oaktom	Con 110 Series
Colony Counter	10WET30	na	Gallenkamp	Colony Counter
Colony Counter	10WET38	na	Darkfield Quebec	Colony Counter
Water Bath	10WET27	1605680347017	Fisher Scientific	Isotemp 210
Distillation Block	10WET12	na	Environmental Express	na
Distillation Block	10WET13	na	MIDI-STIL	na
Refrigerator	C-11	6584	Walkin	na
Refrigerator	WC3	10200716	Sanyo	na
Spectrometer	10WETA	1284818	Hach	DR 2700
Hot Plate	10WET34	2608US	Presto	Tilt'n Drain Big Griddle
Smart Chem Discrete Analyzer	10WT36	W0902154	West Co Scientific Instruments	Smart Chem 200
Hot Plate	10WET40	440895	Corning	na
Stir Plate	10WET41	1889080719259	Fisher Scientific	na
Stir Plate	10WET42	776940355770	Barnstead/Thermolyne	S46725/Cimarec 2
Vortex Mixer	10WET44	27302	American Scientific Prod.	S8223-1
Extractor	10WET45	0205PUB370	Horizon Technology	Spe-dex 4790
Extractor	10WET46	0205FUB369	Horizon Technology	Spe-dex 4791
Extractor	10WET47	0205FUB368	Horizon Technology	Spe-dex 4792
Extractor	10WET48	0205FUB367	Horizon Technology	Spe-dex 4793
Closed Cup - Penske	10WT49	10AZ-10	Precision Scientific	na
Refrigerator	WC2	A091200156	Summit Commercial	SCR485L
pH/BOD meter	10WT54	110813032026	Hach	LBOD10101
pH probe	10WT54	122223032017	HACH	na
pH/BOD meter/Fluoride	10WT53	110300052350	Hach	HQ40d
pH/BOD meter/Fluoride - probe	10WT53	152392938004	Hach	HQ40d
Hot Block	10WET55	na	Environmental Express	na
Oven	10WET65	611729-434	Fisher Scientific	13-247-650G(6905)

ATTACHMENT IVA - MINNEAPOLIS EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

pH Probe	11662571034	11662571034	Hach	PHC301
pH Probe	121952571033	121952571033	Hach	PHC301
pH Probe	122143032067	122143032067	Hach	LBOD101
pH Probe	712202002	712202002	Switchcraft	PHW77-SS
Turbidity Meter	10WT59	11050C0092997	Hach	2100Q
Hand Held Brix Refractometer	10WT60	Fisher catalog # 13-946-21	Fisher	na
Oven	10WET19	na	VWR Scientific	1370F
Quanti-Tray Sealer Model 2x	10WET56	4836	Quanti-Tray	89-10894-02
IC	10WT61	1881000121132	Metrohm	881 Compact IC
Lachat	10WT62	120400001409	Quick Chem	8500
Autotitrator	10WT63	1814001009181	Metromn	905 USB Sample Processor
Probe	10WT63	1281705	Metromn	905 USB Sample Processor
JT Backer Speedisk Expanded Extration Station	10WET66	L02N23	J.T. Baker	Speedisk Expanded Extraction Station
Desiccator	10WET68	na	Sanplatec Corp	DryKeeper
Desiccator	10WET69	na	Boekel	na
Desiccator	10WET70	na	Boekel	na
Desiccator	10WET71	na	Boekel	na
Desiccator	10WET72	na	Boekel	na
Desiccator	10WET73	na	Boekel	na
Desiccator	10WET74	na	Boekel	na
Desiccator	10WET75	na	Boekel	na
Meter	10WETE	120400069964	Hach	HQ440d
Meter - probe	10WETE	172612618021	Hach	PHC20101
Oven	10WT77	614389-852	Fisher Isotemp Oven	6905
Oven	10WET78	614389-853	Fisher Isotemp Oven	6905
Smart Chem Discrete Analyzer	10WT79	W0407060	West Co Scientific Instruments	Smart Chem 200 (P/N 399-W001-01)
Hot Plate	10WT81	21-697	Presto	Tilt'n Drain Big Griddle
Lachat	10WT82	100700001229	Hach Quick Chem QC 8500 Series 2	8500
COD Reactor	10WT83	900402106	HACH	45600
COD Reactor	10WT84	870509093	HACH	16500-10
Distillation Block	10WT85	2106 - no visible SN, 2106 was the only identifiable ID	Environmental Express	na
Water Bath	10WT86	601061689	Precision Scientific Water Bath	Coliform Incubator Bath
Oven	10WT88	41762572	Fisher Scientific	151030521
Fridge	WC4	4315123638037	Danby Designer	DBC120BLS
COD Reactor block	10WET57	160200C0071	HACH	DRB 200
Hot Block	10MET03	4952CEC2361	Environmental Express	na
Distillation Block	10WT89	4071305	NA	Midi-Vap 4000
Desiccator	10WT89	NA	PLAS Labs	NA
Desiccator	10WT90	NA	PLAS Labs	NA

ATTACHMENT IVB - MONTANA EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

INSTRUMENT	PACE ID	SERIAL NUMBER	MANUFACTURER	MODEL
NIST Thermometer	160283107	160283107	Fisher Scientific	15-077-55-11729765160283107
NIST Thermometer	111855001	111855001	Fisher Scientific	15-077-55; 255NK; FB50262
IR Gun	160285052	160285052	Fisher Scientific	06-664-38 11729785
Balance	11MT09 (40020019)	40020019	Sartorius	LC620S
Balance	11MT07 (B027060)	B027060	Fisher	A200DS
Balance	11BAL2 (G3251202300491)	G3251202300491	Ohaus	ARC120
Balance	11BAL4	B504529759	Ohaus	SP202
Balance	11BAL5	B508634908	Mettler	ML3002E
Balance	11BAL6	B525074608	Mettler	X5105DU
Autosampler	11MT04	3225A31213	Hewlett-Packard	7673
Autosampler	11MT04	3120A28856	Hewlett-Packard	7673
SVOA GC	11MT04	275A16778	Hewlett-Packard	5890
IC Autosampler	11MT05	7101378	Dionex	AS40-1
Ion Chromatograph	11MT05	05120175	Dionex	ICS1000
Autoanalyzer Autosampler	11MT06	311162	Astoria Pacific	311
Autoanalyzer Detector	11MT06	305352	Astoria Pacific	305A
Autoanalyzer Heater Unit	11MT06	303437	Astoria Pacific	303A
Autoanalyzer Photometer	11MT06	350376	Astoria Pacific	350
Autoanalyzer Power Supply	11MT06	304224	Astoria Pacific	304A
Autosampler power supply	11MT06	5766	Perstorp	509
Autosampler pump	11MT06	NA	Perstorp	502
Spectrophotometer	11MT08	104218	Spectronic	Aquamate
Oven	11MT10	1451	Fisher	Isotemp 255D
Oven	11MT11	20900168	Fisher	Isotemp 630F
Muffle Furnace	11MT12	32400731	Sybron	Thermolyne
Concentrator	11MT13	TB9814N8062	Zymark	TurboVap II
Concentrator	11MT14	4082	Zymark	TurboVap II
Furnace	11MT15	0479 16654	Sybron Thermolyne	1300
N-Evap	11MT16	11771	Organomation	112
Waterbath	11MT17	698100224	Precision Scientific	
Sonicator	11MT19	RUA080390744	Fisher	FS60
Furnace	11MT22	3167	Leco	S-144DR
Turbidimeter	11MT23	610064	HF Scientific	Micro 1000
Sonicator	11MT24	NA	Heat Systems	Sonicator XL
Sonicator	11MT25	B1090019	Branson	Sonfier 450
Concentrator	11MT33	96312005	Tekmar/Dohrmann	Tekmar 3000 Purge and Trap concentrator
VOA GC	11MT33	US00009537	Aglient	6890
Autosampler	11MT33	CENT-W-417042312	EST	Centurion
Block Digestor	11MT34	1800-733	Lachat	BD-46
AutoSampler	11MT38	CENTW417042312	O-I-Analytical	4552
Concentrator	11MT38	99274012	Tekmar Dohrmann	3100
GC System	11MT38	US00032765	Agilent	6890
MS Detector	11MT38	US94240027	Agilent	5973
pH meter	11MT40	81207936	Accumet	AR50

ATTACHMENT IVB - MONTANA EQUIPMENT LIST CONTINUED (CURRENT AS OF ISSUE DATE)

Oven	11MT41	20600109	Fisher	Isotemp 630F
Oven	11MT42	9212-016	Precision Scientific	Thelco 130 DM
GC System	11MT43	US00021845	Agilent	6890
Concentrator	11MT43	EV431073112	EST	Evolution
AutoSampler	11MT43	CENT-W-416041012	EST	Centurion
Flow Analyzer	11MT44	120400001407	Lachat	8500
For Calculation acodes	11MT45	NA	NA	NA
listed as generic instrument in Epic	11MT46	NA	NA	NA
Sieve Shaker	11MT47	6290	Gilson	SS-15
Sieve Shaker	11MT48	10-2394	W.S. Tyler	RX_29
Concentrator	11MT51	4254	Zymark	Turbo Vap II
Custom Shaker	11MT55	NA	Custom	NA
Oven	11MT56	801N0068	Fisher516G	
Autoclave	11MT57	1277081210300	ThermoFisher	ST75925
Metals Block Digester	11MT58	S388CEC2479	Environmental Express	Hot Block
ICP	11MT60	20071505	ThermoFisher	ICAP6500 Duo
Autosampler	11MT60	71011378	CETAC	ASX-520
Chiller	11MT60	51520175	ThermoFisher	ThermoFlex900
Centrifuge	11MT61	34721368	Damon	IEC HN-S
Block Digestor	11MT62	1800-296	Lachat	BD-46
unused	11MT63	NA	NA	NA
Handheld pH	11MT64	H00013	Thermo Scientific	Star A121
Spectrophotometer	11MT65	5A4S008017	Thermo Scientific	Evolution 201
ASTM Hydrometer	NA	NA	NA	152 H
Hood	11MT66	NA	NA	NA
Hood	11MT67	NA	NA	NA
Hood	11MT68	NA	NA	NA
Hood	11MT69	NA	NA	NA
Hood	11MT70	NA	NA	NA
Hood	11MT71	NA	NA	NA
Hood	11MT72	NA	NA	NA
Hood	11MT73	NA	NA	NA
Concentrator	11MT74	04316	Caliper Life Science	Turbo Vap II
pH meter	11MT75	XL94104028	Fisher Scientific	accumet XL200
Oven	11MT76	972	Fisher Scientific	116G
GC System	11MT78	US10238089	Agilent	G1540N
Autosampler	11MT78	CN81047578	Agilent	G2913A
Autosampler	11MT78	CN82750941	Agilent	G2913A
Autosampler Tray	11MT78	CN21720602	Agilent	G2614A
TCLP Rotator A	11MT79	NA	NA	NA
TCLP Rotator B	11MT79	NA	NA	NA
TCLP Rotator C	11MT79	NA	NA	NA
Filter Pump 1.5	11MT80	996305884	Edwards	904160
Filter Pump 2	11MT81	42396	Edwards	5KC37NN470GX
pH meter	11MT82	X27760	Thermo Scientific	OrionSTARA215
TKN Digestor	11MT83	HTLC1015510459	Hatch, Lachat BD40HT	TSLA1015411724
Filter Pump 2	11MT84	25963	Edwards	5KC37NN470GX

ATTACHMENT IVB - MONTANA EQUIPMENT LIST CONTINUED (CURRENT AS OF ISSUE DATE)

Sieve Shaker	11MT85	1216080311F	Endecotts	NA
Oven	11MT86	42022678	ThermoFisher Scientific	51028872
Drying Cabinet	11MT87	NA	NA	NA
Sieve Shaker	11MT88	1217120535J	Endecotts	NA
Oven	11MT89	42087930	Fisher Sci 180L	Cat# 151030521
Muffle Furnaceb Kiln	11MT90	SN 035988	Delphi	NA
Mercury analyzer	11MT91	050702ASX, 090701QTS	CETAC	ASX-400, QuickTrace
IC Autosampler	11MT92	09090574, 09080900, 09100402, 09090060, 09090425	Dionex	AS,ICS Series, ICS-2100,ICS-3000 DC, ICS-3000 SP
IC Autosampler	11MT93	9090060, 9090425	Dionex	ICS-3000 DC, ICS 3000 SP
Metals Block Digester	11MT94	NA	Smartblock	NA
Autopipette	FSA4	87271	Hamilton	999 uL Adj. Vol.
Autopipette	FSA5	87794	Hamilton	999 uL Adj. Vol.
Autopipette	FSA6	078528	Hamilton	300 uL Adj. Vol.
Autopipette	FSA7	KJ06363	Thermo Scientific	2-5mL
Autopipette	FSA8	OU17387	Thermo Scientific	Cat #4641110N
Autopipette	FSA9	K39243G	Eppendorf	Batch# G422723K
Autopipette	IN-1103 - WC4	E02008781	Oxford	1000-5000uL
Autopipette	WC2	4035876	Eppendorf	0.1-1mL
Autopipette	WC5	033269	Hamilton	.25-1mL
Autopipette	WC7	GJ45632	Finnpipette	1-10mL
Autopipette	WC8	KH45108	Thermo Scientific	2-5mL
Autopipette	WC9	82121	Hamilton	0.1-1mL
Autopipette	WC10	I42616F	Eppendorf	20-200uL
Autopipette	MET1	080395	Hamilton	.05-0.3mL
Autopipette	MET2	80953	Hamilton	0.1-1mL
Autopipette	MET3	L32587E	Eppendorf	.02mL-.2mL
Autopipette	MET4	MU29157	Thermo Scientific	.5-5mL
Autopipette	IC1	39492	Hamilton	0.1-1mL
Autopipette	IC3	o81405	Hamilton	0.1-1mL
Autopipette	IC5	KH11148	Finnpipette	0.5-5mL
Bottletop Dispenser	BT13 HNO3	8655	OPTIFIX	EMD
Bottletop Dispenser	BT1 MeCl2	24915	Brinkmann	NA
Bottletop Dispenser	BT2 MeOH	24992	Fisher	NA
Bottletop Dispenser	BT3 MeCl2	12M10591	Eppendorf	NA
Bottletop Dispenser	BT4 Hexane	07Z7769	Dispensette	NA
Bottletop Dispenser	BT5 Ammonium Acetate	AF 2153	Fisher	NA
Bottletop Dispenser	BT6 Sodium Acetate	AF6770	Fisher	NA
Bottletop Dispenser	BT7 Ethanol	AF6862	Fisher	NA
Bottletop Dispenser	BT8 Digest Solution	AF9468	Fisher	NA
Bottletop Dispenser	BT9 Potassium Dichromate	AG4962	Fisher	NA
Bottletop Dispenser	BT10 2M KCl	14024979	Fisher	NA
Bottletop Dispenser	BT11 NaHCl3	14024938	Fisher	NA
Bottletop Dispenser	BT 12 CaCO3	AK6234	Satorius	NA

ATTACHMENT IVB - MONTANA EQUIPMENT LIST CONTINUED (CURRENT AS OF ISSUE DATE)

Bottletop Dispenser	BT 14 ICP/Metals	14200358	Fisher	NA
Bottletop Dispenser	BT 15 Metals/HMP	75123	Brinkmann	NA
Bottletop Dispenser	BT 16	NA	Dispensette	NA
Bottletop Dispenser	BT 17/Inorganics	NA	Dispensette	NA
Bottletop Dispenser	BT 16 IC water	17309419	Fisher	NA
Refrigerator	MTC-1	NA	Sanyo	NA
Refrigerator	MTC-11	WA93300079	Frigidaire	FRU17B2JW18
Freezer	MTC-13	A100600186	SPT	UF-160S
Refrigerator	MTC-14	A94B200112T	Saturn	S494
Refrigerator	MTC-16	1201CENH00159	Centaur Plus	CSD-2DR-BAL
Freezer	MTC-17	NB37116248F40631	SPT	UF-150W
Refrigerator	MTC-18	04EL9046H	Imperial	F3AD13201TFC015
Refrigerator	MTC-4	920940742	Kenmore	546.9901741
Refrigerator	MTC-9	T33587106	Traulsen	620010
Refrigerator	MTC-19	NA	Gourmia	GMF-600
Freezer	MTC-20	WB22221798	Frigidaire	FFC1SC3AWO
Freezer	MTC-21	WB64429495	Kenmore	253.165421
Refrigerator	MTC-22	25033501	Arctic King	AFRM016
Refrigerator	MTC-23	D80-24154501	Arctic King	AFRM016AEB
Pulverizer	N/A	N/A	Retsch	RS100
Vacuum Pump	N/A	41032	Edwards	E2M2
Vacuum Pump	N/A	E22922	Dayton	SA55NXGTB-4142
Automated Temperature Monitoring System	DocuTemp		DocuTemp	
Thermometer	5040005112	NA	LogTag	UTRIX-16
Thermometer	5040005144	NA	LogTag	UTRIX-16
Thermometer	5040005107	NA	LogTag	UTRIX-16
Thermometer	5040005110	NA	LogTag	UTRIX-16
Thermometer	5040005109	NA	LogTag	UTRIX-16
Thermometer	5040005145	NA	LogTag	UTRIX-16
Thermometer	5040005113	NA	LogTag	UTRIX-16
Thermometer	5040005106	NA	LogTag	UTRIX-16
Thermometer	5040005111	NA	LogTag	UTRIX-16
Thermometer	5040005108	NA	LogTag	UTRIX-16
Thermometer	5040005114	NA	LogTag	UTRIX-16
Thermometer	5040005137	NA	LogTag	UTRIX-16
Thermometer	5040005141	NA	LogTag	UTRIX-16
Thermometer	5040005142	NA	LogTag	UTRIX-16
Thermometer	5040005143	NA	LogTag	UTRIX-16
Fire Extinguisher	FE-1	V-983066	Halon	A335
Fire Extinguisher	FE-2	ZT-849854	Ansul Sentry	A10H
Courier Van	FE-3	ZU-092145	Ansul Sentry	A02VB
Fire Extinguisher	FE-4	CF-322188	Fire Master	AA05-1
Fire Extinguisher	FE-5	V-185947	Fire Master	AA0S
Fire Extinguisher	FE-6	BZ-614843	Fire Master	AA10S
Fire Extinguisher	FE-7	BZ-614849	Fire master	AA10S
Fire Extinguisher	FE-8	CF-322139	Fire Master	AA05-1

ATTACHMENT IVB - MONTANA EQUIPMENT LIST CONTINUED (CURRENT AS OF ISSUE DATE)

Fire Extinguisher	FE-9	ZD-589859	Ansul Sentry	A10H
Fire Extinguisher	FE-10	AY-438621	Fire master	AA05-1
Fire Extinguisher	FE-11	AB-619738	Ansul Sentry	AA05-1
Fire Extinguisher	FE-12	ZD-589837	Ansul Sentry	A10H
Eye Wash Station	SE-1	NA	Guardian	NA
Shower station	SS-1	NA	Guardian	NA
First Aid Kit	FA-1	NA	ALSCO	NA

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

VIRGINIA EQUIPMENT			
INSTRUMENT	**PACE ID**	**MANUFACTURER**	**MODEL #/SN#**
CVAA Mercury Analyzer	12HG1	Cetac	M-6100/060402 QT6
Autosampler		Cetac	ASX-400/070401 ASX-4
Hardware		Venture Systemax	SYX PHM800PRO/106381144
Software		Cetac	Quicktrace Hg Analyzer System Version 1.2.1
ICP Atomic Emission Spectrometer	12ICP1	Perkin Elmer	Optima 3000XL/069N4081202
Autosampler		Cetac	ASX-520/090511A520-new in 2006
Hardware		Compudyne	X86 Model 7/4747
Software		Perkin Elmer	Winlab 32 ICP Optical Emission Software Ver2.2
ICPMS Atomic Emission Spectrometer	12ICM1	Perkin Elmer	ELAN 9000 / AJ11920712
Autosampler/Pump		ESI Fast System	SC 4DX / X4DX-HS-TSP-16-101109
Recirculator		Polyscience	NA
Software		Perkin Elmer	Version 3.4
Hardware		Dell XP	X12-51522
ICPMS Atomic Emission Spectrometer	12ICM3	Perkin Elmer	ELAN 9000 / AJ3050909
Autosampler/Pump		ESI Fast System	
Recirculator		Polyscience	
Software		Lenovo	
Hardware		Perkin Elmer	Version 3.4
Lachat	12WTA4	Zellweger Analytics	Lachat Quikchem FIA+ 8000 Series/A83000-1480
Lachat Reagent Pump		Zellweger Analytics	RP-150 Series/A82000-1527 replacement 2005
Autosampler		Cetac	ASX-500 Model No 510/109932ASX
Autodilutor		Zellweger Analytics	8000 Series/A81010-277 Out of service ~2002
Micro Distillation Equipment (Ammonia)		Lachat MicroDist 5/09	081200001033
Hardware		Midwest Comp Depot	3035
Software		Omnion	FIA Data System
Lachat	12WTAB	Lachat	Lachat QuickChem 8500 Series 2 Serial Number 10070000129
Lachat Reagent Pump		Lachat	RP 150 Series Serial number A82000-1961
Autosampler		Cetac	ASX500 Model 510 Serial number 010025ASX
Hardware		Hewlett Packard	Hp compaq
Software		Omnion	FIA Data System
Ion Chromatograph	12WTAC	Metrohm	930 Flex IC
Regenerant Dispenser		Metrohm	IC-05
Autosampler		Metrohm	Model 850 Sample Processor
Hardware		Dell	SN#CBDUC284-70821-553-OGIP
Software		Metrohm	IC Net 2.3
Ion Chromatograph	12WTA7	Metrohm	Model 881 Advanced Compact IC 1881000122119
Regenerant Dispenser		Metrohm	800 Dosino
Autosampler		Metrohm	Model 858 Advanced Sample Processor
Hardware		Dell	Optiplex 790
Software		Metrohm	IC Net 2.3

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

TKN Block Digester	12TKN2	Lachat	Model BD-40/TSLA1013511403
Autotitrator, Alkalinity	12WETD	ManTech	TitraSip/MT-1B5-957
Autosampler		ManTech	AutoMax 73 Sampler
Hardware		Hewlett Packard	Prodesk
Software			PC Titrate for Windows v.3
BOD Warmer #1		Thermo Precision	60541072
BOD Incubator #4	12BOD4	Fisher	Model 3720/300007704
BOD Incubator #5	12BOD5	Fisher	Model 3720A/300064399
BOD Incubator #6	12BOD6	Fisher	Model 3720A/300088990
BOD Reader	12WET2	Thermo Electron Corp	BOD Auto EZ BOD Reader 10060020/A0074
BOD Hardware		Hewlett Packard/Compaq	24A41601N8
BOD Software		Thermosystems	BOD Auto EZ 2001
TOC	12WTA3	OI Analyzer	SN H129732449E
TOC		OI Autosampler	SN E129788451
TOC	12WTA9	OI Solids Analyzer	SN A129733824
Autosampling Module		OI Corporation	No Model/621290637-92120
IR Detector		OI Corporation	No Model/2A0002T
Hardware		HP	Compaq
Software		OI Corporation	V1.4.2
TOC	12WTA8	OI Analyzer	SN P407730312P
Autosampling Module		OI Corporation	Model 1088 AS
IR Detector		OI Corporation	Model 1030 / B622737366
Hardware		Lenovo	Thinkcentre
Software		OI Corporation	1.4.2
Hardware 2004		ABS	52X MTRP/10085322
Software 2004		EZ Solids	EZ_Solids Program June 23, 2004
Autosampler		Orion	AS 3000/B0019
Bacteria Incubator	12INC1	Shel Lab	1545/11052906
Coliform Incubator Bath	12INC2	ThermoFisher	253/SN202682-185
Microscope 10X/30X		National Optical	446TBL-10
Bacteria Incubator	12INC3	Shel Lab 1996 (Sterility chk)	1520
Quanti Tray Sealer		IDEXX	89-10894-02 4788
Oven	12O010	VWR	1330GM/05039804
Oven	12OV3	Fisher	6926/614203-180
Oven	12OV4	Shel Lab	SM05/0405Z114
Oven	12OV6	Fisher Scientific	100L/42130594
Muffle Furnace	12MFL5	Fisher Scientific	Isotemp/70100004
Water Bath		Fisher Scientific	FS140/FS010507
Metals Digestion Blocks	HB1, HB2	CPI	05-C0530/000424 1005-CPI ModBlock Inst
Metals Digestion Block	HB3	CAi	SmartBlock 125i
Metals Digestion Block	HB4	AGS Scientific	Durablock QB17064
Balance (Metals)	12BAL3	AND	GF 1200 / 10318953
Balance	12BAL4	Sartorius	LA 3200D / 13407528
Balance	12BAL1	Sartorius	Genius / 13003773
Balance	12BALA	Acculab	027UC1079
Balance	12BALB	Sartorius	BP1105 / 50206779
Balance	12BALC	Denver Instruments	040DCD057
Balance	12BALD	Mettler	B549797353
Stir Plate		Thermoline Type 7200	903971255007

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Refrigerator 2R (Metals)		Sanyo	SR-362OK/051105496
Refrigerator #3		True Mfg Co.	T-49/1-2953805
Refrigerator #5		True Mfg Co.	T-49/1-3060851
Refrigerator #8		True Mfg Co	T-35/I-3016399
Refrigerator #10		Gibson	
Refrigerator #12		Beverage-Air	9029136/KR481AS
Refrigerator #13		US Cooler Walk-in	29716
Refrigerator #14		SubZero	
Mixer		Thermolyne	M37615/376950140798
Stir/Hotplate		VWR	12365-392 / 050914023
COD Reactor-Hot block	CODR1	HACH	45600-00/920600007477
COD Reactor- Hot block	CODR2	HACH	16500-10/5944
Dessicator	12DES1	Labconco	55300/171400
Dessicator	12DES2	Labconco	55300/232878
Dessicator	12DES3	Glass	
Dessicator	12DES4	Fisher	
Dessicator	12DES5	Boekel	
Dessicator	12DES6	Plas Labs	
Dessicator	12DES7	Plas Labs	
Dessicator	12DES8	Plas Labs	
Dessicator	12DES9	SanPlatec	
Mixer		Fisher Scientific	Model 15/103
Rotator		Lab-Line	Model 1345/1002-1791
Autoclave	12CLV2	Tuttnauer/Brinkmann	3545EP
pH Meter	12WETG	OrionStar	A215/X27234
Turbidimeter	12WETF	Orion	AQ3010/3494427
Dissolved Oxygen Meter		YSI	5100
BOD Software		YSI	5120 BODANALYST
Spectrophotometer	12WTA1	HACH	DR 5000
Closed Cup Flashpoint Tester	12FP1	Koehler	K16200
Water Purification System, DI		Barnstead	E-Pure
Water Purification System, RO pure LP Low pressure RO System		Barnstead Thermolyne	Model D2622 SN 496000209600 Cartridge Changes noted in log book
Resistivity Meter for RO system		Sybron Barnstead	Model 02770 Resistivity Log Sheet is posted by system
Autotitrator		ManTech	Titrasip / MT 1B5-957
Autosampler		ManTech	AutoMax 73 / 19105065
Hardware		Hewlitt Packard	Prodesk
Software		Windows	PC Titrate for Windows v.3
NIST Thermometer		Fisher	170324450
NIST Thermometer		Fisher	170610086
IR Temperature Gun		Fisher	140792808
IR Temperature Gun		Fisher	1506677071
Bottle Top Dispenser	BT-1		01L84732
Bottle Top Dispenser	BT-2		01L09379
Bottle Top Dispenser	BT-3		12L16083
Bottle Top Dispenser	BT-4		Metals H2SO4
Bottle Top Dispenser	BT-5		02M57520
Bottle Top Dispenser	BT-6		Wet Chem Trap Soln
Bottle Top Dispenser	BT-7		Wet Chem H2O
Bottle Top Dispenser	BT-8		Wet Chem Digest reagent

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Bottle Top Dispenser	BT-9		H2SO4
Bottle Top Dispenser	BT-10		11L09388

Duluth Equipment				
INSTRUMENT	**Pace ID**	**SERIAL #**	**MANUFACTURER**	**MODEL**
Balance	12BAL5	13003775	Sartorius	ME4145
Balance	13BAL1	304562	Mettler	P1200
Balance	13BAL2	12200149	AND	HR-120
Balance	13BAL4	N0088210	Denver Instruments	XL-1810
Balance	13BAL5	B551880610	Mettler	XSE 204
Balance	13BAL7	B549797355	Mettler	XSE 104
Balance	13BAL8	M89136	Mettler	AT261
COD Reactor	13COD1	950900013204	HACH	45600-00
Incubator	13INC7	9209-113	Precision Scientific	66551
BOD Incubator	13INC6			
BOD Incubator	13INC5	300168083	Thermo	Isotemp
Incubator 35°C	13INC4	12	LabLine CO2	3010
Incubator (Water Bath)	13INC3		LabLine	460NS
Muffle Furnace	13MFL1		Lindberg	51442
Oven	13OVN1	1200600	VWR (Shel Lab)	1370G
Oven	13OVN2		Thelco	28
Oven	13OVN3	8A-365	Blue M	OV-8A
Oven	13OVN4	S175-517150-SS	Lindberg/Blue M	
Oven	13OVN5	42094122	ThermoFisher	
Spectrophotometer UV VIS	13WET1	HEDN238001	Thermo	9423AQ2100E
Turbidimeter	13WET2	100002146	HACH	2100AN
Lachat	13WET3	50100000097	Zellweger Analytics	8500
Ion Chromatograph	13WET4	091708136940	Zellweger Analytics	8500
Lachat Autosampler	-	010591A520	Zellweger Analytics	ASX 520
Lachat	13WET5	40900000051	Zellweger Analytics	8500
Lachat Autosampler	-	A81010-007	Zellweger Analytics	ASX 600
pH Meter	13WET6	B07284	Thermo Orion	Star Series
LDO Meter/Probe	13WET7	(wet chem)	HACH	HQ30d flexi
pH Meter	13WET8	13043	Orion	720A
pH Meter	13WET9	43996	Orion	301
pH Meter	13WET10	143491	SPER Scientific	Large Display pH Pen 580051
pH Meter	13WETA	069292	Thermo Orion	420
Conductivity Meter	13WETB		HACH	Sension5
D.O. Meter/Probe	13WETC	(ATL)	HACH	HQ30d flexi
Titrator, Amperometric	13WETD	96090001089	HACH	19299-00
Distillation Unit Microblock	13WETE		Environmental Express	
Micro Distillation Unit	13WETF		Lachat	21 place
Color Test Kit	13WETG		HACH	CO-1
Residual Chlorine Meter	13WETH	HI 701	Hanna	
Spectrophotometer UV VIS	13WETJ	1648363	HACH	DR 3900
pH/Conductivity Meter	13WETK	x37428	Orion	
Turbidimeter	13WETL	2668277	Orion	Aquafast
COD Reactor	13COD2	910404562	HACH	45600-00

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

COD Reactor	13COD2	910404562	HACH	45600-00
Autoclave	13CLV1	37827	Market Forge	Sterilmatic STM-E
Autoclave temp gauge	13CLV1T			
Autoclave Pressure gauge	13CLV1P			
Buret, Class A	13BUR1	0230		
Buret, Class A	13BUR2	2103		Class A
Buret, Class A	13BUR3	7249		Class A
Autodispenser	13DSP1		North Central Labs	
Autodispenser	13DSP2	JY16291	SCILOGEX	
Digester (Phosphorus)	13DIG1		CA1	Smartblock 226
Digester, Block	13DIG2		SPC Science	DigiPrep
Hotblock (TKN)	13TKN1		Technicon	BD 40
Hotblock (TKN)	13TKN2	STU6U00860	Seal Analytical	BD 50 Block
Microscope		814602	American Optical Corp	Forty
BOD Incubator	13INC1		Room	
Stir Plate		757960584897	Thermolyne	SP18425
Stir Plate			Corning	PC 520
Hotplate	13HPT1	61920359996	Thermolyne	Ciramec 3 HP 47135
Hotplate	13HPT2	1.07303E+12	Thermolyne	Ciramec 3 HP 47135-60
Hotplate	10WET43	1000191	Fischer	
Sonicator	13SON1		VWR	Aquasonic 50-T
Sealer, QuantiTray		01174		2X/89-10894-00
Sterilizer	13STL1		E2E	
Light Box (ATL)			Hall Productions	1218
UV Lamp	13UVL1	691	UVP, Inc	UVGL-25
SPE StepSaver 7-station Funnel	13SPE1		Environmental Express	Cat No. G1106
SPE StepSaver 7-station Funnel	13SPE2		Environmental Express	Cat No. G1107
Mercury Analyzer Model III CVAFS	12Hg2	1103401	Brooks Rand	
Autosampler		4936A14632	Brooks Rand	
Total Hg Purge and Trap		11078001	Brooks Rand	
Hg Speciation Purge and Trap		41107301	Brooks Rand	
Mercury Guru Software				4.1
Hood	13HOOD1			
Hood	13HOOD2			
Hood	13HOOD3			
Hood	13HOOD4			
Hood	13HOOD5			
Hood	13HOOD6			
Hood	DB-1		ESCO	
Hood	DB-2		ESCO	
Distillation Block	MDS-A	1021401	Brooks Rand	
Distillation Block	MDS-C	1034401	Brooks Rand	
Distillation Block	13MDS1	2077	Environmental Express	
Evaporator for SPE System	13VAP01	08-0701	Horizon Technology	SPEED VAP III
Evaporator for SPE System	13VAP02	NA	Toastmaster	TM-201GR
QuantiTray Sealer	13QT1			
Light Meter	13LM1	160519482	Traceable	Model: 06-662-63, 11774266

ATTACHMENT IVC - VIRGINIA & DULUTH EQUIPMENT LIST CONT. (CURRENT AS OF ISSUE DATE)

Light Timer	13TIMER1			
Light Timer	13TIMER2			
Light Timer	13TIMER3			
BOD	13BOD1	17292	Skalar Analytical	99314818
Water Purification System (main)	13DI1		Culligan	
Water Purification System (subsequent)	13DI2		Barnstead	
Water Purification System	13DI1-A	1090090938202	Barnstead	D4641
Water Filter/DIW System	13DI1-B			

ATTACHMENT VA - MINNEAPOLIS LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

DOCUMENT NAME	NUMBER
Analysis of Air Samples for Volatile Organic Compounds by Gas Chromatography/PID-FID method TO-3	S-MN-A-003
Cleaning, Certification, Leak Checking and Preparation for Shipment of SUMMA Passivated Canisters	S-MN-A-004
Determination of Fixed Gases in Air by Modified 3C	S-MN-A-005
Methane, Ethane, Ethene, and Propane in Water by GCFID mod. 3810 and RSK 175	S-MN-A-007
Analysis of Whole Air Sample for Volatile Organic Compound by GC/MS EPA TO15/TO14	S-MN-A-013
Analysis of TO17 Active Air Samples	S-MN-A-018
Analysis of Benzene for Fenceline Monitoring	S-MN-A-021
Sample Management	S-MN-C-001
Bottle Preparation	S-MN-C-003
Subcontracting Samples	S-MN-C-004
Internal Chain of Custody	S-MN-C-005
Final Report and Deliverable Contents	S-MN-C-007
Processing Tentatively Identified Compounds (TICs) For GC/MS	S-ALL-O-038
Document Numbering	S-ALL-Q-003
Laboratory Documentation	S-ALL-Q-009
Quality Report to Corporate	S-ALL-Q-014
Review of Laboratory Management System	S-ALL-Q-015
Manual Integration	S-ALL-Q-016
3P Program	S-ALL-Q-022
Use and Operation of LabTrack	S-ALL-Q-028
Mintminer Data File Review For Data Integrity Monitoring	S-ALL-Q-029
Operation of Data Checker	S-ALL-Q-030
Data Recall	S-ALL-Q-035
Method Validation and Instrument Verification	S-ALL-Q-047
Hazard Assessments	S-ALL-S-001
LMS Sub-Learn Center System and Training Administrator Responsibilities	S-ALL-T-002
Analysis of Polychlorinated Biphenyls in Oil, Soil, Water, Wipe and Air Matrixes	S-MN-O-432
Determination of Diesel Range Organics in Water and Soil (Wisconsin modified DRO)	S-MN-O-466
Determination of Pesticides in Water and Soil	S-MN-O-574
Determination of EDB and DBCP in Aqueous Samples	S-MN-O-576
The Determination of Diesel Range Organics, Residual Range Organics and Total Extractable Hydrocarbons	S-MN-O-578
Determination of C10-C32 Hydrocarbons in Soil	S-MN-O-587
Sample Preparation and Analysis of Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam	S-MN-O-588
Analysis of PCBs in Soil and Sediment Matrices by Modified 8082A GEHR	S-MN-O-590
The Determination of Specific Aromatic Compounds and Gasoline Range Organic in Water and Soils	S-MN-O-427

ATTACHMENT VA- MINNEAPOLIS LABORATORY SOP LIST CONT. (CURRENT AS OF ISSUE DATE)

Purgeable Total Petroleum Hydrocarbons in Water (8015 Mod / CA LUFT)	S-MN-O-525
Volatiles Water Sample Compositing Procedure	S-MN-O-541
Purgeable Total Petroleum Hydrocarbons in Water (NWTPH)	S-MN-O-555
Determination of Gasoline Range Organics by Method AK101	S-MN-O-556
Preparation and Analysis of Samples for the Determination of Dioxins and Furans by USEPA Method 8290/8290A	S-MN-H-001
Preparation and Analysis of Samples for the Determination of 2,3,7,8-TCDD using USEPA Method 1613B, Drinking Water	S-MN-H-003
Preparation and Analysis of Samples for the Determination of PCDDs, PCDFs, and PCBs by modified USEPA Method 23, TO9, or NY State Guidelines	S-MN-H-005
Method 1668, PCB Congener (WHO List)	S-MN-H-009
Preparation and Analysis of Samples for the Determination of Dioxins and Furans by 8280M	S-MN-H-013
Preparation and Analysis of Samples for the Determination of Chlorinated Biphenyl Congeners	S-MN-H-014
Preparation and Analysis of Samples for the Determination of Polybrominated Diphenyl Ether Congeners	S-MN-H-016
Parent and Alkylated PAH in Solid and Liquid, GCMS SIM	S-MN-H-024
Screening of Chlorinated Biphenyl Congeners by Homolog Group for Track Back Fingerprinting	S-MN-H-025
Preparation and Analysis of Samples for the Determination of Chlorinated Biphenyl Congener	S-MN-H-026
Avalon Software Development	S-MN-H-027
Selected PFAA by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry	S-MN-H-028
Selected PFAA by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry EPA 537 Modified	S-MN-H-029
Selected PFAA by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry; PFPROPRA and PFOA via Isotope Dilution; Matrix Matched Extracted Calibration Curve	S-MN-H-030
Selected PFAA by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry – MPCA Guidance for PFC Analysis	S-MN-H-031
Microwave Digestion of Fish Oil	S-MN-I-301
Digest Procedure for Aqueous Samples to be Analyzed by Induct Coupled Plasma (SW-846)	S-MN-I-458
Metals Analysis by ICP/MS - Method 6020 and 200.8	S-MN-I-492
Preparation of Aqueous Samples for ICPMS Analysis	S-MN-I-523
Microwave Digestion of Oil	S-MN-I-602
Speciated Arsenic Analysis by Hyphenated Technique	S-MN-I-608
Speciated Chromium Analysis by Hyphenated Technique	S-MN-I-609
Speciated Manganese Analysis by Hyphenated Technique	S-MN-I-610
Speciated Selenium Analysis by Hyphenated Technique	S-MN-I-611
Phosphor Powder and Ground Glass Preparation by Hot Block	S-MN-I-306
TCLP/SPLP	S-MN-I-312
Inductively Coupled Plasma Atomic Emission Spectroscopy (RCRA)	S-MN-I-313
Hardness by Calculation	S-MN-I-338
Mercury in Liquid and Solid/Semis-Solid Waste	S-MN-I-359
Percent Solids (Moisture)	S-MN-I-367
Metals Preparation for Solid samples, Wipes and Filters	S-MN-I-460
Metals End Cap Preparation For the Determination of Mercury	S-MN-I-490
Lead in Ambient Air	S-MN-I-601
Preparation of Attic Dust and Filter Cartridge Samples	S-MN-I-604
California Waste Extraction Test (WET)	S-MN-I-612

ATTACHMENT VA- MINNEAPOLIS LABORATORY SOP LIST CONT. (CURRENT AS OF ISSUE DATE)

SAR Calculation	S-MN-I-613
Extractable Base/Neutral and Acid Organic Compounds in Liquid, Solid, and TCLP Matrices by Gas Chromatography/Mass Spectrometry Capillary Column Technique	S-MN-O-436
8270-C/D Extractable Base/Neutral and Acid Organic Compounds in Water and Liquid Matrices by GC/MS Capillary Column Technique w/Selective Ion Monitoring	S-MN-O-507
Extractable Base/Neutral and Acid Organic Compounds in Liquid by EPA Method 625	S-MN-O-532
Analysis of Air samples by GC/MS - Method TO-13	S-MN-O-534
Sulfolane Extraction and Analysis in Liquid Matrices by GCMS	S-MN-O-569
High Volume Injection for 8270C SIM	S-MN-O-570
The Determination Of MN Department Of Agriculture List 2 (MDA2) Pesticides In Aqueous And Solid Samples By GC/MS 8270D	S-MN-O-583
1,4-Dioxane Extraction and Analysis in Liquid Matrices by GC/MS: Capillary Column Technique	S-MN-O-591
Analysis of Volatile Organic Compounds by GC/MS Method 8260	S-MN-O-521
Analysis of Volatile Organic Compounds by GC/MS Method 624	S-MN-O-529
Analysis of Volatile Organic Compounds in Water Method 524.2	S-MN-O-546
Method For Sonicator Tuning	S-MN-O-414
Cleaning Glassware in the Organic Laboratory	S-MN-O-465
Sonication Extraction Technique for Base/Neutral and Acid Compounds (3550C Modified)	S-MN-O-495
Continuous Liquid-Liquid Extraction (SW3520) for Base/Neutral and Acid Compounds	S-MN-O-496
Spike Verification in the Organic Prep Lab	S-MN-O-497
Preparation of Anhydrous Sodium Sulfate for Extraction Purposes	S-MN-O-500
Nitrogen Evaporation Technique	S-MN-O-503
Sample Concentration Technique	S-MN-O-504
Separatory Funnel Extraction for Polyaromatic Hydrocarbons by 8270-SIM	S-MN-O-506
Solvent Exchange into Hexane	S-MN-O-509
Continuous Liq/Liq extraction for Method 8270C (Dual pH) by SW 3520C	S-MN-O-539
Soil Extraction for PAH Analysis by GC/MS:SIM (3550C)	S-MN-O-540
Separatory Funnel Extraction	S-MN-O-566
Preparation For MDA List 2 Herbicides In Aqueous Sample Matrix	S-MN-O-580
Sample Preparation For MDA List2 Herbicides In Solid Sample Using Microwave Extraction	S-MN-O-581
Soil Extraction for PAH Analysis by GC/MS:SIM (3546)	S-MN-O-586
Quality Manual	Quality Manual
Data Archiving and Retrieval	S-MN-L-106
Reagent Water Quality	S-MN-L-110
Generation of EDD	S-MN-L-112
Preventive, Routine, and Non-routine Maintenance	S-MN-L-114
Receipt and Storage of Laboratory Supplies	S-MN-L-117
Data Review Process	S-MN-L-132
Syringe Technique	S-MN-L-139
Procedure for Handling Aqueous Organic Extractable Samples Containing Sediment	S-MN-L-142
Purchasing Laboratory Supplies	S-MN-L-143
Laboratory Housekeeping	S-MN-L-145
Sample Homogenization and Sub-Sampling	S-MN-L-147
Control Chart Generation and Trend Analysis	S-MN-Q-205
Control of Hazardous Energy Program - Lockout/Tagout	S-MN-Q-249
Procedure for Handling of USDA regulated soils	S-MN-Q-253
Estimation of Measurement Uncertainty	S-MN-Q-255
Management of Change	S-MN-Q-257
Proficiency Testing Program	S-MN-Q-258

ATTACHMENT VA- MINNEAPOLIS LABORATORY SOP LIST CONT. (CURRENT AS OF ISSUE DATE)

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Use of A2LA Terms and Symbols	S-MN-Q-260
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Corrective and Preventative Actions	S-MN-Q-262
Monitoring Temperature Controlled Units	S-MN-Q-263
Support Equipment	S-MN-Q-264
Document Control and Management	S-MN-Q-268
Determination of Limit of Detection and Limit of Quantitation (MDL)	S-MN-Q-269
Review of Analytical Requests	S-MN-Q-270
Internal and External Audits	S-MN-Q-271
MCL Violation Reporting	S-MN-Q-272
Preparation of Standard Operating Procedures	S-MN-Q-273
Spreadsheet Validation	S-MN-Q-274
Standard and Reagent Management and Traceability	S-MN-Q-275
Use of the Pacelink System	S-MN-Q-276
Training and Employee Orientation	S-MN-Q-279
Chemical Hygiene Plan/Safety Manual	2017
MN Contingency Plan	2017
Waste Management Training Requirements	S-MN-S-002
Waste Handling and Management	S-MN-S-003
Air Quality Monitoring and Fume Hood Monitoring	S-MN-S-004
Biochemical Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD)	S-MN-I-348
Determination of Hexane Extractable material (HEM) and Silica Gel Treated – Hexane Extractable Material (SGT-HEM) in Solids	S-MN-I-357
Hexavalent Chromium in Water and Wastewater	S-MN-I-358
Alkalinity, Titrimetric	S-MN-I-365
Fluoride in Water and Wastewater	S-MN-I-470
Determination of Total Phosphorus in Aqueous Samples by SmartChem	S-MN-I-473
Specific Conductivity	S-MN-I-474
Settleable Solids	S-MN-I-486
Standard Test Method for Screening Apparent Specific Gravity and Bulk Density Waste	S-MN-I-493
Determination of Total Recoverable Phenolics by Flow Injection Colorimetry	S-MN-I-494
Turbidity	S-MN-I-501
Chlorine, Total Residual in Water	S-MN-I-502
Use and Maintenance of the Konelab	S-MN-I-507
Determination of Nitrate/Nitrite in Surface and Wastewater by SmartChem Colorimetric Analysis	S-MN-I-508
Determination of Chloride by Konelab	S-MN-I-509
Determination of Sulfate by Konelab	S-MN-I-510
Determination of Nitrite by Konelab(Spectrophotometric Method)	S-MN-I-514
Paint Filter Liquids Test	S-MN-I-516
Determination of Hexane Extractable material (HEM) and Silica Gel Treated – Hexane Extractable Material (SGT-HEM) in Water	S-MN-I-520
Dissolved Oxygen	S-MN-I-524
Measurement of pH in Water, Soil, and Waste	S-MN-I-526
Determination of TSP and PM 10	S-MN-I-527
Measurement of Solids in Water and Wastewater	S-MN-I-528
Total CN in Water - Macro Distillation	S-MN-I-529
Weak Acid Dissociable Cyanide in Water - Macro Distillation	S-MN-I-530

ATTACHMENT VA- MINNEAPOLIS LABORATORY SOP LIST CONT. (CURRENT AS OF ISSUE DATE)

Data Correctness Calculations	S-MN-I-562
COD by Hach 2700 (SM 5220D/ EPA 410.4)	S-MN-I-563
Total Recoverable Cyanide and Amenable Cyanide in Water	S-MN-I-564
Delta Airlines Anodizing Line	S-MN-I-582
Determination of Inorganic Anions by Ion Chromatography	S-MN-I-583
Determination of Nitrate/Nitrite on the Lachat by Cadmium Reduction	S-MN-I-584
Determination of Sulfate on the Lachat	S-MN-I-585
Sample Appearance	S-MN-I-586
Determination of Phenolic Compounds by Flow Injection Analysis Colorimetry	S-MN-I-591
Determination of Chloride by Flow Injection Analysis Colorimetry	S-MN-I-592
Determination of Ortho Phosphate in Waters by Flow Injection Analysis Colorimetry	S-MN-I-593
Determination of Hexavalent Chromium by Flow Injection Analysis	S-MN-I-594
Determination of Suspended Sediments	S-MN-I-595
Measurement of Ferrous Iron by Spectrophotometer	S-MN-I-607
Determination of Ammonia by Flow Injection Analysis Gas Diffusion Separation Method	S-MN-I-614
Total Coliform Bacteria	S-MN-MB-001
Fecal Coliform by MF	S-MN-MB-002
Heterotrophic Plate Count	S-MN-MB-003
Total Coliform Bacteria by Membrane Filtration	S-MN-MB-005
Sample Container Sterility Verification	S-MN-MB-006
Total Coliform Bacteria and E. Coliform Bacteria	S-MN-MB-007
Drierite Regeneration Procedure	S-MN-O-557

ATTACHMENT VB- MONTANA LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

DOCUMENT NAME	NUMBER
MT Contingency Plan	2018
Waste Handling and Management	S-MT-S-001
Use of the LogTag Monitoring System	S-MT-Q-002
USDA Regulated Soils	S-MT-Q-003
Manual Integration	S-MT-Q-004
Phosphorus, Ortho and Total	S-MT-I-002
Sulfides	S-MT-I-005
Specific Conductivity SW2510B	S-MT-I-007
Measurement of Solids in Water and Wastewater	S-MT-I-008
The Determination of Nitrate-Nitrite by Flow Analyzer	S-MT-I-009
TKN By Colorimetry	S-MT-I-010
Water Soluble Sulfate and Chloride	S-MT-I-013
The Determination of Percent Moisture in Soil and Solid Samples	S-MT-I-014
Determination of Inorganic Anions by Ion Chromatography	S-MT-I-018
Determination of Ammonia Nitrogen by Automated Phenate	S-MT-I-019
Chlorophyll-a	S-MT-I-020
Measurement of pH in Water, Soil, and Waste	S-MT-I-021
Settleable Solids	S-MT-I-030
Acidity	S-MT-I-032
Multi Increment Soil Sampling	S-MT-I-033
Turbidity	S-MT-I-034
Nitrite by SM4500 NO2B	S-MT-I-036
Total Persulfate Nitrogen	S-MT-I-042
Organic Matter	S-MT-ME-001
Acid-Base Accounting - Sobek	S-MT-ME-004
pH Paste	S-MT-ME-006
Total Sufur by LECO Analyzer	S-MT-ME-012
Available Nitrate and Ammonia	S-MT-ME-022
Particle Size Analysis	S-MT-ME-024
Coarse Fragment	S-MT-ME-035
Soil Sieve	S-MT-ME-041
Mechanical Hydrometer/Grain Size Analysis	S-MT-ME-043
Soil Agronomy Metals Extraction Procedures	S-MT-M-032
Preparation of Aqueous Samples for Analysis by ICP	S-MT-M-037
Preparation of Solid Samples for ICP	S-MT-M-038
Toxicity Characteristic Leaching Procedure (TCLP)	S-MT-M-039
Inductively Coupled Plasma Atomic Emission Spectroscopy	S-MT-M-040
Measurement of Mercury by CVAA	S-MT-M-044
Analysis of Volatile Organic Compounds by GC/MS	S-MT-O-004
The Determination of Extractable Petroleum Hydrocarbons by Method MA-EPH	S-MT-O-001
Petroleum Hydrocarbons as Diesel in Water and Soil	S-MT-O-002
Purgeable Total Petroleum Hydrocarbons in Water and Soil	S-MT-O-003
Volatile Petroleum Hydrocarbons (VPH)	S-MT-O-005
Preparation of Anhydrous Sodium Sulfate for Extraction Purposes	S-MT-O-008

ATTACHMENT VC - VIRGINIA AND DULUTH LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

VIRGINIA SOPS	
DOCUMENT NAME	**NUMBER**
Sample Management	S-VM-C-001
Bottle Preparation	S-VM-C-002
Subcontracting Samples	S-VM-C-003
Reagent Water Quality	S-VM-Q-002
Preventative, Routine and Non-Routine Maintenance	S-VM-Q-003
Data Review Process	S-VM-Q-026
Waste Handling and Management	S-VM-S-001
Waste Management Training Requirements	S-VM-S-002
Air Quality Monitoring and Fume Hood Monitoring	S-VM-S-005
Chemical Hygiene Plan/Safety Manual	Manual
Contingency Plan	2017
Hazard Assessments	S-ALL-S-001
Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry 200.7/6010C	S-VM-M-001
Mercury Analysis by EPA 245.1, 7470, 7471	S-VM-M-004
Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry 200.8/6020A	S-VM-M-006
Alkalinity, Titrimetric SM 2320B	S-VM-I-002
Acidity, Titrimetric SM 2310B	S-VM-I-003
Specific Conductivity/Salinity SM 2510B / SM 2520B	S-VM-I-005
Turbidity EPA 180.1	S-VM-I-006
Total Residual Chlorine, SM 4500 Cl-G	S-VM-I-008
Determination of Chloride, SM 4500 Cl-E	S-VM-I-009
pH, SM 4500 H+B, EPA 9045D	S-VM-I-010
Measurement of Solids in Water and Wastewater SM 2540B,C,D,G, USGS I 3765	S-VM-I-011
Hexavalent Chromium, SM 3500 Cr-B	S-VM-I-012
Determination of Phosphorus, EPA 365.1, SM 4500 P-B	S-VM-I-013
Orthophosphate, EPA 365.3	S-VM-I-014
Determination of Ammonia, EPA 350.1	S-VM-I-015
Determination of Total Kjeldahl Nitrogen, EPA 351.2	S-VM-I-016
Determination of Nitrate/Nitrite, EPA 353.2	S-VM-I-017
Determination of Anions by IC, EPA 300.0	S-VM-I-018
Determination of Total Organic Carbon, SM 5310C, EPA 9060A	S-VM-I-019
Chemical Oxygen Demand by Closed Reflux, Colorimetric Determination SM 5220D	S-VM-I-020
Biochemical Oxygen Demand, Carbonaceous Biochemical Oxygen Demand SM 5210B	S-VM-I-021
Total Coliform and Fecal Coliform Bacteria by MF SM 9222B and SM 9222D	S-VM-I-022
Total Coliform and E.Coli Bacteria by SM 9223B Colilert and Colisure	S-VM-I-023
Heterotropic Plate Count by SimPlate	S-VM-I-024
Sample Container Sterility Verification	S-VM-I-025
Total Coliform and E.Coli Bacteria by SM 9223B QuantiTray	S-VM-I-026
Determination of Chlorophyll a, SM 10200H	S-VM-I-027
Cation/Anion Balance	S-VM-I-029
Determination of Percent Total Solids, SM 2540G	S-VM-I-031
Total Amine Analysis, Spectrometry ASTM D2327	S-VM-I-032
Determination of Sulfide, SM 4500 S2-F	S-VM-I-033
Determination of Oxidation-Reduction Potential (eH,ORP), ASTM D1498	S-VM-I-034
Paint Filter Liquids Test, EPA 9095B	S-VM-I-037
Closed Cup Flash Point, EPA 1010A	S-VM-I-038

ATTACHMENT VC - VIRGINIA AND DULUTH LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

DULUTH SOPS	
DOCUMENT NAME	**NUMBER**
Sample Management	S-DUL-C-001
Bottle Preparation	S-DUL-C-002
Subcontracting Samples	S-VM-C-003
Collection of Tap Water Grab Samples	S-DUL-C-004
Hazard Assessments	S-ALL-S-001
Waste Handling and Management	S-VM-S-001
Air Quality Monitoring and Fume Hood Monitoring	S-ALL-S-005
Safety Manual	N/A
Contingency Plan	N/A
Activate Sludge, Respiration Inhibition Test	S-DUL-BIO-001
Termination of Acute and Chronic Toxicity Tests	S-DUL-BIO-002
Fish Acquisition, Holding and Euthanization	S-DUL-BIO-003
Reference Toxicant Control Chart Limits and Maintenance	S-DUL-BIO-004
Conducting Acute Reference Toxicant Tests	S-DUL-BIO-005
Chronic Reference Toxicant Tests	S-DUL-BIO-006
Training and Documentation for Bioassay	S-DUL-BIO-007
Analytical Balance Usage for Bioassay	S-DUL-BIO-008
Culturing Ceriodaphnia dubia	S-DUL-BIO-009
Conducting Acute Toxicity Tests	S-DUL-BIO-010
Culturing Daphnia magna	S-DUL-BIO-011
Conducting Chronic Toxicity Tests	S-DUL-BIO-012
Brine Shrimp Preparation	S-DUL-BIO-013
Reconstituted and Culture Waters	S-DUL-BIO-014
Glassware Preparation	S-DUL-BIO-015
Bioassay Instruments	S-DUL-BIO-016
Preparation of Selenium and B12 Supplement	S-DUL-BIO-017
Percent Moisture (ASTM D2974-13)	S-DUL-I-001
Hexane Extractable Material (9071B)	S-DUL-I-002
Residual Chlorine, DPD Colorimetric Method	S-DUL-I-003
Total and Ortho Phosphorus (365.1)	S-DUL-I-004
Specific Gravity	S-DUL-I-005
Oil and Grease, Hexane Extraction	S-DUL-I-006
Ion Chromatography	S-DUL-I-007
Organic Nitrogen	S-DUL-I-008
Sulfide, colorimetric	S-DUL-I-009
Surfactants	S-DUL-I-010
Total Suspended Solids (USGS I3765 and SM2540D)	S-DUL-I-011
pH (SM4500H+B)	S-DUL-I-012
Alkalinity	S-DUL-I-013
Turbidity	S-DUL-I-014
Total and Ortho Phosphorus	S-DUL-I-015
Total Dissolved Solids	S-DUL-I-016
Biochemical Oxygen Demand (BOD)	S-DUL-I-017
Determination of Color	S-DUL-I-018
Conductivity, Specific Conductance	S-DUL-I-019
Chemical Oxygen Demand (COD)	S-DUL-I-020
Eh	S-DUL-I-021
Dissolved Oxygen	S-DUL-I-022
Total Solids by SM2540B	S-DUL-I-023
Volatile Solids by EPA 160.4	S-DUL-I-024
Cyanide, Total and Available (SM4500CN-E and G)	S-DUL-I-025
Phenolics	S-DUL-I-026
Nitrate Nitrogen Electrode (SM 4500 NO3 D)	S-DUL-I-027
Chlorophyll A	S-DUL-I-028
Nitrate+Nitrite Nitrogen (Automated Cadmium Reduction) (353.2)	S-DUL-I-029
Ammonia Nitrogen- Automated Phenate Method	S-DUL-I-030
Total Kjeldahl Nitrogen (TKN)	S-DUL-I-031
Chloride Automated Mercuric Thiocyanate Method (SM4500 Cl-E)	S-DUL-I-032
Total Residual Chlorine Amperometric Titration Method	S-DUL-I-033
Nitrite Nitrogen Colorimetric Method	S-DUL-I-034
Dishroom Procedures	S-DUL-I-035

ATTACHMENT VC - VIRGINIA AND DULUTH LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

Hexavalent Chromium	S-DUL-I-036
Fluoride, Ion-Selective Electrode Method	S-DUL-I-037
Sulfite in Water	S-DUL-I-038
Ammonia Nitrogen - Selective Electrode Method (SM4500NH3-D)	S-DUL-I-039
Tannin and Lignin	S-DUL-I-040
Hardness by USGS I-1338-85	S-DUL-I-041
SOUR (Specific Oxygen Uptake Rate) Test	S-DUL-I-042
Dissolved Oxygen Winkler Titration (360.2)	S-DUL-I-043
Total Res. Chlorine LL Amp Titration (SM4500CL E)	S-DUL-I-044
HPC Simplate by SM9215E	S-DUL-MB-001
Fecal Coliforms Membrane Filter (SM9222D)	S-DUL-MB-002
Colilert-18 and Colisure (SM9223B)	S-DUL-MB-003
Methyl Mercury (1630)	S-DUL-M-001
Mercury in Water (1631E)	S-DUL-M-002
Mercury in Solids (1631E)	S-DUL-M-003
Reagent Water Quality	S-DUL-Q-001
Calibration Procedures	S-DUL-Q-002

ATTACHMENT VIA – MINNEAPOLIS LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)
SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Pace Analytical Services, LLC - Minneapolis MN

Accreditation Body	Accrediting Agency	Certification# (or Lab ID)	AIR	ENVIRONMENTAL					DRINKING					
				DW	NPW	WW	HW	SCM	AIR	DW	NPW	WW	HW	SCM	TISSUE
A2LA + DoD	A2LA	2926.01	X		X			X	X	X	X			X	X
Alabama	Dept of Environmental Management	(40770)								X					
Alaska - DW	Dept of Environmental Conservation	(MN00064)		X						X					
Alaska - UST	Dept of Environmental Conservation	17-009	X		X			X			X			X	
Arizona	Dept of Health Services	(AZ0014)	X	X		X	X			X		X	X		
Arkansas	Dept of Environmental Quality	18-041-0 (88-0680)		*		X	X					X	X		
California	CA Environmental Laboratory Accreditation Program (ELAP)	2929		X		X	X			X		X	X		
CNMI Saipan	CNMI Bureau of Environmental and Coastal Quality	MP0003								X					
Colorado	Dept of Public Health and Environment	(MN00064)		X						X					
Connecticut	Dept of Public Health	(PH-0256)		X	X	X		X		X		X		X	
EPA Region 8-Wyoming - DW	US EPA Region 8 (via Minnesota)*	8ThIS-L		8*						8*					
Florida	Dept of Health	E87605 (38-05/24/2018 (E87605)	X	X	X			X	X	X	X			X	X
Georgia	Dept of Natural Resources	(959)		X						X					
Guam	Guam EPA	17-001r								G					
Hawaii	Dept of Health	(MN00064)		X						X					
Idaho - Inorganics	Dept of Health & Welfare	(MN00064)		X											
Idaho - Organics	Dept of Health & Welfare	(MN00064)		X						X					
Illinois	Illinois EPA	004256 (200011)		X	X			X		X	X			X	
Indiana	Dept of Health	(C-MN-01)		X						X					
Iowa	Dept of Natural Resources	(368)		X		X		X		X					
Kansas	Dept of Health and Environment	E-10167		X	X			X		X	X			X	
Kentucky - DW	Energy and Environment Cabinet	90062		X						X					
Kentucky - WW	Energy and Environment Cabinet	90062				X						X			
Louisiana - DEQ	Dept of Environmental Quality	03086 (84596)	X		X			X	X		X			X	X
Louisiana - DW	Dept of Health	LA180001		X						X					
Maine	Dept of Health and Human Services	2017021 (MN00064)	X	X	X			X		X	X			X	
Maryland	Dept of the Environment	(322)		X						X					
Massachusetts	Dept of Environmental Protection	(M-MN064)		X	X										
Michigan	Dept of Environmental Quality	(9909)		X						X					
Minnesota	Dept of Health	1404017 (027-053-137)	X	X	X			X	X	X	X			X	X
Minnesota - MDA	Dept of Agriculture	via MN cert			X			X							
Minnesota - Petroleum	Dept of Health (via Dept of Commerce approval)*	(240	*	*	*	*	*	*	*	*	*	*	*	*	*
Mississippi	Dept of Health	(MN00064)		X						X					
Montana	Dept of Public Health and Human Services	CERT0092		X						X					
Nebraska	Dept of Public Health and Human Services	(NE-OS-18-06)		X						X					
Nevada	Dept of Conservation and Natural Resources	MN000642018-1 (MN10064)		X	X			X		X	X			X	
New Hampshire	NH Environmental Laboratory Accreditation Program (ELAP)	(2081)	X	X	X			X		X					
New Jersey	Dept of Environmental Protection	(MN002)	X	X	X			X	X	X	X			X	X
New York	Dept of Health	(11647)	X	X	X			X		X	X			X	
North Carolina - DW	Dept of Public Health and Human Services	(27700)		X						X					
North Carolina - DEHR	Dept of Environmental Quality	330			X			X							
North Dakota	Dept of Health	R-036		X	X			X		X	X			X	
Ohio - DW	Ohio EPA	(41244)								X					
Ohio - VAP	Ohio EPA - Voluntary Action Program (VAP)	(CL101)	X								X			X	
Oklahoma	Dept of Environmental Quality	2017-053 (9907)		X	X			X		X	X				X
Oregon - NwTPH	OR Environmental Laboratory Accreditation Program	MN300001-009 (MN-300001)			X			X							
Oregon - Secondary	OR Environmental Laboratory Accreditation Program	MN200001-010 (MN-200001)	X	X	X			X		X	X			X	X
Pennsylvania	Dept of Environmental Protection	036-068-00368		X	X			X		X	X			X	
Puerto Rico	Dept of Health	(MN00064)		X						X					
South Carolina	Dept of Health and Environmental Control	74001001 (74001)								X	X			X	
Tennessee	Dept of Environment and Conservation	(TN02818)		X						X					
Texas	Commission on Environmental Quality	T104704192-18-13 (T104704192)	X	X	X			X		X	X			X	
Utah	Dept of Health	MN00064 2017-9 (MN00064)	X	X	X			X	X	X	X			X	
Virginia	Dept of General Services	9491 (460163)	X	X	X			X	X	X	X			X	X
Washington	Dept of Ecology	C486-18a (C486)	X	X	X			X		X	X			X	
West Virginia - DEP	Dept of Environmental Protection	382			X			X			X			X	
West Virginia - DW	Dept of Health & Human Resources	9952 C								X					
Wisconsin	Dept of Natural Resources	(999407970)		X	X			X		X	X			X	X
Wyoming - UST	via A2LA	2926.01			X	X	X	X							

ATTACHMENT VIB- MONTANA LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)
SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Pace Analytical Services, LLC - Billings, MT
Certifications

Accrediting Authority	Accrediting Agency	Certification # (State ID)	Certification Parameters
A2LA	A2LA	3590.01	DW, NPW, SCM
Idaho	DoH&W	MT00012	DW
Minnesota	DoH	1337407 (030-999-442)	DW, NPW, SCM
Montana	DoPH&HS	CERT0040	DW
Washington	DoE	C933	DW, NPW,SCM
Nevada	DoC&NR-DoEP	MT000122018-1	CWA(NPW), NPW(RCRA), SWM(RCRA)
North Dakota	DOH	R-209	SDWA, CWA(NPW),RCRA
North Dakota	DOH	R-209	RCRA (EPA 9056A)
Wyoming (UST)	Via A2LA	Via A2LA	UST
EPA Region 8 + Wyoming (DW)	US EPA Region 8 (via Minnesota)	8TMS-L	DW

ATTACHMENT VIC - VIRGINIA & DULUTH LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE) SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Pace Analytical Services, LLC - Virginia, MN
Certifications
EPA ID: MN01084

State	Agency	Cert #	Parameters			TNI
			DW	NPW	SCM	
Minnesota	Dept of Health	1323599	Y	Y	Y	Primary
Alaska	Department of Environmental Conservation	17-007	N	Y	N	NA
North Dakota	Dept of Health	R-203	N	Y	Y	NA
Wisconsin	Dept of Natural Resources	998027470	N	Y	Y	NA
Montana	Department of Heath and Human Services	CERT0103	Y	N	N	NA
Washington	Department of Ecology	C1007	N	Y	Y	NA

Pace Analytical Services, LLC - Duluth, MN
Certifications
EPA ID: MN00037

State	Agency	Cert #	Parameters			TNI
			DW	NPW	SCM	
Minnesota	Dept of Health	1382680	Y	Y	Y	Primary
Nevada	Dept of Conservation and Natural Resources	MN000372018-1	N	Y	N	NA
North Dakota	Dept of Health	R-105	Y	Y	Y	NA
Wisconsin	Dept of Natural Resources	999446800	Y	Y	N	NA
Montana	Department of Health and Human Services	CERT0102	Y	N	N	NA

ATTACHMENT VII - METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days	
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	$\leq 6^oC$	14 Days	
Alkylated PAHs	8270 M SIM	Water		$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	Yes
Alkylated PAHs	8270 M SIM	Solid		$\leq 10^oC$	1 Year/40 Days	
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days	
Total Alpha Radium (see note 3)	9315	Solid		None	180 days	
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM 4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.	Yes
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.	
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	28 days	
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days	Yes
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)	Yes
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours	Yes
Base/Neutrals and Acids	8270	Solid	8oz Glass	$\leq 6^oC$	14/40 Days	
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6ºC; Na sulfite if Cl present	14/30 Days	Yes
Biomarkers	8270 M SIM	Water	≤ 6ºC; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6ºC; pH<2 1:1 HCl (optional)	Yes
Biomarkers	8270 M SIM	Solid	≤ 10ºC	1 Year/40 Days	≤ 10ºC	
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6ºC	48 hours	Yes
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	30 Days	
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	Client Dependent	
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; ≤ 6ºC	28 Days	
Cation Exchange	9081	Solid	8oz Glass	None	unknown	
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days	Yes
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes	
Chlorophyll	SM10200H	Water	Brown Plastic or aluminum foiled bottle	≤ 6ºC	48 Hours to filtration	
COD	SM5220D, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	Yes
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6ºC; $Na_2S_2O_3$	8 Hours	
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6ºC	8 Hours	
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 6ºC; $Na_2S_2O_3$	8 Hours	
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 6ºC	24 Hours	
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 6ºC; $Na_2S_2O_3$	24 Hours	
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 6ºC	8 Hours	
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	30 Hours after collection	
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6ºC	24 Hours	
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days	
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days	
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days	
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I, N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6ºC; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)	Yes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Diesel Range Organics-Alaska DRO	AK102	Solid	8oz Glass	≤ 6ºC	14/40 Days	
Diesel Range Organics-Alaska DRO	AK102	Water	250 mL HCl Glass	pH<2 HCl; ≤ 6ºC	14/40 Days	Yes
Diesel Range Organics-TPH DRO	8015	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Diesel Range Organics-TPH DRO	8015	Water	500 mL Amber HCl Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7days if unpreserved, 14 if preserved/40 Days	Yes
Diesel Range Organics-TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10ºC	1 Year if frozen/40 Days	
Diesel Range Organics-NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Diesel Range Organics-NwTPH-Dx	Nw-TPH-Dx	Water	250mL HCl Amber Glass	pH <2 HCl; ≤ 6ºC	14/40 Days; 7 Days from collection to extraction if unpreserved	Yes
Diesel Range Organics-Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6ºC	10/47 Days	Yes
Diesel Range Organics-Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass HCl or 250mL HCl	≤ 6ºC; pH <2 HCl	7/40 Days	Yes
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6ºC	1 year	Yes
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	1 year	Yes
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ -10ºC	1 year	Yes
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	30/45 Days	Yes
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6ºC	30/45 Days	Yes
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10ºC	30/45 Days	Yes
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days	
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6ºC; $Na_2S_2O_3$	7/21 Days	Yes
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6ºC; $Na_2S_2O_3$ if Cl present	14 Days	Yes
Endothall	548.1	Water	Amber Glass	≤ 6ºC; $Na_2S_2O_3$	7/14 Days	Yes
Enterococci	EPA 1600	Water	100mL Plastic	≤ 6ºC	8 Hours	
Explosives	8330/8332	Water	1L Amber Glass	≤ 6ºC	7/40 Days	Yes
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days	Yes
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days	
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 6°C	8 Hours	
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	40mLGlass vial	HCL	Immediate	
Flashpoint/ Ignitability	1010	Liquid	500 mL Plastic/Glass	None	28 Days	
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6°C; pH <2 H_2SO_4 or HCl	7/40 Days	Yes
Fluoride	SM4500Fl-C,D	Water	Plastic/glass	None	28 Days	Yes
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days	
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days	Yes
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days	Yes
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)	Yes
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days	Yes
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved	Yes
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days	Yes
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days	Yes
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days	Yes
Glyphosate	547	Water	Glass	≤ 6°C; $Na_2S_2O_3$	14 Days (18 Months frozen)	Yes
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs	
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days	
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days	

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6ºC	14/7 Days if extracts stored ≤ 6ºC or 14/14 Days if extracts stored at ≤ -10ºC	Yes
Hardness, Total ($CaCO_3$)	SM2340B,C/ 130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months	
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	≤ 6ºC; $Na_2S_2O_3$	8 Hours	
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	≤ 6ºC; $Na_2S_2O_3$	8 Hours	
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	14/28 Days	Yes
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C, D	Water	Plastic/Glass	≤ 6ºC	24 Hours (see note 4)	Yes
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C, D	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)	Yes
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)	Yes
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6ºC	24 Hours after extraction	
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months	
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days	
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months	
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10ºC	1 Year if frozen	
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days	Yes
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis if the sample is not oxided in the bottle; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved	Yes
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10ºC	28 Days if frozen	Yes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Mercury, Methyl	1630	Water	Teflon/fluoropolymer or Glass	4 mL/L HCl for fresh water, 2 mL/L H2SO4 for saline samples, or fill to the top with sample so there is no headspace and maintain ≤ 6ºC preservation	6 months if preserved; Distillate – one week if refrigerated; Ethylated distillate – analyze within 48 hours; Or the samples must be acid preserved within 48 hours of sampling	Yes
Mercury	7471	Solid	8oz Glass Jar	≤ 6ºC	28 Days	
Mercury	7470/245.1/ 245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days	Yes
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10ºC	28 Days if frozen	Yes
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days	
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days	
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days	
Metals (ICP/ICPMS)	6010/6020/ 200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days	
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10ºC	180 Days if frozen	Yes
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days	Yes
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	unpreserved	7 Days	Yes
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days	
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	14 Days	
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6ºC	14 Days	Yes
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6ºC	14 Days	
Nitrogen, Ammonia	SM4500NH3/ 350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	Yes
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6ºC	28 Days	
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	Yes
Nitrogen, Nitrate	SM4500-NO3/ 352.1	Water	Plastic/Glass	≤ 6ºC	24 Hours preferred	Yes
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6ºC	28 Days	
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/ 353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	Yes
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/ 353.2	Water	Plastic/Glass	≤ 6ºC	48 Hours	Yes
Nitrogen, Organic	SM4500-Norg/ 351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	Yes
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days	

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours	
Odor	SM2150B	Water	Glass	≤ 6ºC	24 Hours	
Oil and Grease/HEM	1664A/ SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6ºC	28 Days	Yes
Oil and Grease/HEM	9071	Solid	Glass	≤ 6ºC	28 Days	
PBDEs	1614	Water	1L Amber Glass	≤ 6ºC	1 Year/1 Year	Yes
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6ºC	1 Year/1 Year	
PBDEs	1614	Tissue	Aluminum Foil	≤ -10ºC	1 Year/1 Year	Yes
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days	
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass		Pest: 7/40 Days; PCB: 1 Year/1 Year	Yes
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; < 6ºC	14/30 Days	Yes
Perchlorate	331	Water	Plastic/Glass	>0-6ºC	28 Days	Yes
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10ºC	1 Year if frozen/40 Days	Yes
Pesticides, Organophospho-rous (OP)	8141	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Pesticides, Organophospho-rous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	1 Year/1 Year	Yes
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6ºC	1 Year/1 Year	
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10ºC	1 Year if frozen/1 Year	Yes
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6ºC but above freezing	1 Year/1 Year	Yes
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6ºC but above freezing	1 Year/1 Year	
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10ºC	1 Year/1 Year	YEs
Oil Range Organics- ORO		Solid	8oz Glass Jar	≤ 6ºC	14/40 Days	
Oil Range Organics- ORO		Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes	
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A	Yes
Particulates	PM-10	Air	Filters	None	180 Days	Yes
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days	
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	14 Days	
pH	SM4500H+B/ 9040	Water	Plastic/Glass	None	15 minutes	Yes
pH	9045	Solid	Plastic/Glass	None	Contact local lab	Yes
Phenol, Total	420.1/420.4/ 9065/9066	Water	Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days	Yes
Phosphorus, Orthophosphate	SM4500P/ 365.1/365.3	Water	Plastic	Filter; $\leq 6^oC$	Filter within 15 minutes, Analyze within 48 Hours	Yes
Phosphorus, Total	SM4500P/ 365.1/365.3/ 365.4	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days	Yes
Phosphorus, Total	365.4	Solid	Plastic/Glass	$\leq 6^oC$	28 Days	Yes
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days	
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days	
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days	Yes
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	$\leq -10^oC$	1 Year if frozen/ 40 Days	Yes
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days	Yes
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days	Yes
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days	Yes
Radium-228 (see note 3)	9320	Solid				
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	$\leq 6^oC$	14/40 Days	
Saturated Hydrocarbons	SOP S-MN-O-567	Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)	Yes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Saturated Hydrocarbons	SOP S-MN-O-567	Solid	≤ 10ºC	1 Year/40 Days	≤ 10ºC	
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6ºC	28 Days	
Solids, Settleable	SM2540F	Water	Glass	≤ 6ºC	48 Hours	Yes
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6ºC	7 Days	Yes
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6ºC	7 Days	
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6ºC	7 Days	
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6ºC	7 Days	Yes
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6ºC	7 Days	Yes
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6ºC	7 Days	Yes
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6ºC	7 Days	Yes
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6ºC, Field Filtered	28 Days	Yes
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days	
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg	
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg	
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days	
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days	
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6ºC	28 Days	Yes
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days	Yes
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days	
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6ºC	7 Days	
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes	
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6ºC	48 Hours	Yes
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days	
Total Organic Carbon (TOC)	9060/Walkley Black	Solid	Glass	≤ 6ºC	28 Days	
Total Organic Halogen (TOX)	SM5320/9020/9021	Water	Glass; no headspace	≤ 6ºC	14 Days	Yes
Tritium	906.0	Water	Glass	None	180 days	
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6ºC	48 Hours	Yes
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HCl	180 days	

Parameter	Method	Matrix	Container	Preservative	Max Hold Time	Additional Volume for MS/MSD
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6ºC	14 Days preserved	Yes
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6ºC; packed jars with no headspace	7/28 Days	
Volatiles	TO-14	Air	Summa Canister	None	30 Days	
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	Client Dependent	
Volatiles	TO-15	Air	Summa Canister	None	30 Days	
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours	
Volatiles	8260	Solid	5035 vial kit	See note 1	14 days	Yes
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	14 Days	Yes
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6ºC	14 Days	
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)	Yes
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6ºC; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days	Yes
UCMR3 Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days	
UCMR3 Hexavalent Chromium	218.7	Water	HDPE or propylene	Na_2CO_3/$NaHCO_3$/$(NH_4)_2SO_4$; pH>8	14 Days	
UCMR3 Chlorate	300.1	Water	Plastic or glass	EDA	28 Days	
UCMR3 Hormones	539	Water	Amber glass	$Na_2S_2O_3$, 2-mercaptopyridine-1-oxide, sodium salt	28 Days	
UCMR3 Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days	
UCMR3 Volatiles	524.3	Water	40 mL amber glass vials	Ascorbic acid. Maleic acid pH~2	14 Days	
UCMR3 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days	
UV254	SM5910B	Water	Glass	≤ 6ºC	48 Hours	

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4ºC, **and** one vial methanol preserved at ≤6ºC **and** one container of unpreserved sample stored at ≤6ºC.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

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Review

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Quality Manager	03 Oct 2018, 12:49:16 PM	Reviewed

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STANDARD OPERATING PROCEDURE

DETERMINATION OF FIXED GASES IN AIR

Reference Methods: Modified 3C, ASTM D1946-90/2006

Local SOP Number:	S-MN-A-005-rev.15
Effective Date:	Date of Final Signature
Supersedes:	S-MN-A-005-rev.14

APPROVALS

Laboratory General Manager — Date: 07OCT2016

Laboratory Quality Manager — Date: 07 Oct 2016

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-A-005-Rev.15

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1. Purpose/Identification of Method

1.1. This is a gas chromatographic (GC) method for determining fixed gases in air/vapor samples as delineated in EPA Method 3C and ASTM D1946.

2. Summary of Method

2.1. Samples are collected in sampling bags or Summa® canisters. The samples are stored and analyzed at ambient temperatures.

2.2. The J&W Scientific GS-GASPRO 60m x 0.32mm id column (-80 to 300°C) and a Liquid Nitrogen (LN2) cryogenic valve are used for the determination of fixed gases in air by Modified 3C and ASTM D1946 analysis. A Thermal Conductivity Detector (TCD) is used for all compound identification.

2.3. Compound identifications are made by analyzing gas standards containing the targeted compounds, under the same conditions used for samples, and comparing resultant compound retention times. Quantitative determinants are made by comparing the response produced for each compound from a known concentration standard to the response produced for each compound detected in the sample. Compound response in the sample is compared to a calibration curve to determine the analyte concentration in the sample followed by normalization to 100%.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. Parameters: This SOP applies to compounds listed in Attachment I. Additional compounds may be analyzed if all quality control criteria are met. The range of sensitivity for this method generally is in the percent level.

4. Applicable Matrices

4.1. This SOP is applicable to whole air.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes can be found on Attachment I. All current MDL's are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Carrier gas can pick up small amounts of contaminants in the distribution system. A purifier or filter should be installed to eliminate this contamination.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment, and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Air	Summa® canister	None	Ambient	Must be analyzed within 28 days of collection.
	Tedlar® bag (or equivalent)	None	Ambient	Must be analyzed or transferred to a Summa ® canister within 14 days of collection

8. Definitions

8.1. Absolute Pressure-PSIA—Pounds/Square Inch Absolute. Pressure measured with reference to absolute zero as opposed to atmospheric pressure. Can also be expressed as kPa or mm Hg. (To covert PSIG to PSIA add 14.7)

8.2. Analyte – The specific entity an analysis seeks to determine.

8.3. Batch – A grouping of no more than twenty samples of similar matrix which are prepared and/or analyzed together with the same method and the same lots of reagents within the same time frame, as designated by the method.

8.4. Gauge Pressure-PSIG—Pounds/Square Inch Gage. Pressure measured in pounds/square inch at ambient atmospheric pressure. Can also be expressed as mm Hg psig.

8.5. Reporting Limit (RL) – The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, unless otherwise specified. Reporting limits are generally two times the MDL.

8.6. Standard – A substance or material, the properties of which are known with sufficient accuracy, to permit its use to evaluate the same property in a sample.

9. Equipment and Supplies

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item#/Description
Gas Chromatograph	An analytical system complete with a temperature programmable oven for separation of target analytes.	Hewlett Packard 5890 or equivalent
Analytical Column	60m x 0.32mm id column	J&W Scientific/113-4362/GS-GasPro or Equivalent
Thermal Conductivity Detector	0.65 Watts, 12VDC, C9 Indent, 0.40x0.40 orifice, negative polarity	Precision Dynamics/G3313-S13/TCD
Tedlar Bag	Tedlar Bag or equivalent	
Summa® Canister	Summa® Canister or equivalent	
Data System	A computer system that allows the continuous acquisition and storage on machine-readable media of all chromatography obtained throughout the duration of the chromatographic program.	Agilent ChemServer or equivalent
Data Processing Software	A computer system that allows for the integration and reduction of data against a calibration curve for the determination of the concentration of each analyte.	Target 4.1 or equivalent
Data Package and Review Software	Compiles pdf images of all the data to be available for package generation and secondary review.	Gandalf or equivalent
Data Reporting Software	Laboratory Information Management System (LIMS)	Horizon or equivalent

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
3CNorm Primary Standard	He 36% CO 4.0% CH4 40% CO2 20%	Praxair ME CD20CH2C-A3
3CNorm Second Source	He 36% CO 4.0% CH4 40% CO2 20%	GTS/Welco MECD20CH2C
O_2N_2 Primary Standard	O_2 20% N_2 80%	Praxair NI OX20C-A3
O_2N_2 Second Source Standard	O_2 20% N_2 80%	GTS/Welco NIOX20C

Note: Oxygen and methane cannot be purchased in a single standard due to concerns of shipping cylinders containing mixtures in the explosive range.

11. Calibration and Standardization

11.1. Table 11.1 ICAL levels (%)

Compound	0.1 mL loop	0.5 mL loop	1.0 mL loop
CO	0.4	2	4
CO_2	2	10	20
CH_4	4	20	40
He	3.6	18	36
The O_2N_2 standard is injected separately			
O_2	2	10	20
N_2	8	40	80

11.2. Table 11.2 Calibration and Standardization

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Averaged Response Factor Linear Regression	±20% $r \geq 0.995$	If not met, re-perform ICAL
Second Source Verification Standard (ICV)	Immediately after each initial calibration	CCV criteria ±10% unless otherwise specified in a QAPP	If the requirements for ICV are not met, verify if there are any apparent issues with the initial analysis. Reanalyze one more time. Only two injections of the same standard are permitted prior to recalibrating the instrument.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and every 10 environmental samples.	% Diff ±20%. Client, QAPP, or state requirements may supersede.	If the requirements for continuing calibration are not met, verify if there are not any apparent issues with the initial analysis prior to reanalysis of standards. Only two injections of the same standard are permitted prior to recalibrating the instrument.

12. Procedure

12.1. Instrumental Analysis

12.1.1. If the sample is in a canister, the canister must be pressurized to a point where sample can be obtained from it.

12.1.1.1. Using a pressure gauge, measure and record the pressure on the sample tag.

12.1.1.2. Add argon if necessary until the pressure reads 0psig. The connection from the house line to the can should have a pressure gauge in series for accurate measurements of canister pressure.

12.1.1.2.1. Make sure that argon is used for this pressurization step. Other inert gases may interfere with the analysis.

12.1.1.2.2. Record the final canister pressure on the sample tag. Also note on the tag the date the can was filled, the initials of the person doing the work, and the gas used to fill the canister.

12.2. After the analytical system is demonstrated to be in control, an aliquot of sample is removed from either the sampling bag or pressurized Summa® canister.

12.3. If any compounds are above the calibration range, a dilution is run. This is achieved by using smaller loops until a value that is in the calibration range is obtained.

12.4. Target analytes are quantitated using the external standard method and normalization to 100%.

13. Quality Control

13.1. Table 13.1 Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Summa® canister pressurized with Argon	One per 10 samples or less	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze associated samples. ***Exceptions:*** If sample is ND, report sample without qualification; If the sample result is greater than 10 times the method blank detection, the sample can be reported without qualification; If the sample result is less than 10 times the method blank detection and the sample cannot be reanalyzed, report the sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Tedlar bag filled with primary standard	One per 10 samples or less	±30%, or internally generated limits updated annually.	Analyze a new LCS; Perform system maintenance prior to new LCS run ***Exceptions:*** If LCS recovery is greater than the QC limits and these compounds are not detected in the associated samples, the sample data may be reported without qualification.
Laboratory Control Sample Duplicate (LCSD)	Tedlar bag filled with primary standard	One per 10 samples or less	± 30%, or internally generated limits updated annually. %RPD: 30%	Analyze a new LCS; Perform system maintenance prior to new LCS run ***Exceptions:***

				If LCS recovery is greater than the QC limits and these compounds are not detected in the associated samples, the sample data may be reported without qualification.
Sample Duplicate	Duplicate sample analysis	One per 10 samples or less	%RPD: 30%	Flag data if the RPD exceeds 30%.

14. Data Analysis and Calculations

14.1. Calculation of Dilution Factor

$$\text{Dilution factor} = \frac{(P_F - P_I) + 15}{15}$$

Where: P_F = Final canister pressure in psig
P_I = Initial canister pressure in psig

14.2. Normalization is automatically performed by the data processing method.

14.3. Calculation of Sample Concentration

$$C_x = \frac{(A_x)(DF_{Norm})}{(Rx)}$$

Where: C_x = Concentration of compound x in %
A_x = Area of compound x
DF_{Norm} = Dilution factor from Equation
R_x = Average RF for compound x from the most recent calibration curve

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 and 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See tables in section 11 and 13.

17. Contingencies for Handling Out-Of-Control Data or Unacceptable Data

17.1. If not specifically listed in the table in section 11 or 13, the contingencies are as follows: if there is no additional sample volume to perform re-analysis, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. The frequency and criteria of the duplicate has been modified from 3C. A sample will be analyzed in duplicate per each set of ten injections, between continuous calibration checks and system blanks.

19.2. The initial calibration was verified using a second source standard, which is not required by the method.

19.3. Argon is used as the carrier gas since Helium is a target analyte.

20. Instrument/Equipment Maintenance

20.1. Please refer to the GC 5890 instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Please refer to the GC 5890 instrument manual for troubleshooting steps performed by the lab.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Department of Defense (DoD) Quality Systems Manual – most current version

25.5. EPA Method 3C – "Determination of carbon dioxide, methane, nitrogen, and oxygen."

25.6. Standard Test Method for Analysis of Natural Gas by Gas Chromatography, ASTM D 1945-03, 2010

25.7. Standard Practice for Analysis of Reformed Gas by Gas Chromatography, ASTM D 1946-90, 2006.

25.8. References to Normalization:

25.8.1. Section X1.2.5.3 of ASTM 1945

25.8.2. Section 9.5 and 10.2.1 of ASTM 1946

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – List of Target Analytes with MDLs and RLs

26.2. Attachment II – ALTEF Sampling Bag Study (5 days)

26.3. Attachment III – ALTEF Sampling Bag Study (14 days)

27. Revisions

Revison Number	Reason for Change	Date
S-MN-A-005-Rev.14	12.1.1.2 – updated verbiage to include 'if necessary'	02Sep2015
S-MN-A-005-Rev.15	Updated to Pace Analytical Services, LLC throughout document	07Oct2016

ATTACHMENT I - Target Analytes with MDLs and RLs

Target Analyte	MDL (in %)	RL (in %)
Helium	0.87	3.6
Oxygen		2.0
Nitrogen		8.0
Carbon Monoxide	0.16	0.40
Methane	0.48	4.0
Carbon Dioxide	0.29	2.0

ATTACHMENT II – ALTEF Sampling Bag Study (5 days)

Pace Analytical®	Document Name: **ALTEF Sampling Bag Study**	Document Revised: 01May2012 Page 1 of 2
	Document No.: **F-MN-A-139-rev.00**	Issuing Authority: Pace Minnesota Quality Office

Pace's % Methane Recovery Study using ALTEF® Sampling Bags Supports a 5 day analysis holding time

ATLEF® Study and Product Information:

With the bulk of Tedlar® production going into the solar panel industry, sampling bag vendors have developed new materials that require testing prior to use. Each new product is thoroughly tested for stability in the lab before selecting the material that works best for each specific analysis. It was in this context that we discovered the ATLEF® sampling bag is ideally suited for % methane analysis. With a specially formulated film based on poly (vinylidene fluoride) or PVDF the ALTEF® sampling bag material is ideally suited for % methane analysis.

ALTEF® bags can be ordered from Factory Direct Safety & Environmental INC. The part number for a one liter bag is FD02-01-01A1. Here is a link that will provide more detail about the ATLEF® product.

http://www.factorydirectsafety.com/products/gas-sampling-bags/altef-bags/

Summary of ATLEF® Recovery Study:

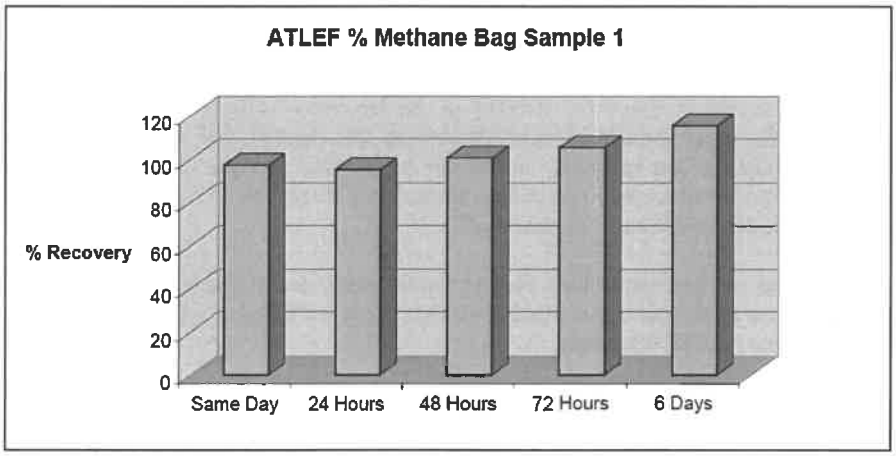
- Two ATLEF® sampling bags were filled with 40% Methane and analyzed on a GC/TCD.
- True value of the contents of the bag was 40% and the graphs below list the percent recovery based on the instrument calibration for method 3C/ ASTM1946.
- The range of concentrations for Methane over six days were 36-46%. This is +/- 15% RSD of the expected true value of 40%.
- Acceptable criteria quality control for the method 3C/ASTM1946 is +/-20% RSD. This criteria applies calibration curves, continuing calibration checks, laboratory control spikes, and laboratory sample duplicates.

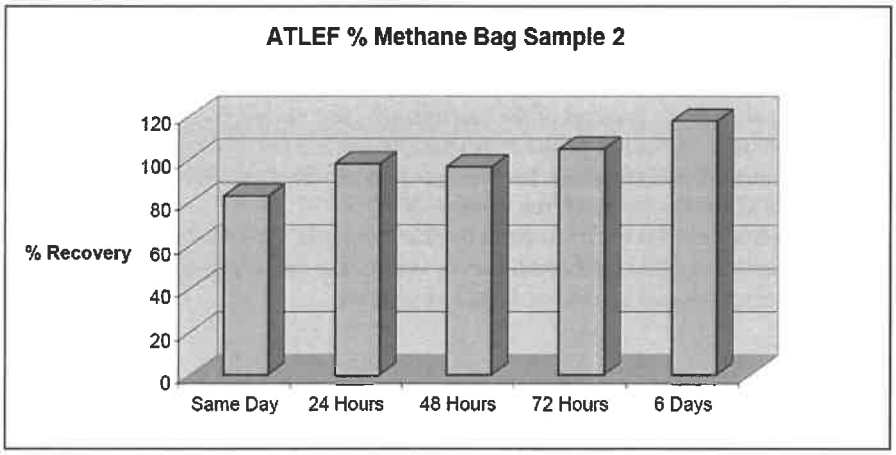
ATTACHMENT II – ALTEF Sampling Bag Study (5 days) - continued

Pace Analytical®	Document Name: **ALTEF Sampling Bag Study**	Document Revised: 01May2012 Page 2 of 2
	Document No.: **F-MN-A-139-rev.00**	Issuing Authority: Pace Minnesota Quality Office

Results of ATLEF® Recovery Study:

ALTEF® sampling bags have proven stability for the use of Methane collection for up to five full days from the collection date. Percent Recovery results from our two sample study are listed below.





ATTACHMENT III – ALTEF Sampling Bag Study (14 days)

Pace's % Methane Recovery Study Using ALTEF® Sampling Bags Supports a 14 day Analysis Holding Time

Summary of ATLEF® Recovery Study:

- ATLEF® real-life sampling bags were filled onsite and analyzed for methane on a GC/TCD.
- Samples are analyzed first within the accepted 5-day hold time and again at 14 days.
- The concentration of the bag is calculated based on the instrument calibration for method 3C/ ASTM1946 and normalized to 100%.
- The relative percent difference for methane as compared to the original concentration ranges from 1.2% to 2.9%.
- Acceptable criteria quality control for the method 3C/ASTM1946 is +/-30% RPD.

ATLEF® Study and Product Information:

With the bulk of Tedlar® production going into the solar panel industry, sampling bag vendors have developed new materials that require testing prior to use. Each new product is thoroughly tested for stability in the lab before selecting the material that works best for each specific analysis. It was in this context that we discovered the ATLEF® sampling bag is ideally suited for % methane analysis. With a specially formulated film based on poly(vinylidene fluoride) or PVDF the ALTEF® sampling bag material is ideally suited for % methane analysis.

ALTEF® bags can be ordered from Factory Direct Safety & Environmental INC. The part number for a one liter bag is FD02-01-01A1. Here is a link that will provide more detail about the ATLEF® product.

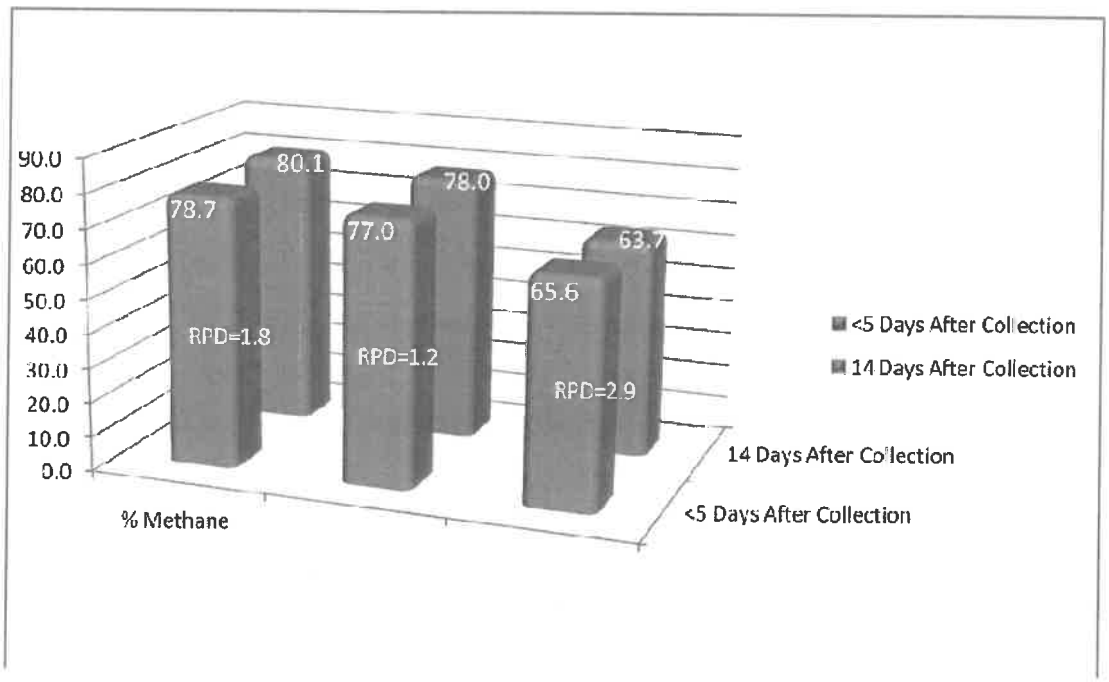
http://www.factorydirectsafety.com/products/gas-sampling-bags/altef-bags/

Results of ATLEF® Recovery Study:

With ATLEF® sampling bags we have demonstrated that we can extend the hold time for % methane analysis from five days to fourteen days from sample collection.

ATTACHMENT III – ALTEF Sampling Bag Study (14 days) - continued

ALTEF® sampling bags have proven stability to recover % methane up to two full weeks from the collection date. Relative percent difference (RPD) results from our three real-life sample study are listed below.



Document Information

Document Number: ENV-SOP-MIN4-0005 **Revision:** 01

Document Title: Analysis of Whole Air Sample for Volatile Organic Compound by GC/MS EPA TO15/TO14

Department(s): Air

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Notes

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Signature Manifest

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Title: Analysis of Whole Air Sample for Volatile Organic Compound by GC/MS EPA TO15/TO14

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0005

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Quality Manager	23 Oct 2018, 11:40:44 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	Assistant General Manager	23 Oct 2018, 12:52:53 PM	Approved
David Randall (008925)	Manager - Lab Services	23 Oct 2018, 01:07:40 PM	Approved

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. The purpose of this Standard Operating Procedure (SOP) is to provide quality control and analytical guidance for the analysis of whole air samples and soil vapor samples contained in Summa ® passivated canisters, Silco ® lined canisters (or equivalent), or sampling bags using gas chromatography/mass spectrometry. This SOP is based on Environmental Protection Agency (EPA) Compendium Method TO-15/TO-14. All analysis performed under the Ohio VAP program is only to be evaluated based on the criteria specified in EPA Compendium Method TO-15.

2. SUMMARY OF METHOD

2.1. Samples are received in Summa ® canisters or Silco ® lined canisters (or equivalent). The gauge pressure upon arrival is measured and recorded. The canister is then pressurized to positive psi gauge pressure using an inert gas. The canister is connected to an autosampler tree and preconcentrator system, which concentrates the sample prior to injection into a GC/MS. The data is then analyzed for the desired volatile organic compounds.

2.2. This method addresses an extensive set of VOCs by incorporating a multisorbent, dry purge technique for water management.

2.3. An aliquot of the whole air sample is concentrated prior to gas chromatographic (GC) separation and mass spectrometry (MS) full scan detection or Select Ion Monitoring (SIM) detection. Samples expected to contain VOCs in a range of 0.005 parts per billion by volume (ppbv) to 500 ppbv can be analyzed by this technique.

2.4. If samples are received in sampling bags, see section 7.0 for appropriate holding times and actions to transfer the samples to a Summa ® canister to complete analysis as described in this SOP.

3. SCOPE AND APPLICATION

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. Parameters: This procedure is designed to analyze whole air samples collected in Summa ® canisters, Silco ® lined canisters (or equivalent), or sampling bags for some of the volatile organic compounds (VOCs), or hazardous air pollutants (HAPs), found in Title III of the Clean Air Act Amendments of 1990. This SOP is related to only those VOCs that have been found to be stable when collected in Summa ® polished stainless steel canisters, Silco ® lined canisters or sampling bags (or equivalent). VOCs are defined as any organic compound having an initial boiling point less than or equal to 250°C measured at 1 atm. Attachment I lists target VOCs applicable to this method.

3.3. This SOP is based on the EPA Compendium Method TO15 which can also be applied to TO14. As such, this SOP serves to cover both analyses. See EPA Compendium Method TO15 Section 3 and Attachment I for compound list.

3.4. For Ohio VAP, if requirements specified in this SOP are not able to be met, Pace Analytical will narrate any potential bias or justification for reporting in the project narrative on the final report. Additional narratives are provided as needed on a case by case basis in the event of the following occurrences: instrument failure, limited sample volume, report revisions or matrix interferences.

3.5. All references contained in this SOP with regards to spiking volumes, standard concentrations and instrument configuration are based on recommended parameters and may be subject to change based on instrument technology and application.

4. APPLICABLE MATRICES

4.1. This SOP is applicable to whole air samples and soil vapor samples contained in Summa ® passivated canisters, Silco ® lined canisters (or equivalent), or sampling bags using gas chromatography/mass spectrometry.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. The most current reporting and detection limits can be found in the Laboratory Information Management System (LIMS).

6. INTERFERENCES

6.1. Carrier gas potentially contains small amounts of contaminants and is filtered prior to use in instrumentation. Other interferences are sample specific and are dealt with as they occur.

6.2. Interferences in samples can result from contamination of the canisters. To minimize this problem, processes must be implemented to ensure that the canisters are contamination free. See SOP ENV-SOP-MIN4-0002 - Procedure for Cleaning, Certification, Leak Checking, and Preparation for Shipment of SUMMA Passivated Canisters, or equivalent replacement.

6.3. Contamination of analytical equipment can also occur when samples containing high concentrations of VOCs are analyzed. The resulting "carryover" contamination varies from system to system. The analyst needs to use best judgment when evaluating sample data following samples with large detection levels.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Collection, Preservation, Storage and Holding Time Table

7.1.1. The holding time indicated below is the maximum allowable time from collection to analysis per the analytical method. If the samples fail to meet the holding time, data will be qualified accordingly on the analytical checklist and on the final report with the appropriate footnote.

7.1.2. Note for Ohio VAP: As applicable, to the best of the laboratory's knowledge, if holding times are not met the laboratory will qualify the data accordingly indicating the bias present due to the exceedance.

Sample type	Collection per sample	Preservation	Storage	Hold time
Air	Samples are collected into evacuated Summa ® canisters, Silco ® canisters (or equivalent). The canisters are then shipped back to Pace Analytical Services, LLC for analysis.	None	Ambient sample storage	Samples collected in Summa ® canisters, Silco ® canisters (or equivalent) must be analyzed within 30 days from collection. Samples collected in Minnesota are to be collected in canisters and must be analyzed within 14 days of collection per the Minnesota Pollution Control Agency (MPCA). If samples have been collected in bags, the samples need to be transferred to a Summa Canister within two days to maintain a 30 day holding time. The holding time is potentially extended to 72 hours per client specific QAPPS. Collection in a bag may result in higher reporting limits. See Attachments VIII-X for instructions and documentation for the transfer procedure. Ohio VAP samples must be transferred to a Summa Canister within two days from collection to extend the holding time of collection to analysis to 30 days.

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Absolute canister pressure = Pg + Pa, where Pg = gauge pressure in the canister (kPa, psig) and Pa= barometric pressure.

8.3. Absolute pressure - Pressure measured with reference to absolute zero as opposed to atmospheric pressure, usually expressed as kPa, mm Hg or psia.

8.4. Cryogen - A refrigerant used to obtain very low temperatures for sample concentration. A typical cryogen is liquid nitrogen (bp - 195.8°C).

8.5. Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

8.6. Gauge pressure - Pressure measured above ambient atmospheric pressure as opposed to absolute pressure. Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

8.7. MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

8.8. MS-SIM - The GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly.

8.9. Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

8.10. Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Equipment and Supplies Table

Supply	Description	Vendor/ Item # /
Gas Tight Syringes	0.010, 0.025, 0.05, 0.1, 0.25, 0.5, 1, 5, and 10 mL	Fisher, or equivalent
Neat liquid standards	at least 95%	O2Si, or equivalent
Glass static dilution flask	2L, equipped with a Mini-inert cap	Fisher, or equivalent
Oven	capable of maintaining a temperature of 65°C	Fisher, or equivalent
Summa ® passivated canisters or Silco ® lined canisters (or equivalent)	6L or 1L capacity	Restek
Dual pressure/vacuum gauge	high accuracy	Omega Engineering, or equivalent
Nitrogen		Praxair
Organic free water	DI Water	n/a
Gas Chromatograph	Equipped with a split/splitless injection port and electronic pressure control (EPC) or equivalent. See 9.1.1 and 9.1.2 for operating parameters.	Agilent Technologies 6890N or equivalent
Mass Selective Detector	With Chemstation operating software and WinTarget data processing software or equivalent. See 9.1.3 for operating parameters.	Hewlett Packard 5973 or equivalent
Pre-concentrator	With 7016 canister manifold autosampler. See 9.1.4 for operating parameters.	Entech 7200
Capillary Column	DB-5 60m x 0.32mm capillary column or DB-624 60m x 0.32mm with a 1.8 µm film thickness or equivalent.	J & W Scientific
Helium Cylinder	High purity grade high-pressure helium cylinder for column carrier gas equipped with a dual stage pressure regulator.	Praxair
Chemstation	Data Acquisition Software	See master list for current version
Target	Data Processing Software	See master list for current version
EPIC Horizon	Data Reporting Software (LIMS)	See master list for current version
Gandalph	Data Packaging Software	See master list for current version

9.1.1. Chromatograph Suggested Operating Parameters:

9.1.1.1. Initial temp: 40°C

9.1.1.2. Ramp A: 10°C/min to 150°C

9.1.1.3. Ramp B: 35°C/min to 240°C

9.1.1.4. Hold 1 min

9.1.1.5. EPC Pressure: 9 psi

9.1.1.6. Temp 250°C

9.1.1.7. Split Flow 20mL/min

9.1.2. Injection port parameters:

9.1.2.1. EPC pressure: 9 psi

9.1.2.2. Temperature: 250°C

9.1.2.3. Purge valve: Initial value On, Off time 0.0 min.

9.1.2.4. Split flow: 20 mL/min.

9.1.3. Suggested Mass spectrometer parameters:

9.1.3.1. Electron volts: 70 nominal

9.1.3.2. Scan range: 35 to 300 amu

9.1.3.3. Scan time: At least 2 scans/peak, not to exceed 1 sec/scan

9.1.3.4. Interface temp: 250°C

9.1.3.5. The GC/MS system must be set up to meet manufacturer's specification. The mass calibration and resolution of the GC/MS are verified by the analysis of the tune standard, p-bromofluorobenzene (BFB). For more information, refer to the Chemstation User's Guide and the GC/MS User's Guide.

9.1.4. Entech Pre-Concentrator suggested settings:

9.1.4.1.

During Concentration	Temperature (°C)
Module No. 1, Empty Cryotrap	-40
Module No. 2, Sorbent Packed Cryotrap	-40
Focusing Trap	-160

9.1.4.2.

Desorb/Transfer/Inject	Preheat (°C)	Final Temp(°C)
Module No. 1, Empty Cryotrap	10	10
Module No. 2, Sorbent Packed Cryotrap	-50	230
Focusing Trap	N/A	N/A

9.1.4.3.

Media Concentrated/Transferred	Volume (cc)	Flow Rate (sccm)
Internal Standard & Surrogate	20	50
Sample	5 to 300	150
Sweep/Dry Purge	75	100
Transfer to Packed Column	40	15

9.1.4.4. Sample Transfer

Line Conditioning Sample Flush Before Trapping	10 sec
Carrier Flush Before Trapping	2 to 4 min.
Sample Transfer to Focusing Trap	2 to 4 min.
Sample Injection	0.6 to 2 min.

9.1.4.5.

System Bakeout	Temperature (°C)	Time (min.)
Module No. 1	150	7
Module No. 2	220	7

9.1.4.6.

Regulated Zones	Temperature (°C)
8-Port Valve	100
GC Transfer Line	110
Manifold Transfer Line	100
16-Position Select Valve	100
Sample Container	Ambient

10. REAGENTS AND STANDARDS

10.1. Target analyte standards are obtained from various vendors and verified for accuracy.

10.1.1. Calibration Mix is used for Initial Calibration (ICAL), Continuous Calibration (CCV) and Laboratory Control Spike (LCS). Second Source is used for initial calibration verification. Surrogate, Tuning and Internal standard solutions are obtained from vendors in solution form. These solutions are stored per manufacturer's specifications and have an expiration date of one year after being opened or the manufacturer's expiration date, whichever is sooner.

10.2. Reagents and Standards Table

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Calibration Standard	This is a custom mix that includes all compounds of interest at 1-5ppmv.	The calibration standard is purchased in the form of a pressurized cylinder from a source independent of the second source verification mix (Spectra Gas, Linde, Custom Gas Solutions, or equivalent).
Initial Calibration Verification (second source standard)	This is a custom mix that includes all compounds of interest at 1ppmv.	The ICV is purchased in the form of a pressurized cylinder from a source independent of the calibration mix.
Internal Standard/ Surrogate/ BFB Standard	This is a custom mix that includes all internal standards, surrogate, and tuning standard at 10ppmv.	The internal, surrogate, and BFB standards are purchased in the form of a pressurized cylinder (Restek or equivalent).

10.3. Working Standard Dilutions and Concentrations

10.3.1. All standards prepared into canisters in the air lab will be assigned a 30 day holding time, similar to the air samples. Upon expiration, the expired standard is removed from use and a new standard must be prepared.

Standard	Standard(s) Amount	Concentration of Std	Solvent	Solvent Volume	Final Total Volume	Final Concentration
30 ppbv Calibration Standard	1350cc	1-5ppmv	Nitrogen	15 L Can	30psig	30-150 ppbv
1 ppbv Calibration Standard	1500cc	30 ppbv	Nitrogen	15 L Can	30 psig	1-5 ppbv
Initial Calibration Verification Standard (ICV) (Second Source)	1350cc	1 ppmv	Nitrogen	15L Can	30psig	30 ppbv
Internal Standards/Surrogate/BFB Standard	675cc	10 ppmv	Nitrogen	15L Can	30psig	150 ppbv
Method Blank	value less than RL	N/A	Nitrogen	300cc	300cc	less than RL
LCS	100cc	30ppbv	N/A	N/A	100cc	10ppbv

Ical Level	Concentration	Calibration Standard Used	Amt of Cal Standard Used
Level 1	0.100-0.500 ppbv	1-5 ppbv std	30 cc
Level 2	0.200-1.00 ppbv	1-5 ppbv std	60 cc
Level 3	0.500-2.50 ppbv	1-5 ppbv std	150 cc
Level 4	1.00-5.00 ppbv	1-5 ppbv std	300 cc
Level 5	10.0-50.0 ppbv	30-150 ppbv std	100 cc
Level 6	20.0-100. ppbv	30-150 ppbv std	200 cc
Level 7	30.0-150. ppbv	30-150 ppbv std	300 cc

10.3.2. Calibration Standard 30-150 ppbv: Using the 1000cc gas tight syringe, pull 675cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister that has been humidified with 50ul H_2O. A second pull of 675cc is pulled from the TO15 Stock Standard Cylinder and transferred to the evacuated 15L canister. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 30-150 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.3. Calibration Standard 1-5 ppbv: Using the 1000cc gas tight syringe, pull 750cc from the 30-150 ppbv Calibration Standard into a clean, evacuated 15L canister that has been humidified with 50 µL H_2O. A second pull of 750cc is pulled from the 30-150 ppbv Calibration Standard and transferred to the evacuated 15L canister. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 1-5 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.4. ICV 30 ppbv: Using the 1000cc gas tight syringe, pull 675cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H_2O. A second pull of 675cc is pulled from the TO15 Stock Standard Cylinder and transferred to the evacuated 15L canister. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 30 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.5. The ICV (Second Source Standard) is analyzed by injecting 100cc from the 30-150 ppbv ICV/Second Source standard (see above table)

10.3.6. To prepare Internal Standard/Surrogate/BFB: Using the 1000cc gas tight syringe, pull 675cc from the 10ppmv TO15 Internal Standard/Surrogate Stock Cylinder into a clean, evacuated 15L canister, that has been humidified with 50 µL H_2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 150 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

10.3.7. The tune standard, p-bromofluorobenzene (BFB), must be 50ng or less on column.

10.3.7.1. The tune standard can be evaluated from the continuing calibration verification (CCV) standard so long as all criteria can be evaluated and met, since the BFB is present in all injections.

10.3.8. Internal standard compounds and surrogate standard compounds are used in the analysis.

10.3.8.1. Internal Standards: 1,4-Difluorobenzene and Chlorobenzene-d5

10.3.8.2. Surrogate*: p-bromofluorobenzene (BFB)

*Surrogates are not a method requirement and therefore only reported at specific request of the client. Surrogates are not evaluated for Ohio VAP samples.

11. CALIBRATION AND STANDARDIZATION

11.1. Calibration Criteria Table

Calibration Metric	Parameter / Frequency	Criteria	Comments
Instrument Tune	Before any standard, method blank, or sample analysis can occur using the GC/MS system, it must be demonstrated that the GC/MS is capable of producing compliant spectra when p-bromofluorobenzene (BFB) is analyzed. Attachment II lists the required spectral criteria. The instrument performance check must be analyzed initially and once every 24-hour period. The tune period begins at the time of injection of the BFB.	If the BFB spectrum meets the criteria listed in Attachment II, proceed with standard and sample analysis. If the BFB spectrum fails to meet the criteria listed in Attachment II, the MS must be retuned. Repeated failures potentially indicate the need for MS maintenance such as cleaning the ion source.	The GC/MS is set up according to the manufacturer's specifications. The MS source and mass filter are adjusted by monitoring the mass spectra of Perfluorotributylamine (PFTBA). Prepare a standard solution of BFB at a concentration that allows the collection of 50ng or less under the optimized concentration parameters (see Section 10.3). This is met by injecting 20cc during the trapping. The BFB is introduced into the system through microscale purge and trap. The spectrum of BFB must be acquired by averaging three scans; the apex and the scans that immediately proceed and follow the apex. Background subtraction is accomplished using a single scan taken before the BFB peak.
Initial Calibration (ICAL)	Initial Calibration is performed as a result of extensive instrument maintenance, CCV outliers for analytes of interest, Internal Standard Non Compliance, and at the discretion of the analyst. All standards, method blanks, laboratory control spikes (LCS), and samples must be analyzed using the same conditions. A calibration curve must consist of a minimum of 5 standards (6 for quadratic) and spans the expected monitoring range established for each compound of interest to determine instrument response and linearity. The lowest level of the curve must be at or below the reporting limit for each analyte. A typical calibration curve can cover a range from 0.1 to 30 ppbv. Section 10.3	The %RSD for all calibrated target compounds must be ±30%. Alternately for all target compounds, linear regression is used with an r^2 value of 0.995 or greater. A quadratic curve is utilized if the r^2 (equals COD in Equation 12) value is 0.990 or greater and six calibration points are included in the curve. Curves must not be forced through zero. *For Ohio VAP: quadratic curve fit is only to be used for analytes that have historically exhibited nonlinear response.* The area response for each internal standard in each calibration level must be within 40% of the area response of the mid-point of the initial calibration. The relative retention time (RRT) of each compound must agree within ± 0.06 RRT units of the RRT from the mid-point of the initial calibration curve. Per the Pace Quality Assurance Manual, the reporting limit standard must be evaluated to	Initial calibrations are not meant to be a replacement of necessary instrument maintenance. Calibration curve fits are possible indicators of instrument performance or deterioration. Analytes that traditionally are average or linear responders that suddenly display quadratic curve fits could be a sign of a system that is deteriorating. Quadratic cannot be used to extend the calibration range for compounds that normally exhibit a linear response, perform necessary maintenance to return the system to good working order. This is not to eliminate the use of quadratic curve fits, some analytes always present a quadratic response and that is acceptable. If an analyte fails to meet ICAL criteria, the analyst should report sub list of compounds and/or re-analyze samples under compliant conditions. For initial calibrations not meeting criteria, evaluate the

	contains standard preparation information. Calibration points may be dropped on the lower or upper end so long as the criteria listed above is met. If lower calibration points are dropped, the reporting limit of the compound must be raised to the lowest remaining calibration point if it exceeds the current compound reporting limit. If upper calibration points are dropped, samples must be interpreted with the new upper calibration point representing the new upper range for the compound. At no point is it acceptable to drop a mid-point calibration from an initial calibration.	determine if the curve fit is presenting bias. The level corresponding to the reporting limit must be quantitate back after processing the curve and be ± 40% of the expected true value. See section 11.2 for corrective actions.	system for potential issues. Perform instrument maintenance if necessary. Prepare new standards and rerun the initial calibration. Calibration is performed using the internal standard technique. See Attachment III for internal standard groups. The data is evaluated using WinTarget. See section 11.2 for acceptance criteria.
Initial Calibration Verification (ICV)	A second source standard must be analyzed following an initial calibration curve which contains all the analytes of interest.	The spike level of the ICV must be near the midpoint level of the calibration curve. The ICV is considered acceptable if the recoveries of the analytes fall within 60-140%. See section 11.2 for corrective actions.	The Entech 7200 Concentrator automatically adds 20cc of the internal standards and surrogate (Section 10.3) to each analysis during trapping. Using the Target data processing software, evaluate the calibration data. See Section 11.2 for corrective actions.
Continuing Calibration Verification (CCV)	After an acceptable tune has been evaluated, the initial calibration curve for each compound of interest must be checked and verified before sample analysis can occur each day. This is accomplished by analyzing continuing calibration verification (CCV) standard at 10-50 ppbv (see section 10.3 Ical Level 5). The CCV is the same source as the ICAL standard. The CCV is analyzed after a compliant tune once every 24-hour period during sample analysis unless an initial calibration is analyzed. If there is an initial calibration performed, it will take the place of the CCV and no	The %D for each target compound in the continuing calibration verification (CCV) standard must be less than or equal to 30 percent. The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve. See 11.3 for corrective actions.	Calculate the RRF for each target compound from the continuing calibration standard using Equation 5 (see section 14). See 11.3 for corrective action. Note: If a CCV fails biased high and the associated samples are non-detect, the samples may be reported as the high bias has no impact on the non-detect results.

	CCV will be analyzed until after the next tune period.		

11.2. Corrective Action for Initial Calibration Verification (ICV)

11.2.1. If ICV fails criteria, the analyst must consult with his or her supervisor or manager before moving forward. Possible corrective actions include:

11.2.1.1. The system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second ICV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance.

11.2.1.2. Check pressure on the standard canister.

11.2.1.3. Check system for leaks.

11.2.1.4. Check to see that standards were made correctly.

11.2.2. If the ICV was injected during a period in which it could not be evaluated immediately and was followed by a SDG, data impact is evaluated and reported with necessary footnotes pending supervisor or manager approval.

11.2.3. If the technical acceptance criteria fail for the initial calibration curve, inspect the system for any possible leaks. A high baseline and reduced response potentially indicates a leak.

11.2.4. Examine the response factors of each calibration level. If the response factors of all the compounds for one level appear to be significantly different, analyze that same level calibration standard again.

11.2.5. If the same results occur after reanalysis, a new standard canister must be made and analyzed.

11.2.6. If a leak or other system problem cannot be found, try to clean the ion source or perform column maintenance.

11.2.7. Samples to be injected for analysis following the initial calibration should not be scheduled, where possible, until an acceptable initial calibration and ICV have been analyzed. In the event the calibration curve or ICV cannot be evaluated prior to sample scheduling, such as the case with the initial calibration being injected after normal hours of operation, the initial calibration and ICV must be reviewed for failures prior to data workup of scheduled samples. In the event of failures in the initial calibration or ICV, client samples are reviewed for potential impact and reanalyzed or reported with adequate technical justification. Technical justification would be if there are samples available that require only a short list of analytes that exclude the failures, analysis may continue for those analytes or if a high bias is identified and the samples are non-detect for target analytes. Clearly document which elements are not acceptable on the analytical checklist for secondary review by a data validator. For samples being evaluated under the Ohio VAP program, all compounds must meet specifications. Those samples not meeting these criteria must be reanalyzed under compliant conditions.

11.2.8. Recalibration must be performed if any major change has been made to the GC/MS system such as replacing the GC column, cleaning the MS source or repair.

11.3. Corrective Action for Continuing Calibration Verification

11.3.1. If the CCV does not meet criteria, the system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second CCV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance.

11.3.1.1. Check pressure on the standard canister.

11.3.1.2. Check system for leaks.

11.3.1.3. Check to see that standards were made correctly.

11.3.1.4. Document all maintenance and corrective action measures taken in the maintenance logbook, run logbook or checklist accordingly based on the actions taken.

11.3.2. If, following corrective action attempts, the CCV does not meet criteria, then a new calibration curve must be analyzed.

11.3.3. Samples are not to be scheduled for analysis until CCV criteria has been met or technical justification has been given for the analysis to continue. Technical justification includes the scheduling of samples that meet CCV criteria for a shorter list of volatile compounds. In the event the CCV cannot be evaluated prior to the scheduling of samples for analysis, as in the case where the CCV is scheduled for injection after standard business hours, the subsequently scheduled samples will be evaluated for impact based on the results for the associated CCV, and either reanalyzed or narrated to indicate impact if reanalysis is not possible. For samples being evaluated under the Ohio VAP program, all compounds must meet specifications. Those samples not meeting these criteria must be reanalyzed under compliant conditions.

12. PROCEDURE

12.1. Analytical Sequence

12.1.1. The following is the GC/MS analytical sequence for samples each 24-hour period:

12.1.1.1. CCV, which also serves as the Laboratory Control Standard for the batch of twenty samples. Two CCVs are permitted to be analyzed on each system before an Initial Calibration is needed.

In the event the CCV is not compliant for analytes of interest in the samples, and Initial Calibration is required before sample analysis. An ICAL is followed then by the ICV. The ICV may be used as the Laboratory Control Standard or LCS.

12.1.2. Laboratory Method Blank: 300cc of nitrogen from a clean 6L canister.

12.1.2.1. 20 field samples

12.1.2.2. Sample duplicate, minimum of one in 20 samples

12.1.2.3. Any necessary dilutions from previously analyzed samples (see the dilution preparation section of Attachment V).

12.1.2.4. In the event that time remains in the 24-hour tune period, an additional method blank and LCS must be analyzed in order to analyze additional reportable samples.

12.2. Sample Analysis

12.2.1. Upon receipt, the canister pressure of each sample is measured and recorded on the canister sample tag.

12.2.1.1. If the canister pressure is less than 5 psig, the canister pressure must be increased before analysis can occur.

12.2.1.1.1. Add clean nitrogen or helium gas to the sample canister. For a six-liter canister, 5 psig is the desired final pressure. A one-liter canister requires a final pressure of 10 psig for adequate sample volume for analysis.

12.2.1.1.2. Record the final canister pressure on the canister sample tag noting which gas was added. Also, note the information in the final analytical results report.

12.2.1.1.3. Calculate the resultant dilution factor using Equation 16 in section 14.16.

12.2.1.1.4. This dilution factor is applied to Equation 17 in section 14.17.

12.2.2. Once the GC/MS system is demonstrated to be in control, an aliquot of the air sample is removed from the canister and pre-concentrated using the Entech 7200 pre-concentrator and 7016 autosampler manifold.

12.2.3. Analyze the samples under the same operating conditions as the instrument calibration and quality control samples.

12.2.4. Analyze a duplicate sample for every 20 samples analyzed.

12.2.5. If time remains in the 24-hour tune period in which an initial calibration was performed, it is possible to continue to analyze samples without the analysis of a CCV standard.

12.2.6. If the tune period has expired, an instrument tune, CCV standard, and method blank must be analyzed before samples can be analyzed.

12.2.7. If time remains in the tune period after a batch of no more than 20 samples and its re-runs have been analyzed, it is possible to analyze additional samples after a new LCS and method blank have been analyzed.

12.2.8. Technical Acceptance Criteria can be found in Section 12.7.

12.2.9. Procedures for the determination of Air Phase Petroleum Hydrocarbons (APH) can be found on Attachment XI. Reporting of APH can only be conducted per each state or client acceptance of the data, i.e. Ohio VAP only allows the analysis of the TO15 analytes listed in Table 1 and as specified the Pace scope of accreditation. See the most current certificates for the approved analyte lists, any analytes not approved must be clearly indicated on the report and affidavits as being compounds not certified by the VAP program.

12.3. Qualitative Analysis

12.3.1. The compounds listed in Attachment I are identified by an analyst competent in the interpretation of mass spectra. Sample mass spectrum is compared to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the target compound identifications: (1) elution of the sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

12.3.2. The relative retention time (RRT) of the sample component must agree within ± 0.06 RRT units of the RRT of the standard component using the CCV standard as reference.

12.3.3. Standard and sample mass spectra are compared using reference spectra obtained on the GC/MS system being used. The mass spectra used for comparison are from the same standard as that being used for RRT comparison. Mass spectral requirements are as follows:

12.3.3.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.3.3.2. The relative intensities of ions specified above must agree within ± 20% between the standard and sample spectra.

12.3.3.3. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process favors false positive.

12.3.4. Non-target sample components are library searched using the latest NIST library for the purpose of tentative identification. These components are referred to as TICs - Tentatively Identified Compounds) and are noted as such in any final report with a qualifier of "J" unless the client specifies differently. The "J" qualifier indicates an estimated value. Guidelines for identification are as follows:

12.3.4.1. Characteristic ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample.

12.3.4.2. The relative intensities of the major ions must agree within ± 20%.

12.3.4.3. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for background contamination or presence of co-eluting peaks.

12.3.4.4. If in the technical judgment of the analyst, no valid identification can be made, the compound is to be reported as an unknown with possible classification, such as hydrocarbon.

12.3.4.5. TIC searches are reported only upon client request. These results are considered estimated and do not fall under any scope of accreditation held by Pace.

12.4. Identified target analytes are quantitated using the internal standard method using the extracted ion current profile (EICP) area of the characteristic ions of analytes listed in Attachment III. This ion is referred to as the quantitation ion.

12.5. The RRF from the initial calibration curve analysis is used to quantitate samples and method blanks. Calculate the concentration of the sample component using Equation 17 in section 14.17.

12.5.1. Additional curve fit equations are in section 14.

12.6. The internal standard method of quantitation is also used to determine an estimated concentration for Tentatively Identified Compounds (TIC). The nearest internal standard to the TIC is used as a reference to estimate the concentration of the TIC. If the nearest internal standard exhibits interferences, the next closest internal is used. The estimated concentration is obtained using Equation 17 with the following exceptions:

A_x =Total ion chromatogram area of the TIC,
A_i =Total ion chromatogram area of the specific internal standard;
R_f =1.0

Estimated TIC concentrations are flagged with a qualifier of "J" which indicates that the quantitated amount is an estimate. TICs are not considered certified analytes under any scope of accreditation. If TICs are reported, they must be clearly indicated as not being certified analytes under the program that the work is related to.

12.7. General Technical Acceptance Criteria

12.7.1. For data to be reported without qualification, the following criteria must be met for all samples, CCVs, method blanks, and laboratory control sample (LCS).

12.7.1.1. The EICP area response for each internal standard must be within ±40% of the EICP area response in the midpoint of the most recent Initial Calibration. See Attachment III for a list of analytes and assigned internal standards.

12.7.1.2. The retention time for each of the internal standards must be ±0.33 minutes of each of the internal standard (IS) retention times in the most recent ICAL 10.0 Standard.

12.7.1.3. Recoveries for surrogate standard compounds (where required) must fall within ±30% of the true value.

12.7.2. If the technical acceptance criteria are not met, samples must be reanalyzed with appropriate batch QC to confirm results under compliant operating conditions. This confirmation is performed by reanalyzing the corresponding QC that was out of range with the samples and if found to be not in range, narrative for bias will be noted, as applicable. See Section 11.2 and 11.3 for corrective action for calibration failures and Section 13.1 for all other samples (including QC). If, in the determination of the analyst, reanalysis of a sample could negatively impact results, possibly due to a need to pressurize a canister, data may be qualified and reported with the approval of a supervisor or manager. All reports with nonconformance to the technical criteria must be fully narrated to the client. For work performed under the Ohio VAP program, narration of nonconformance must include an indication of bias in the data.

12.7.3. There will be times in which analytical results are obtained outside the linear range due to highly contaminated sites. Pace makes every attempt to dilute the samples within the linear range of the instrument. If sufficient dilutions cannot be made to achieve results within the linear range because of sample size or concentration, the data will be reported qualified as estimated. If the bias can be noted, it will be qualified accordingly in the final report, i.e. biased high or biased low.

13. QUALITY CONTROL

13.1. QC Table

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	A clean canister filled with humidified nitrogen is analyzed on the GC/MS system to demonstrate that the system is free of interferences. The Method Blank is prepared in the same manner as any standard or sample and	Analyzed once every 24-hour period or every 20 samples, whichever comes first. The Method Blank is analyzed before samples can be analyzed, and	An instrument blank analysis is allowed after any sample that has known VOCs present that exceed the upper calibration limit of the method to demonstrate that the system is free of possible carryover effects. When possible, historical	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report

	analyzed in the same manner.	after daily ICAL or CCV.	data can be used to determine if there are high levels of contaminants present, possibly causing carry over in the system. The method blank must not contain any target analyte at a concentration equal to or greater than its reporting limit and must not contain additional compounds with elution characteristics and mass spectral features that interfere with identification and measurement of a method analyte. The internal standard must be within ± 40% of the mean area response of the IS in the most recent calibration. The retention time of each of the internal standards must be within ± 0.33 minutes between the method blank and the most recent calibration standard.	sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. NOTE: For Ohio VAP samples, if the detection is above the reporting limit and corrective actions as listed in this table do not result in acceptable data, the samples must be re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results. NOTE: Specific clients may require method blanks be analyzed from canisters that were previously certified as clean and that have not left the laboratory.
Laboratory Control Sample (LCS)	The laboratory control standard is prepared from the same standard as the calibration standard) as outlined in section 10.3. The LCS standard is from the same source as the ICAL standard.	A LCS must be analyzed once every 24-hour period or every 20 samples, whichever is more frequent.	The percent recovery for each analyte in the LCS must be within the internally generated recovery limits and can be found in the LIMS system.	If a LCS fails to meet the recovery limit criteria, inspect the system for the possibility of a poor sampling. If the LCS fails and no error in sampling was found, preparation and injection of a second analysis can be conducted. If that second analysis fails, the system must be recalibrated and all affected samples must be reanalyzed. If the samples cannot be reanalyzed, qualify the data accordingly with an appropriate footnote on the final report indicating the bias present. For Ohio VAP samples, if the outlier is an analyte of interest and corrective actions as listed in this table do not result in acceptable data, the QC and samples must be

				re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results. Failures that produce a high bias with samples that show results as non-detect may be reported for Ohio VAP.
Duplicate Samples	Client provided samples.	Duplicate sample analysis is performed once per 20 samples	The RPD between the sample and the sample duplicate must be < 25%.	If the RPD fails to meet criteria, the instrument must be evaluated to determine if there was an error with the analysis. If there is no evidence of malfunction, the parent sample must be reanalyzed to confirm results. If the data confirms, report the original data and qualify the bias accordingly. Contact the client for further instructions. The client can choose to re-submit the sample or have the lab qualify the data and narrate as appropriate. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.
Internal Standards	Internal standard is added to every injection at a concentration of 10ppbv.	Aliquot is added to each analysis at the pre-concentrator.	The EICP area response for each internal standard must be within ±40% of the EICP area response in the mid-point of the initial calibration. See Attachment III for a list of analytes and assigned internal standards.	Examine the instrument for possible errors or malfunctions and correct any discovered. Re-analyze the samples and associated batch QC: samples, method blanks, laboratory control spike, and sample duplicates). Report the reanalyzed sample results accordingly. If there is no evidence of error or malfunction, re-analyze the affected QC and samples. If the data confirms, report the original data and qualify the bias accordingly. Unless matrix interference was detected, Ohio VAP samples must be re-analyzed undiluted. If the outlier corrective actions do not result in acceptable data, the samples must be re-analyzed. The narrative for any report that includes qualified data must also include a discussion of any bias in the results.

Surrogate	Labeled compounds that behave similarly to target analytes that are meant to represent the efficiency of the system related to the matrix	Included in each injection per client specific QAPPs. Surrogates will not be injected into any samples or standard/QC solutions for analysis intended for Ohio VAP certified data.	Internally generated control limits. Most current limits are found in the LIMS.	Confirm that there are no errors in the calculations, surrogate solutions, and internal standards. Verify instrument performance. If no problems are found, prepare and reanalyze the sample. If the reanalysis is within limits and holding times, then report only the reanalysis. If the reanalysis is within limits, but out of hold, then report both sets of data. If the reanalysis is still out of limits, then report both sets of data.

14. DATA ANALYSIS AND CALCULATIONS

14.1. See the most current Quality Manual for calculations

14.2. Concentration of each component in the flask (Static Dilution Technique, section 10.4.1)

Equation 1

$$\text{Concentration}(\text{mg/L}) = \frac{(V_i)(d)}{V_f}$$

where:

V_i=Volume of liquid neat standard injected into the flask in mL;
d=Density of the liquid neat standard in mg/mL;
V_f=Volume of the flask in liters.

Caution: In the preparation of standards by this technique, make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask.

14.3. The concentration in ppbv of each component in the flask is determined using Equations 2 and 3 (Static Dilution Technique, section 10.4.1)

14.3.1. First determine the volume of the compound as a gas using Equation 2:

Equation 2

$$V = \frac{nRT}{P} \quad \text{where,} \quad n = \frac{(V_i)(d)}{M}$$

where,
V=Volume of injected compound at STP in liters;
n=Moles;
R=Gas constant (0.08206 L-atm/mole °K);
T=Ambient temperature in °K;
P=Ambient pressure in atm;
V_i=Volume of liquid neat standard injected into the flask in mL;
d=Density of the neat standard in g/mL;
M=Molecular weight of the compound in g/mole.

14.3.2. Now calculate the concentration in the flask in ppbv using Equation 3:

Equation 3

$$\text{ppbv} = \frac{V}{V_f}(10^9)$$

where:
V=Gas volume of compound as determined in Eq. 8 in liters;

V_f=Volume of static dilution flask in liters.

14.4. The concentration in ppbv of each compound in the canister can be determined using Equation 4 (Static Dilution Technique, section 10.4.1)

Equation 4

$$\text{ppbv} = \frac{(V_i)(C_x)}{V_c}$$

where:
V_i=Volume removed from static dilution flask and injected into the canister in liters;
C_x=Concentration of compound x in the static dilution flask in ppbv;
V_c=Final canister volume in liters.

14.5. Relative Response Factor (RRF): Tabulate the area response of the primary ion (Attachment III) for each compound and the associated internal standard. Use the internal standard, which has a retention time nearest to the compound of interest. Calculate the relative response factors (RRF) for each compound using Equation 5:

Equation 5

$$\text{Relative Response Factor (RRF)} = \frac{(A_x)(C_i)}{(A_i)(C_x)}$$

where,
A_x=Area of the primary ion for compound x to be measured;
A_i=Area of the primary ion for the internal standard associated with compound x;
C_i=Concentration of the internal standard in ppbv;
C_x =Concentration of compound x to be measured in ppbv.

14.6. Mean Relative Response Factor. Calculate the mean RRF for each compound using the RRF from the five (or six, where n=6)-point calibration using Equation 6:

Equation 6

$$\overline{R_f} = \frac{\sum_{n=5} R_f}{n}$$

where,
$\overline{R_f}$ =Average relative response factor;
R_f=Relative response factor from calibration curve;
n=Number of data points.

14.7. Standard Deviation ($\sigma_{(n-1)}$).

Equation 7

$$\sigma_{(n-1)} = \sqrt{\sum_{i-1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

14.8. %Relative Standard Deviation (%RSD). Using the average RRF from Equation 6 and the standard deviation from Equation 7, calculate the %RSD using Equation 8:

Equation 8

$$\%\text{RSD} = \frac{S_{(n-1)}}{\overline{R_f}} x100$$

14.9. Mean area response for Internal Standard:

Equation 9

$$\bar{y} = \sum_{i=1}^{n} \frac{y_i}{n}$$

where,
$\bar{y}$ = mean area response

y = Area response for the internal standard for each initial calibration standard

14.10. If a linear regression is used, the regression produces the slope and intercept terms for a linear equation according to Equation 10:

Equation 10

$y = ax + b$

where:
y = instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = the intercept, do not include the origin (0) as a calibration point

14.11. To calculate the sample concentration by the internal standard method using the linear regression equation, use Equation 11:

Equation 11

$C_s=[(A_sC_{is}/A_{is})-b]/a$

where:
A_s = Area of the peak for the target analyte in the sample
A_{is} = Area of the peak of the internal standard
C_s = Concentration of the target analyte in the calibration standard
C_{is} = Concentration of the internal standard
a = Slope of the line (also called the coefficient of C_s)
b = The intercept

14.12. To calculate the coefficient of determination (or r^2) for a quadratic curve fit, use Equation 12:

Equation 12

$$COD = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - Y_i)^2}{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2}$$

where:
y_{obs} = Observed response for each concentration from each initial calibration standard
y = Mean observed response from the initial calibration (See equation 6)
Y_i = Calculated response at each concentration from the initial calibration (See Equation 5)
n = Total number of calibration points in the equation, 6 points for quadratic
p = Number of adjustable parameters in the polynomial equation

14.13. Calculate the sample concentration by the internal standard method using the quadratic regression by comparing peak heights to the calibration curve.

Equation 13

Regression equation (quadratic):

$y = ax^2 + bx + c$

14.14. Percent Difference (%D). The %D in the RRF of the daily RRF of an individual compound compared to the mean RRF for that compound in the most recent calibration curve is determined as follows:

Equation 14

$$\%D = \frac{|R_i - R_c|}{R_i}(100)$$

where,
R_i=The average RRF from the initial calibration curve for compound *x*;
R_c=RRF for compound *x* from the CCV standard.

14.15. Calculate the percent recovery of the LCS using Equation 15:

Equation 15

$$\text{Percent Recovery} = \frac{C_q}{C_a}(100)$$

where:
C_q=Quantitated concentration of compound x in ppbv;
C_a=Actual concentration of compound x in ppbv.

14.16. Calculate the resultant dilution factor using Equation 16:

Equation 16

$DF = (P_f + 14.7) / P_i + 14.7)$
where:
P_i = Pressure reading of canister prior to pressurization (psig)
P_f = Pressure reading of canister after pressurization (psig)
DF = Dilution factor

To convert Hg to psig:
Multiply by 0.491559 or divide by 2.036

PSIG reading is converted to One Atmosphere:
One Atmosphere = 14.7 psig = 29.21 inches of Hg

See Attachment V for the application of dilution factors for filling canisters.

14.17. Calculate the concentration of the sample component using Equation 17:

Equation 17

$$C_x = \frac{(A_x)(C_i)(D_f)}{(A_i)(R_f)}$$

where:

C_x=Concentration of compound x in ppbv;
A_x=EICP area of the quantitation ion for compound x;
C_i=Concentration of the internal standard associated with compound x in ppbv;
D_f=Dilution factor from Equation 12 (if no dilution was performed, D_f equals 1.)
A_i=EICP area of the quantitation ion for the internal standard associated with compound x;
R_f=Average RRF for compound x from the most recent calibration curve.

14.18. The RPD between the sample and the sample duplicate can be calculated using Equation 18:

Equation 18

$$RPD = \frac{|A-B|}{(A+B)/2} \times 100$$

Where:
RPD = Relative Percent Difference
A = Sample Value
B = Duplicate Value

14.19. Convert ppbv to µg/m^3 using Equation 19:

Equation 19

$$\frac{(x\ ppbv \times MW\ \frac{g}{mol})}{24.055\ \frac{L}{mol}} = y\ \frac{\mu g}{m^3}$$

Where:

MW = Molecular Weight
24.055 L/mol = Molar Volume of an ideal Gas

$$PV = nRT$$

$$V = \frac{nRT}{P}$$

Where:

V = Volume in liters
N = mols of ideal Gas (1 mol)
R = Ideal gas constant
T = Temperature in Kelvin

$$V = \frac{(1mol) \times (0.082 \frac{(L \times atm)}{(mol \times K)} \times 293.15K}{1\ atm}$$

$$V = 24.055 \frac{L}{mol}$$

14.20. Preparation of Working TO15 Standard can be calculated using equation 20:

Equation 20

$$\frac{X}{Y} \times C = Z$$

Where:

X = Volume (L) spiked from stock
Y = Volume (L) of container
C = Concentration (ppbv) of Stock
Z = Concentration (ppbv) of working standard

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. See tables in section 11 & 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. See tables in section 11 & 13.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

17.2 Any data that is reported not meeting method specifications will be qualified accordingly using footnotes in the LIMS or custom qualifiers using the text field. These footnotes will be designated next to the analytes impacted with a letter/number combination with a summary of definition in the footnote section of the final report. Depending on the client data quality objective, an additional case narrative may be included in the final report and the qualifiers will be summarized in that section of the report as well. As indicated throughout the document, Ohio VAP requires the bias be included in the data qualification and associated case narrative.

18. METHOD PERFORMANCE

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Three performance criteria are used to demonstrate method validity which are: (1) method detection limit (MDL), (2) replicate precision, and (3) accuracy - % recovery of LCS.

18.3. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per ENV-SOP-NW-0018 (or equivalent replacement), Method Detection Limit Studies for each matrix per instrument.

18.4. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) ENV-SOP-NW-0025 (or equivalent replacement), Training and Employee Orientation.

18.5. **Periodic performance evaluation (PE)** samples are analyzed periodically to demonstrate continuing competence per SOP ENV-SOP-NW-0011 (or equivalent replacement). Results are stored in the Quality office.

19. METHOD MODIFICATIONS

19.1. Pace utilizes 1,4-Difluorobenzene and Chlorobenzene-d5 as internal standards. This is a modification from the three recommended internal standards in the method. Pace has demonstrated with MDLs, ICAL/ICV and PTs that this does not impact the data results.

19.2. Pace utilizes clean nitrogen for cleaning and filling canisters used for samples and standards. This is a modification from the method use of zero air.

19.3. Pace initial calibrations are accepted with average response models, as well as linear and quadratic regression models. Pace utilizes percent drift when analyzing continuing calibrations with regression models. Acceptance criteria utilized is the same as percent difference (±30% of the midpoint of the most recent ICAL). This process has been adapted from EPA method 8260B. Refer to section 25.8.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1. All maintenance activities are listed in a maintenance logbook.

20.2. Refer to the instrument user's manual for instrument maintenance.

21. TROUBLESHOOTING

21.1. Not applicable to this SOP.

22. SAFETY

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

23.1. Procedures for handling waste generated during this analysis are addressed in ENV-SOP-MIN4-0098, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. POLLUTION PREVENTION

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Department of Defense (DoD) Quality Systems Manual- most current version.

25.5. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO15.

25.6. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO14A MA DEP Air Phase Petroleum Hydrocarbon (APH) method, 12/2009.

25.7. Method 8260B: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). Section 7.4.5.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1. ATTACHMENT I: Target Compound List

26.2. ATTACHMENT II: Required BFB Key Ions and Ion Abundance Criteria

26.3. ATTACHMENT III: Characteristic Ions for Target Compounds

26.4. ATTACHMENT IV: Calibration of THC as Gas

26.5. ATTACHMENT V: Canister Dilution Factors

26.6. ATTACHMENT VI: Air Laboratory Standard Preparation Procedures

26.7. ATTACHMENT VII: Procedures for Analyzing MPCA Samples

26.8. ATTACHMENT VIII: Procedure for Tedlar Bags

26.9. ATTACHMENT IX: Tedlar Bag Transfer Log

26.10. ATTACHMENT X: Common Logbook Abbreviations

26.11. ATTACHMENT XI: Determination of Air Phase Petroleum Hydrocarbons (APH)

26.12. ATTACHMENT XII: Preparation of TO15 SIM standards and ICAL

27. REVISIONS

Document Number	Reason for Change	Date
S-MN-A-013-Rev.22	Section 11.2.7 language updated to be clearer about ICALs per Ohio VAP. Section 11.3.3 language also revisited and added from "Technical justification includes…" to the end of the Section.	1Sept2017
S-MN-A-013-Rev.23	Removed Attachment IX because retired form, subsequent attachment numbers and references adjusted accordingly. Updated reference to new training SOP S-MN-Q-279 in Section 18.4. 1.1 – added last sentence for Ohio VAP "All analysis performed….Method TO-15." 2.1 – added "and preconcentrator system" 2.3 – added "or Select Ion Monitoring (SIM) detection" and changed range lower limit to 0.005 ppbv instead of 0.1. 3.4 – updated language per OH VAP to emphasize notation of bias. Updated 7.1.2 to LLC. Table 9.1 – removed 7100A from Item # of Pre-concentrator 9.1.1 – updated parameters for initial temp, ramp A, ramp B, and Hold 9.1.4.2 – updated Final Temp for Module 2 to 230 instead of 220 9.1.4.3 – updated sample Volume and Flow Rate, updated Transfer Flow Rate 9.1.4.4 – updated line conditioning and sample injection 9.1.4.5 – updated module 2 temperature	04Jan2018

	Table 10.2 – updated description and requirements for Internal Standard row 10.3.1 – added row for Internal Standards/Surrogate/BFB Standard, updated other specifications for every other row, also updated ICAL specs 10.3.2 – updated specs for calibration standard, added "A second pull of 675cc is pulled from the TO15 Stock Standard Cylinder and transferred to the evacuated 15L canister.", updated final concentration 10.3.3 – updated specs for calibration standard, removed "Lastly…15L canister" 10.3.4 – updated specs, removed "Lastly…15L canister" 10.3.5 – updated spec to be from 30-150 ppbv instead of just 50 10.3.6 – added "Using the 1000cc…Standard Preparation Logbook", removed list of analytes and amounts Deleted old sections 10.3.7 and 10.3.8. Current 10.3.7 – updated to p-BFB instead of BFB Current 10.3.8.2 – updated surrogate to just BFB Deleted section 10.4 and all subsections. Table 11.1 ICAL row: in Parameter removed "multiple" from CCV outliers and added "Calibration points may be dropped….from an initial calibration.", in Comments added "For initial calibrations not meeting criteria…. rerun the initial calibration." Table 11.1 ICV row: added "See section 11.2 for corrective actions." To Criteria and Comments columns, removed 7100 from Comments. Table 11.1 CCV row: added "unless an initial calibration is analyzed… analyzed until after the next tune period." To Parameter column. 11.2 – added "Verification (ICV)" 11.2.7 and 11.3.3 – added "For samples being evaluated under the Ohio VAP program, all compounds must meet specifications. Those samples not meeting these criteria must be reanalyzed under compliant conditions." Reworded 11.3.2. 12.1.2 – changed from 500cc to 300cc 12.2.2 – removed 7100A from item # of pre-concentrator 12.7.1.1– edited to "the midpoint of the Initial Calibration" for area response 12.7.2 – added "All reports with nonconformance to the technical criteria must be fully narrated to the client. For work performed under the Ohio VAP program, narration of nonconformance must include an indication of bias in the data." Table 13.1 MB row: added "equal to or" to Acceptance Criteria and added "NOTE: Specific clients may require method blanks be analyzed from canisters that were previously certified as clean and that have not left the laboratory." To Corrective Action Table 13.1 LCS row: added "Failures that produce a high bias with samples that show results as non-detect may be reported for Ohio VAP." To Corrective Action Deleted section 19.4. Added Attachment XII. Attachment III – removed Hexane-d14, toluene-d8, and 1,4-dichlorobenzene-d4 as compounds, added p-bromofluorobenzene. Updated Attachment V. Attachment VI – updated TO15 Standard Prep section, added column for Initial standard concentration to table in Second Source Verification section and updated specs. Completely redid Internal Standard section and table. Attachment XI – updated Second Source Verification to Initial Calibration Verification and updated its parameters, updated parameters for Initial Calibration. Updated SSV to ICV in table, replaced SSV row with ICV.	
ENV-SOP-MIN4-0005-Rev.01	Updated to MasterControl formatting and numbering.	23Oct2018

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ATTACHMENT I - Target Compound List

Compound	CAS RN	TO14 compounds
1,1,1-Trichloroethane	71-55-6	X
1,1,2,2-Tetrachloroethane	79-34-5	X
1,1,2-trichloroethane	79-00-5	
1,1-Dichloroethane	75-34-3	X
1,1-Dichloroethene	75-35-4	X
1,2,4-Trichlorobenzene	95-63-6	X
1,2,4-Trimethylbenzene	95-63-6	X
1,2-Dibromoethane	106-93-4	X
1,2-Dichlorobenzene	95-50-1	X
1,2-Dichloroethane	107-06-2	X
1,2-Dichloropropane	78-87-5	X
1,3,5-Trimethylbenzene	108-67-8	X
1,3-Butadiene	106-99-0	
1,3-Dichlorobenzene	541-73-1	X
1,4-Dichlorobenzene	106-46-7	X
4-Ethyltoluene	622-96-8	
Acetone	67-64-1	
Acrolein	107-02-8	
Acrylonitrile	107-13-1	
Benzene	71-43-2	X
Benzyl Chloride	100-44-7	
Bromodichloromethane	75-27-4	
Bromoform	75-25-2	
Bromomethane	74-83-9	X
Carbon Disulfide	75-15-0	
Carbon Tetrachloride	56-23-5	X
Chlorobenzene	108-90-7	X
Chloroethane	75-00-3	X
Chloroform	67-66-3	X
Chloromethane	74-87-3	X
Cis-1,2-Dichloroethene	156-59-2	X
Cis-1,3-Dichloropropene	10061-01-5	X

ATTACHMENT I (continued) - Target Compound List

Compound	CAS RN	TO14 compounds
Cyclohexane	110-82-7	
Dibromochloromethane	124-48-1	
Dichlorodifluoromethane	75-71-8	X
Dichlorotetrafluoroethane	76-14-2	X
Ethanol	64-17-5	
Ethyl Acetate	141-78-6	
Ethyl Benzene	100-41-4	X
Freon 113	76-13-1	X
Heptane	142-82-5	
Hexachlorobutadiene	87-68-3	X
Hexane	110-54-3	
Isopropyl Alcohol	67-63-0	
M,P Xylene	106-42-3	X
O-Xylene	95-47-6	X
Methyl Butyl Ketone	591-78-6	
Methyl Ethyl Ketone	78-93-3	
Methyl Isobutyl Ketone	108-10-1	
Methyl Tert Butyl Ether	1634-04-4	
Methylene Chloride	75-0902	X
Naphthalene	91-20-3	
Propylene	115-07-1	
Styrene	100-42-5	X
Tetrachloroethene	127-18-4	X
Tetrahydrofuran	109-99-9	
Toluene	108-88-3	X
Trans-1,2-Dichloroethene	156-60-5	
Trans-1,3-Dichloropropene	10061-02-6	X
Trichloroethene	79-01-6	X
Trichlorofluoromethane	75-69-4	X
Vinyl Acetate	108-05-4	
Vinyl Chloride	75-01-4	X

*Current reporting limits can be found in Horizon

Note*: *Any analytes not approved must be clearly indicated on the report with the affidavits as being compounds not certified by Ohio VAP program.

ATTACHMENT II - Required BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (See note)
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

ATTACHMENT III - Characteristic Ions for Target Compounds

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Propylene	41	39	1
Dichlorodifluoromethane	85	87	1
Chloromethane	50	52	1
Dichlorotetrafluoroethane	85	135,87	1
Vinyl Chloride	62	64	1
1,3-Butadiene	54	39	1
Bromomethane	94	96	1
Chloroethane	64	66	1
Ethanol	31	45	1
Trichlorofluoromethane	101	103,105	1
Acetone	43	58	1
Isopropyl Alcohol	45	43	1
1,1-Dichloroethene	61	96	1
Freon 113	101	103,151	1
Methylene Chloride	49	84,86	1
Carbon Disulfide	76	44,78	1
Trans-1,2-Dichloroethene	96	61,98	1
Methyl Tert Butyl Ether	73	41	1
Vinyl Acetate	43	86	1
1,1-Dichloroethane	63	65	1
Methyl Ethyl Ketone	72	43	1
Hexane	57	41,43	1
Cis-1,2-Dichloroethene	96	61,98	1
Ethyl Acetate	43	61,70	1
Chloroform	83	85,47	1
Tetrahydrofuran	42	41,72	1
1,1,1-Trichloroethane	97	99,61	1
1,2-Dichloroethane	62	64	1
Benzene	78	77,50	1
Carbon Tetrachloride	117	119	1
Cyclohexane	56	84,41	1
Heptane	43	41	1
1,2-Dichloropropane	63	41,62	1
Trichloroethene	130	132,95	1

ATTACHMENT III (continued) - Characteristic Ions for Target Compounds

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Bromodichloromethane	83	85	1
Naphthalene	128	127	1
Methyl Isobutyl Ketone	43	58,100	1
Cis-1,3-Dichloropropene	75	39,77	1
Trans-1,3-Dichloropropene	75	39,77	1
Toluene	91	92	1
1,1,2-trichloroethane	97	83,61	1
Methyl Butyl Ketone	43	58	2
Dibromochloromethane	129	127	2
1,2-Dibromoethane	107	109	2
Tetrachloroethene	166	164,131	2
Chlorobenzene	112	77,114	2
Ethyl Benzene	91	106	2
M,P,& O Xylene	91	106	2
Bromoform	173	171	2
Styrene	104	78,103	2
1,1,2,2-Tetrachloroethane	83	85	2
4-Ethyltoluene	105	120,79	2
1,3,5-Trimethylbenzene	105	120	2
1,2,4-Trimethylbenzene	105	120	2
1,3-Dichlorobenzene	146	111,148	2
Benzyl Chloride	91	126	2
1,4-Dichlorobenzene	146	148,111	2
1,2-Dichlorobenzene	146	111,148	2
1,2,4-Trichlorobenzene	180	182,184	2
Hexachlorobutadiene	225	227,223	2
1,4-Difluorobenzene	114	88	IS #1
Chlorobenzene	117	82	IS #2
p-bromofluorobenzene (surr)	95	174,176	2

ATTACHMENT IV - Calibration of THC as Gas

IV-1 THC as gas is calibrated by using the same calibration runs that are used for all other compounds, as well as using the same acceptance criteria.

IV-2 The original calibration files are copied to a target batch. This does not change the raw data in any way, it merely allows the same data to be processed against two different methods

IV-3 The area response is obtained by summing the area in the total ion chromatogram from the first eluting compound of interest till the end of the run. The internal standard is included as part of this value, the response factor is not calculated using the internal standard method. It is solely based on area response and calibration concentration.

IV-4 The calibration concentration at each level is obtained by summing the values of the individual compounds present in the calibration standard.

IV-5 A response factor is obtained as detailed earlier in this SOP. Calibration criteria are the same as stated earlier in this SOP.

IV-6 Custom THC values may be obtained and are noted as such on final reports. These custom values can be based on calibrating using a select list of compounds or a select time frame for example. Requests for these custom values are to be evaluated on an individual basis for analytical feasibility.

ATTACHMENT V - Canister Dilution Factors (6L)

6 Liter Canister		
Initial Pressure	Final Pressure (PSIG)	Dilution Factor
0	5	1.34
-0.5 inHg	5	1.36
-1.0 inHg	5	1.39
-1.5 inHg	5	1.41
-2.0 inHg	5	1.44
-2.5 inHg	5	1.46
-3.0 inHg	5	1.49
-3.5 inHg	5	1.52
-4.0 inHg	5	1.55
-4.5 inHg	5	1.58
-5.0 inHg	5	1.61
-5.5 inHg	5	1.64
-6.0 inHg	5	1.68
-6.5 inHg	5	1.71
-7.0 inHg	5	1.75
-7.5 inHg	5	1.79
-8.0 inHg	5	1.83
-8.5 inHg	5	1.87
-9.0 inHg	5	1.92
-9.5 inHg	5	1.96
-10.0 inHg	5	2.01
-10.5 inHg	5	2.06
-11.0 inHg	5	2.12
-11.5 inHg	5	2.18
-12.0 inHg	5	2.24
-12.5 inHg	5	2.30
-13.0 inHg	5	2.37
-13.5 inHg	5	2.44
-14.0 inHg	5	2.52
-14.5 inHg	5	2.60
-15.0 inHg	5	2.69
-15.5 inHg	5	2.78

6 Liter Canister		
Initial Pressure	Final Pressure (PSIG)	Dilution Factor
-16 inHg	5	2.88
-16.5 inHg	5	2.99
-17.0 inHg	5	3.1
-17.5 inHg	5	3.23
-18.0 inHg	5	3.36
-18.5 inHg	5	3.51
-19.0 inHg	5	3.67
-19.5 inHg	5	3.85
-20.0 inHg	5	4.04
-20.5 inHg	5	4.25
-21.0 inHg	5	4.49
-21.5 inHg	5	4.76
-22.0 inHg	5	5.06
-22.5 inHg	5	5.40
-23.0 inHg	5	5.79
-23.5 inHg	5	6.24
-24.0 inHg	5	6.76
-24.5 inHg	5	7.39
-25.0 inHg	5	8.14
-25.5 inHg	5	9.06
-26.0 inHg	5	10.21
-26.5 inHg	5	11.70
-27.0 inHg	5	13.69
-27.5 inHg	5	16.51
-28.0 inHg	5	20.79
-28.5 inHg	5	28.06
-29.0 inHg	5	43.17
-29.5 inHg	5	93.45
0.5 PSIG	5	1.30
1.0 PSIG	5	1.26
1.5 PSIG	5	1.22
2.0 PSIG	5	1.18

Canister Dilution Equation:

DF = (Pf + 14.7) / Pi ÷ 14.7)
Pi = Pressure reading of canister prior to pressurization (psig)
Pf = Pressure reading of canister after pressurization (psig)
DF = Dilution factor

To convert Hg to psig:
Divide by 2.036

PSIG reading is converted to One Atmosphere:
One Atmosphere = 14.7 psig = 29.21 inches of Hg

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

1 Liter Canister		
Initial Pressure	Final Pressure (PSIG)	Dilution Factor
0	10	1.68
-0.5 inHg	10	1.71
-1.0 inHg	10	1.74
-1.5 inHg	10	1.77
-2.0 inHg	10	1.80
-2.5 inHg	10	1.83
-3.0 inHg	10	1.87
-3.5 inHg	10	1.90
-4.0 inHg	10	1.94
-4.5 inHg	10	1.98
-5.0 inHg	10	2.02
-5.5 inHg	10	2.06
-6.0 inHg	10	2.10
-6.5 inHg	10	2.15
-7.0 inHg	10	2.19
-7.5 inHg	10	2.24
-8.0 inHg	10	2.29
-8.5 inHg	10	2.35
-9.0 inHg	10	2.40
-9.5 inHg	10	2.46
-10.0 inHg	10	2.52
-10.5 inHg	10	2.59
-11.0 inHg	10	2.66
-11.5 inHg	10	2.73
-12.0 inHg	10	2.80
-12.5 inHg	10	2.89
-13.0 inHg	10	2.97
-13.5 inHg	10	3.06
-14.0 inHg	10	3.16
-14.5 inHg	10	3.26
-15.0 inHg	10	3.37
-15.5 inHg	10	3.49

1 Liter Canister		
Initial Pressure	Final Pressure (PSIG)	Dilution Factor
-16 inHg	10	3.61
-16.5 inHg	10	3.74
-17.0 inHg	10	3.89
-17.5 inHg	10	4.05
-18.0 inHg	10	4.22
-18.5 inHg	10	4.40
-19.0 inHg	10	4.60
-19.5 inHg	10	4.82
-20.0 inHg	10	5.06
-20.5 inHg	10	5.33
-21.0 inHg	10	5.63
-21.5 inHg	10	5.97
-22.0 inHg	10	6.34
-22.5 inHg	10	6.77
-23.0 inHg	10	7.26
-23.5 inHg	10	7.82
-24.0 inHg	10	8.48
-24.5 inHg	10	9.26
-25.0 inHg	10	10.20
-25.5 inHg	10	11.35
-26.0 inHg	10	12.80
-26.5 inHg	10	14.66
-27.0 inHg	10	17.17
-27.5 inHg	10	20.07
-28.0 inHg	10	26.07
-28.5 inHg	10	35.19
-29.0 inHg	10	54.12
-29.5 inHg	10	117.17
0.5 PSIG	10	1.63
1.0 PSIG	10	1.57
1.5 PSIG	10	1.52
2.0 PSIG	10	1.48

ATTACHMENT V (continued) - Canister Dilution Factors

AIR CANISTER DILUTIONS

When a sample is over the linear range of calibration for a compound of interest, several compounds of interest, or the matrix of the sample interferes with internal standard detections, a dilution is performed.

SYSTEM DILUTION
The pre-concentrator uses a digital mass flow controller to pull volume of the air sample onto the system.

1x = 300cc
2x = 150cc
5x = 60cc
10x = 30cc
20x = 15cc
30x = 10cc

SERIAL DILUTION
For samples that may require a dilution greater than 30x, the lab performs serial dilutions by emptying the pressurized air in the sample back to ambient conditions (0psig) and refilling the can to 15psig.
This doubles the volume once inside the can and is a 2x

As you multiply this process, the resultant dilution factor is multiplied out.

1. Flush to 0psig fill to 15 = **2x**
2. Flush to 0 and fill again to 15 = **4x**
3. **8x**
4. **16x**
5. **32x**
6. **64x**
7. **128x**
8. **256x**

ATTACHMENT VI - Air Laboratory Standard Preparation Procedures

CALIBRATION STANDARD

The calibration stock standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, Inc, or equivalent. This is a custom mix that includes all compounds of interest at 1ppmv.

TO15 Standard Preparation

Standards are prepared in a 6L or 15L summa canister that has been evacuated to less than 150 mTorr. The canister is humidified with 50 µl of deionized water. A 1000cc gas tight syringe is filled with a desired volume of TO15 stock standard. The summa canister is then pressurized to 30 psig (3 atm) with clean nitrogen from Praxair, resulting in a 30.0 ppbv standard. An aliquot of the 30.0 ppbv standard is withdrawn with a 1000cc gas tight syringe, and transferred to a second 6L or 15L summa canister that has been evacuated to less than 150 mTorr and humidified with 50 µl of deionized water. This second canister is then pressurized to 30 psig (3 atm) with clean nitrogen, resulting in a 1.00 ppbv standard.

The standard ID, date created, analyst initials, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure in psig, final concentration in ppbv and expiration date are recorded in the standard preparation logbook.

Second Source Verification

The second source stock standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas, or equivalent). This includes all compounds of interest at 1ppmv.

The second source standard is prepared in a 6L or 15L summa canister following the same method as the TO15 30.0 ppbv standard.

Canister Volume (L)	Canister Final Pressure (psig)	Canister Final Pressure (atm)	Canister Pressurized Volume (L)	Initial standard concentration (ppmv)	Standard Volume (cc)	Standard Volume (L)	Final Standard Concentration (ppmv)	Final Standard Concentration (ppbv)
6	30	3.04	18.24	1.00	540	0.540	0.030	30.0
15	30	3.04	45.6	1.00	1350	1.35	0.030	30.0
6	30	3.04	18.24	0.030	600	0.600	0.001	1.00
15	30	3.04	45.6	0.030	1500	1.50	0.001	1.00

*The Pressurized canister volume can be obtained from Boyle's Law, stating $P_1V_1=P_2V_2$. At 3.04 atm, a 15L cylinder occupies the same volume as a 45.6L cylinder at 1.00 atm. 1350 cc of a standard is put into the pressurized canister creating a 1.35L/45.6 L dilution factor to result in the standard to determine the final concentration in ppbv.

Internal Standard/ Surrogate/ BFB Standard 150ppbv:

Standards are prepared in a15L summa canister that has been evacuated to less than 150 mTorr. The canister is humidified with 50 µl of deionized water. A 1000cc gas tight syringe is filled with a desired volume of Internal Standard/Surrogate/BFB standard. The summa canister is then pressurized to 30 psig (3 atm) with clean nitrogen from Praxair, resulting in a 150. ppbv standard.

The standard ID, date created, analyst initials, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure in psig, final concentration in ppbv and expiration date are recorded in the standard preparation logbook.

Canister Volume (L)	Canister Final Pressure (psig)	Canister Final Pressure (atm)	Canister Pressurized Volume (L)	Initial standard concentration (ppmv)	Standard Volume (cc)	Standard Volume (L)	Final Standard Concentration (ppmv)	Final Standard Concentration (ppbv)
15	30	3.04	45.6	10.0	675	0.675	0.150	150.

ATTACHMENT VII - Procedures for Analyzing MPCA Samples

VII-1 Samples must be carefully monitored for carryover from previous samples with large detections. Analysts and data reviewers need to verify that each analysis has been evaluated for potential carryover.

- If a compound of interest has an on-column concentration that is greater than 10% of the previous sample, it is assumed that this value is not due to carryover.
- If the compound of interest has an on-column concentration between 2-10% of the previous sample, then the analyst carefully examines other factors relating to sample analysis (i.e. the concentration of related components, the overall concentration of constituents in each sample, etc.). When in doubt, the analyst must re-analyze the sample to confirm that the results are not due to carryover.
- When the compound of interest has an on-column concentration which is less than 2% of the previous sample's concentration, but greater than the method reporting limit, the sample must be analyzed to confirm or eliminate possible carryover.

VII-2 Sample duplicate analysis must be performed at a minimum of 1 in 10 samples analyzed.

VII-3 The relative detection limit for MPCA samples is 0.200 ppbv for all analytes except m&p xylene which has a relative detection limit of 0.400 ppbv.

ATTACHMENT VIII - Procedure for Tedlar Bags

Transfer of Tedlar Bags to SUMMA Canisters

In the event that a sample is collected into a tedlar bag, the client has two days to get the bag to the facility for analytical testing. Pace Analytical Services recognizes a two day holding time for all samples collected in tedlar bags. Upon receipt at the laboratory, the sample in the tedlar bag is transferred into a batch certified, evacuated one liter SUMMA canister for analysis. The sample is subsequently analyzed by the appropriate method within 30 days of transfer.

Procedure for transfer:

- Tedlar bag is received and logged for analysis by Pace Analytical Services
- The sample is delivered to the Air Lab, and the laboratory numbers assigned to the sample is recorded in a logbook (as delivered; see Attachment IX).
- The bag is connected to a clean, evacuated canister (105mTorr).
    - The tip of the bag valve is placed into tubing, connected by a ¼" nut to the sample valve of the canister, secured with a wrench to insure all sample is pulled into the can.
- The bag is opened first. Second, the can is opened.
    - By opening the canister second, the sample is transferred into the can through vacuum (since the can is evacuated to 150mTorr, and the bag is at ambient room pressure).
- After the sample is transferred the sample data and canister number, time and date, is recorded into the transfer logbook (Attachment IX).
- Sample is submitted to the laboratory for analysis.
- A data qualifier is added to the report, notifying the client of the transfer.

ATTACHMENT IX - Tedlar Bag Transfer Log (example)

Pace Analytical

Tedlar Bag Transfer Log

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Sample ID	Can ID	Date Collected	Date when Tedlar Bag was evacuated to the can	Comments

O107 Rev.01 (20Nov2006)
Pace Analytical Services, Inc. – MN

Reviewed by:______________________
Date:______________________

ATTACHMENT X – Common Logbook Abbreviations

RR	**Reanalysis for previously analyzed sample**
DIL	**Dilution for over-range compounds from a previously reported sample**
CONF	**Confirms results from a previously analyzed sample**
C/O	**Possible carryover from a prior sample**
OK	**Analysis is acceptable and sample is reported**

ATTACHMENT XI – Determination of Air Phase Petroleum Hydrocarbons (APH)

This method is designed, based on the Massachusetts APH method, to measure the gaseous-phase concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in air and soil gas. Volatile aliphatic hydrocarbons are collectively quantitated within two carbon number ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantitated within the C_9 to C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 28°C and 245°C. This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance.

Collective Aliphatic/Aromatic ranges: Relative Response Factors are calculated for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons based upon a correlation between the TOTAL mass of aliphatic APH Component Standards eluting within the range of interest and the total ion area count. A Relative Response Factor is calculated for C_9-C_{10} Aromatic Hydrocarbons based upon a correlation between the TOTAL mass of aromatic APH Component Standards eluting within this range and the total area count of extracted ions 120 and 134. Specified APH Component Standards are designated "marker" compounds to define the beginning and end of the hydrocarbon ranges.

- **C_5 through C_8 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute from isopentane to just before n-nonane (C_9).
- **C_9 through C_{12} Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute from n-nonane to just after 1-methylnaphthalene.
- **C_9 through C_{10} Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds which elute from just after o-xylene to just after 1-methylnaphthalene, excluding naphthalene and 2-methylnaphthalene, which are quantitated and evaluated separately as Target APH Analytes.

Hydrocarbon Range	Beginning Marker	Ending Marker
C_5-C_8 Aliphatic Hydrocarbons	0.1 min. before isopentane	0.01 min. before n-Nonane
C_9-C_{12} Aliphatic Hydrocarbons	0.01 min. before n-Nonane	0.1 min. after 1-Methylnaphthalene
C_9-C_{10} Aromatic Hydrocarbons	0.1 min. after o-xylene	0.1 min. after 1-Methylnaphthalene

Standard Information: All APH standards are purchased as 30 component mixtures from a known vendor, such as SPEX CertiPrep or O2Si, in methanol.

Initial Calibration (*Suggested Parameters*)

- Standard Concentration: Components range from 90-260 mg/L
- Prepare a **12 ppbv** working standard by adding 5.4 µl to a clean, evacuated 6L canister which has been humidified with 50 µl of water. Fill to 30psig with nitrogen.

Initial Calibration Verification

- Standard Concentration: Components range from 90-260 mg/L
- Prepare working standard by adding 5.4 µl to a clean, evacuated 6L canister which has been humidified with 50 µl of water. Fill to 30psig with nitrogen.

ATTACHMENT XI (continued) – Determination of Air Phase Petroleum Hydrocarbons (APH)

Initial Calibration and ICV Table:

	Volume (cc)	C5-C8	C9-C12	C9-C10
ICAL-1	10	5.2	6.4	2.4
ICAL-2	25	13	16	6
ICAL-3	50	26	32	12
ICAL-4	125	65	80	30
ICAL-5	250	130	160	60
ICAL-6	500	260	320	120
ICV	125	65	80	30

all expressed in ppbv

Component Mixture		Ions	
Compound	**CAS NO**	**Quant**	**Qual.**
1,3-Butadiene	106990	54	39
Isopentane	78784	43	42
MTBE	1634044	73	41
n-Hexane	110543	57	41/43
Benzene	71432	78	77/50
Cyclohexane	110827	56	84/41
2,3-Dimethylpentane	565593	56	43
n-Heptane	142825	43	41
Toluene	108883	91	92
n-Octane	111659	43	85/57
Ethylbenzene	100414	91	106
2,3-Dimethylheptane	3074713	43	84/85
m-Xylene	108383	91	106
p-Xylene	106423	91	106
o-Xylene	95476	91	106
n-Nonane	111842	43	57
Isopropylbenzene	98828	105	120
1-Methyl-3-ethylbenzene	620144	105	120
1,3,5-Trimethylbenzene	108678	105	120
n-Decane	124185	57	85
1,2,3-Trimethylbenzene	526738	105	120
p-Isopropyltoluene	99876	119	105
Indene	95136	115	116
Butylcyclohexane	1678939	83	55
n-Undecane	1120214	57	42
Naphthalene	91203	128	127
n-Dodecane	112403	57	43
Hexylcyclohexane	4292755	83	82
2-Methylnaphthalene	91576	142	141
1-Methylnaphthtalene	90120	142	141

ATTACHMENT XII – Preparation of TO15 SIM standards and ICAL

CALIBRATION STANDARD

The calibration stock standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, Inc, or equivalent. This is a custom mix that includes all compounds of interest at 1ppmv.

TO15 Standard Preparation - SIM

TO15 Scan standards are prepared in a 6L or 15L summa canister that has been evacuated to less than 150 mTorr. The canister is humidified with 50 µl of deionized water. A 1000cc gas tight syringe is filled with a desired volume of TO15 stock standard. The summa canister is then pressurized to 30 psig (3 atm) with clean nitrogen from Praxair, resulting in a 30.0 ppbv standard.

A 45cc aliquot of the 30.0 ppbv standard is withdrawn with a 60cc gas tight syringe, and transferred to a second 6L summa canister that has been evacuated to less than 150 mTorr and humidified with 50 µl of deionized water. A second 45cc aliquot of the 30.0 ppbv standard is transferred for a total of 90cc. This second canister is then pressurized to 30 psig (3 atm) with clean nitrogen, resulting in a 0.15 ppbv standard. This 0.15 ppbv standard is the SIM primary standard.

The standard ID, date created, analyst initials, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure in psig, final concentration in ppbv and expiration date are recorded in the standard preparation logbook.

Initial Calibration Verification (ICV) - SIM

The second source stock standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas, or equivalent). This includes all compounds of interest at 1ppmv.

The TO15 Scan second source standard is prepared in a 6L or 15L summa canister following the same method as the TO15 30.0 ppbv standard.

Next, a SIM secondary standard is prepared into a second 6L summa canister in the same manner as the SIM primary standard. The resulting 0.15 ppbv standard is the SIM secondary source standard.

Initial Calibration and ICV Table:

	Volume (cc)	Calibration Standard Used	Concentration
ICAL-1	10	0.15	0.005
ICAL-2	20	0.15	0.01
ICAL-3	40	0.15	0.02
ICAL-4	100	0.15	0.05
ICAL-5	200	0.15	0.1
ICAL-6	400	0.15	0.2
ICAL-7	600	0.15	0.3
ICV	200	0.15	0.1

all expressed in ppbv

Continuing Calibration Verification (CCV) – SIM

A CCV standard is analyzed at 0.100 ppbv (ICal level 5). The CCV is the same source as the ICAL standard.

Acceptance Criteria

The analysis of analytes for TO15 SIM has the same acceptance criteria for TUNE, ICAL, ICV, and CCV as specified for TO15 Scan in section 11, Calibration and Standardization. TO15 SIM has the same acceptance criteria for MB, LCS, DUP, IS, and Surrogates as specified for TO15 Scan in section 13, Quality Control.

ATTACHMENT XII (continued) – Preparation of TO15 SIM standards and ICAL

TO-15 SIM Analytes		
Compound	Quant ion	Qualifier ion
Vinyl chloride	62	64
1,3-butadiene	54	39,43
1,1-dichloroethene	61	96
Trans-1,2-dichloroethene	96	61
1,1-dichloroethane	63	65
Cis-1,2-dichloroethene	96	61
chloroform	83	85,47
1,1,1-trichloroethane	97	99
1,2-dichloroethane	62	64
benzene	78	77
Carbon tetrachloride	117	119
1,2-dichloropropane	63	41,62
trichloroethene	130	132
bromodichloromethane	83	85
Cis-1,3-dichloropropene	75	39,77
Trans-1,3-dichloropropene	75	39,77
1,1,2-trichloroethane	83	97
toluene	91	92
1,2-dibromoethane	107	109
tetrachloroethene	166	164
ethylbenzene	91	106
m&p-xylene	91	106
o-xylene	91	106
1,1,2,2-tetrachloroethane	83	85
naphthalene	128	127

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STANDARD OPERATING PROCEDURE

EXTRACTION AND ANALYSIS OF POLYCHLORINATED BIPHENYLS IN OIL, SOIL, WATER, WIPE MATRICES

Reference Methods: SW846 Method 8082 and 8082A

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APPROVALS

Laboratory General Manager — Date: 16 May 2018

Laboratory Quality Manager — Date: 16 MAY 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to provide instruction on the analysis of Polychlorinated Biphenyls (PCBs) by EPA SW846 Methods 8082 and 8082A.

2. Summary of Method

2.1. PCB samples are received from the Prep Lab. The instrument is calibrated and the samples are then run. The data is checked, reported (soils are corrected for moisture unless otherwise requested) and filed.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268.

4. Applicable Matrices

4.1. This SOP is applicable to oil, soil, water and wipe matrices.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 0.10 ug/L for waters, 0.033 mg/Kg for soils, 5.0 mg/Kg for oils and 1.0 total ug for wipes this method. All current method detection limits (MDLs) are listed in the Laboratory Information Management System (LIMS) and are available by request from the Quality Manager.

6. Interferences

6.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

6.2. Interferences by phthalate esters pose a major problem in PCB analysis when using an ECD. These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates, which are easily extracted or leached during the laboratory operation. Avoiding the use of plastics can minimize interferences from phthalates. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. Refer to Pace Analytical Services, LLC SOP on Glassware Cleaning, S-MN-O-465, or equivalent replacement.

6.3. Interferences co-extracted from the samples will vary considerably from source to source. Acid cleanup is performed on all oils, soils, and wipe samples. Acid cleanup on water extracts is performed upon analyst's discretion. Cleanup procedures may be used to remove such interferences. Refer to the appropriate cleanup SOPs if extract cleanup to remove interferences is required.

6.4. Spiked laboratory replicates should be analyzed to validate the precision and accuracy of the analyses.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Sample Collection, Preservation and Holding time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	1L Amber glass jar	Unpreserved	<6°C but above freezing	Must be extracted within 1 year from time of collection and analyzed within 40 days of extraction.

Solid (including soil, oil and wipe)	4 oz glass jar with Teflon lined lid	Unpreserved	<6°C but above freezing	Must be extracted within 1 year from time of collection and analyzed within 40 days of extraction
Caulk	4 oz glass jar with Teflon lined lid	Unpreserved	No thermal preservation required.	SW846 Chapter 4 does not specify a holding time for caulk. Pace utilizes the soil requirements. Will be extracted within 1 year from time of collection and analyzed within 40 days of extraction

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies.

Supply	Description	Vendor/ Item # / Description
Separatory funnel	2L with Teflon® stopcock	Fisher Scientific or equivalent
Teflon® cap		Fisher Scientific or equivalent
Graduated cylinder	100 mL and 1000 mL	Fisher Scientific or equivalent
Erlenmeyer flasks	500 mL with glass stopper or aluminum foil	Fisher Scientific or equivalent
Aluminum foil		Fisher Scientific or equivalent
Ring Stand		Fisher Scientific or equivalent
pH strip	Wide range	Fisher Scientific or equivalent
Autosampler vials	2mL with crimp top caps	Fisher Scientific or equivalent
Balance	Analytical, capable of accurately weighing 0.00 gm. - top loading	Denver MXX-612, A&D EK-410i or equivalent replacements
Sonicator	Ultrasonic cell disrupter, heat systems	Ultrasonics, Inc. Model W-385, Model 3000 or model 2020 Sonicator with #207 3/4 inch disrupter horn or equivalent
Microwave	CEM MarsXpress SCP Novawave	Model 907501 or equivalent Or equivalent
Microwave vessels	CEM MarsXpress/SCP Novawave	Teflon/glass or equivalent
Capping Station	CEM/SCP	Capping Station or equivalent
Beakers	400 mL	Fisher Scientific or equivalent
Tilting dispenser/ pump dispenser	For dispensing solvent	Fisher Scientific or equivalent
Wooden stir sticks	Tongue depressors	Fisher Scientific or equivalent
Syringes	1 mL microsyringe	Hamilton, or equivalent
Collection flasks		Fisher Scientific or equivalent
Powder funnel		Fisher Scientific or equivalent
Glass wool		Fisher Scientific or equivalent
Glass pipettes	Disposable, calibrated per lot	Fisher Scientific or equivalent
Pastuer pipettes	Disposable	Fisher Scientific or equivalent
Soxhlet		Fisher Scientific or equivalent
Round bottom flask	500 mL	Fisher Scientific or equivalent

Boiling Chips		Fisher Scientific or equivalent
Kuderna-Danish (KD) concentrator tubes	10 mL graduated with ground glass stoppers	Fisher Scientific or equivalent
KD Snyder column	Three ball macro	Fisher Scientific or equivalent
KD Evaporation flask	500 mL attach to concentrator tubes with connectors	Fisher Scientific or equivalent
Water bath	Heated with concentric ring cover, capable of temperature control (± 2°C). The bath should be used in a hood	Fisher Scientific or equivalent
Evaporation device	Nitrogen evaporation device with water bath N-Evap	Fisher Scientific or equivalent
Gas Chromatograph (GC)	Agilent 5890 GC or Agilent 6890 GC with dual electron capture detectors and a Agilent 7376 autosampler (or equivalent	Agilent, or equivalent
GC Column	Two wide-bore (30 m x 0.32 mm ID) fused silica GC columns are required. A separate detector is required for each column. The recommended analytical columns are a DB-35MS or RTX-CLP, 30 m x 0.32 mm ID, 0.25 um film thickness (J&W Scientific or equivalent) and a DB-XLB or RTX-CLP2, 30 m x 0.32 mm ID, .25 um film thickness, (J&W Scientific, Folsum, CA or equivalent	J&W Scientific, or equivalent
Injection port	Columns are mounted in a dual GC/ECD with a single injection port/guard column connected to a glass Y	Agilent, or equivalent
Electon Capture Detector (ECD)	The make-up gas must be P-5, P-10 (argon/methane) or nitrogen, according to the instrument specification. The linearity and the response of the ECD may be greatly dependent on the flow rate of the make-up gas to the detector	Agilent, or equivalent
Data Processing Software	See master list for most current version	Target
Data Acquisition Software	See master list for most current version	Chemstation
Data Reporting Software	See master list for most current version	Horizon
Data Review Software	See master list for most current version	Gandalf

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards.

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
Sulfuric Acid (concentrated)	Trace metal grade or equivalent	Fisher / Catalog # A510SK-212
Sodium Sulfate	Prepared by baking at 400° C for 4 hours	Fisher / Catalog # S415-200LB
Baked Sand	Prepared by baking at 400° C for 4 hours	Menards
Methylene Chloride	Pesticide Grade or equivalent	Fisher / Catalog # D151-4
Acetone	Pesticide Grade or equivalent	Fisher / Catalog # A929-4
Hexane	Pesticide Grade or equivalent	Fisher / Catalog # H303-4

Ical Stock Solution Standards	1000 µg/mL 1016/1260 Mix	Restek Catalog # 32039
Surrogate Stock Solution	Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB	Supelco Catalog # 4-8460
Aroclor Stock Standards	Aroclor 1221 200 ug/mL Aroclor 1232 100 ug/mL Aroclor 1242 1000 ug/mL Aroclor 1248 200 ug/mL Aroclor 1254 200 ug/mL Aroclor 1262 1000 ug/mL Aroclor 1268 100 ug/mL	1221-Supelco Catalog # 4-8705 1232-Accustandard Catalog #App-9-160 1242-Chem Service Catalog #F109AJS 1248-Supelco Catalog #4-8703 1254-Supelco Catalog #4-8707 1262-Supelco Catalog #4-4810 1268-Accustandard Catalog #App-9-166
Initial Calibration Verification stock standards	Aroclor 1016 1000 ug/mL Aroclor 1260 1000ug/mL	1016-Accustandard Cat.#App-9-158-10x 1260-Accustandard Cat.#App-9-164-10x

10.2. **Working Standard Dilutions and Concentrations**

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Ical stock solution	0.5 mL of 1016/1260 (stock solution) 0.25 mL pest SS mix	Hexane	24.25 mL	25.0 mL	1016/1260 =20 ug/mL Surr,=2.0 ug/mL
ICV Stock solution	0.1 mL 1016 0.1 mL 1260 0.05 mL Pest SS mix	Hexane	4.75 mL	5.0 mL	1016/1260=20 ug/mL Surr.=2.0 ug/mL
Calibration Std 1	0.025 mL of Ical stock	Hexane	4.975 mL	5.00 mL	0.1 µg/mL Aroclor; 0.01 µg/mL Surr.
Calibration Std 2	0.125 mL of Ical stock	Hexane	4.875 mL	5.00 mL	0.5 µg/mL Aroclor; 0.05 µg/mL Surr.
Calibration Std 3	0.250 mL of Ical stock	Hexane	4.75 mL	5.00 mL	1.0 µg/mL Aroclor; 0.10 µg/mL Surr
Calibration Std 4	0.500 mL of Ical stock	Hexane	4.50 mL	5.00 mL	2.0 µg/mL Aroclor; 0.20 µg/mL Surr.
Calibration Std 5	0.750 mL of Ical stock	Hexane	4.25 mL	5.00 mL	3.0 µg/mL Aroclor; 0.30 µg/mL Surr.
Calibration Std 6	1.00 mL of Ical stock	Hexane	4.00 mL	5.00 mL	4.0 µg/mL Aroclor; 0.40 µg/mL Surr.
Individual Aroclors	0.01ml@1000ug/ml 0.05ml@200ug/ml 0.1ml@100 ug/ml	Hexane	4.99 mL 4.95 mL 4.90 mL	5.00 mL	2.0 µg/mL
Initial Calibration Verification Standard (ICV) (Second Source)	0.500 mL of ICV stock	Hexane	4.50 mL	5.00 mL	2.0-4.0 µg/mL
TCMX/DCB Surrogate Working Solution(Pest SS)	1.2 mL	Acetone	198.8 mL	200 mL	1.2 µg/mL
Continuing Calibration Verification Standard	0.500 mL of Ical stock	Hexane	4.50 mL	5.00 mL	2.0 µg/mL Aroclor; 0.20 µg/mL Surr.
Spiking Standard for LCS and MS (PCB MS)	2.0 mL of 1016 2.0 mL of 1260	Acetone	46.00 mL	50.00 mL	40.0 µg/mL

11. Calibration and Standardization

11.1. Calibration (applies to both primary and confirmation columns)

11.2. Initial Calibration: External standard calibration is used for Aroclor analysis.

11.2.1. Calibration is achieved through the analysis of standards containing the target analytes at a minimum of five different concentrations covering the working range of the instrument. The lowest calibration standard must be at or below the required reporting limit. Refer to the Quality Manual for more information on calibration curves.

11.2.2. Six concentrations of Aroclors 1016 and 1260 (AR1660) and surrogates are used for the initial calibration. The Aroclors 1016 and 1260 include many of the peaks represented in the other five Aroclor mixtures, and can be used to demonstrate a curve that spans the whole chromatogram. Concentrations of AR1660 and surrogates used to perform the initial calibration can be found in table 10.2.

11.2.3. Single point standards for identification and quantitation purposes are analyzed for the remaining Aroclors at 2.0 mg/L. These standards are necessary for pattern recognition. The standards for these other Aroclors should be analyzed before the analysis of the AR1660 initial calibration curve.

11.3. Each Aroclor shall be identified by 3-5 peaks to be used for calibration. The peaks selected from each Aroclor are based on peak heights and strong consistent responses on column. The smallest peaks selected for an Aroclor should be at least 25% of the height of the largest peak or as close as the pattern allows. Each Aroclor shall have at least one peak that is unique to that Aroclor. A book of reference chromatograms for each Aroclor has been created for each instrument. Refer to this book for the preferred peaks used for calibration (F-MN-O-346).

11.3.1. Peaks chosen for each Aroclor are ultimately up to the analyst who identifies each Aroclor at the time of calibration. These peaks will remain consistent throughout the life of the calibration and will be used in all initial calibration standards and subsequent calibration verification standards and samples which are associated with that initial calibration.

11.3.2. On a per client basis, Aroclor peaks will be chosen to make sure that they do not overlap with any other Aroclor peaks chosen with the exception of Aroclors 1016 and 1242, and Aroclors 1260 and 1262. Aroclors 1016 and 1242 will never both be chosen in an individual sample due to their closely related makeup, the same is true of 1260 and 1262.

11.4. Calibration factors (CFs) are determined for each peak chosen for quantitation for each particular Aroclor, in each calibration standard analyzed, including surrogates. The response factor for each Aroclor is calculated as

CF = Response of particular Aroclor in standard/concentration of Aroclor in standard).

11.5. Linearity of the initial calibration is determined through the use of the average CF and relative standard deviation (RSD). For each analyte and surrogate analyzed, calculate the average CF, the standard deviation (SD), and the RSD as follows:

$$Average\ CF = CF_{AVG} = \Sigma CF_i / n,$$

$$SD = \sqrt{(\Sigma (CF_i - CF_{AVG})^2 / n-1)}, \text{ and}$$

$$RSD = (SD/CF_{AVG}) \times 100,$$

Where:
n = the number of calibration standards and
CF_i = the CF of each individual standard.

11.5.1. If the RSD of the calibration factors for a given Aroclor or surrogate is less than or equal to 20% over the calibration range, then linearity is assumed, and the average CF may be used to determine sample concentrations for that analyte.

11.5.2. The RSD for each of the five peaks chosen for a particular Aroclor shall be less than or equal to 20%.

11.5.2.1. For Method 8082: If one or more of the RSDs exceeds 20%, the calibration may still be acceptable provided that the average of the five RSDs for that particular Aroclor is less than or equal to 20%.

11.5.2.2. For Method 8082A: The RSD for each of the five peaks chosen for a particular Aroclor shall be less than or equal to 20%.

11.5.3. If the RSD for a particular Aroclor or surrogate exceeds 20%, then corrective action must be taken before any sample analysis may begin if that analyte is to be determined using this procedure. Alternatively, the initial calibration curve may be evaluated using linear regression provided the correlation coefficient (r) is greater than or equal to 0.99.

11.5.4. If the RSD is greater than 20% and if the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression (quadratic) could be attempted.

11.5.4.1. Non-Linear Calibration: When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

11.5.4.2. A minimum of six points must be used for a second-order regression fit.

11.5.4.3. The coefficient of determination must be $r^2 \geq 0.99$. Second-order regressions should be the last option.

11.5.4.4. Before selecting a second-order regression calibration model, it is important to ensure the following:

11.5.4.4.1. The absolute value of the intercept is not large relative to the lowest concentrations being reported.

11.5.4.4.2. The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

11.5.4.4.3. The distribution of concentrations is adequate to characterize the curvature.

11.6. The same calibration model used for the initial calibration must be used for all subsequent analyses of standards and reagents until the next initial calibration. The model utilized cannot be changed after the ICAL has been processed and approved.

11.7. **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.8. **Initial Calibration Verification (ICV):** The initial calibration is verified by the analysis of an Initial Calibration Verification (ICV) standard made from a source different than that used for the initial calibration standards. The ICV is analyzed for each Aroclor 1660 and surrogates. The ICV is analyzed immediately following the curve and recoveries must be within 80-120% of the true value.

11.9. **ICV Corrective Action:** If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Samples associated with the initial calibration and failed ICV must be reanalyzed.

11.10. **Continuing Calibration Verification:** Verify that the initial calibration each 12-hour shift by injecting a Continue Calibration Verification (CCV) standard prior to conducting sample analysis. The time between injection of CCV standards shall not exceed 12 hours regardless of whether twenty samples have been injected or not. The CCV must be analyzed after each group of 20 samples and at the end of the analytical sequence. Calibration verification standards are analyzed under the same conditions as any other standards analyzed by this procedure.

11.10.1. A calibration factor is determined for each analyte and surrogate in the CVS. The percent difference (%D), is calculated as follows:

$$\%\ Difference = \%D = ([Average\ CF] - [CF_{CVS}])/[Average\ CF] * 100,$$

Where:

[Average CF] = The average calibration factor calculated for the initial calibration
$[CF_{CVS}]$ = The calibration factor for the continuing verification standard

11.10.2. If the %D for each analyte and surrogate is less than or equal to ±15% for Method 8082 and ± 20% for Method 8082A, then the initial calibration is considered valid, and the CFs from the initial calibration may still be used to quantitate sample results and analysis may continue.

11.10.2.1. For Method 8082: Should any analytes fail the %D criteria, the initial calibration as a whole for a given Aroclor may still be considered valid if the %D between the average CF for each of the five peaks and the average of the five CFs from the calibration verification does not exceed ±15%.

11.10.2.2. For Method 8082A: Should any analytes fail the %D criteria (± 20%), the initial calibration would no longer be valid for that given Aroclor.

11.11. **CCV Corrective Action:** If a CCV fails the acceptance criteria, check the instrument operating conditions and analyze another CCV. If the CCV response still fails the acceptance criteria, a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. Exception: If the CCV is outside of the upper control limit, indicating a high bias, associated samples determined to be <RL may be reported (see Attachment V).

11.12. Retention time windows are used to identify the target analytes in sample and standard chromatograms. Analyst experience should weigh heavily in the use of retention time windows and the identification of target analytes. See the procedure described in 12.3.1 for setting of retention time windows.

11.13. Table 11.13. Calibration and Standardization.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average	RSD ≤ 20%	If not met, try non-linear regression fit. If still not met, remake standards and recalibrate and verify before sample analysis.
	Linear Regression	r ≥ 0.99	
	Non-linear Regression	COD ≥ 0.99	
Reporting Limit Standard	Low level standard at or below the reporting limit used to define the	±40% of the true value required by MDH;	If the criteria is not met, the instrument must be recalibrated or the reporting limit adjusted to the next level that meets the

	accuracy of limit of quantitation	±20% for Arizona; ±50% for non MN for each analyte set to linear or quadratic and ±30% of the true value for all levels of a linear or quadratic curve	criteria.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ±15% for 8082 and ±20% for 8082A	If the requirements for initial calibration are not met, review standard preparation. Evaluate the instrument for any errors and perform reanalysis. If the second analysis does not meet criteria, recalibrate the instrument prior to sample analysis.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 20 samples thereafter. Samples must be bracketed with a closing CCV standard. Some clients or outside agencies may require running a CCV more frequently	% Diff ±15% for 8082 and ±20% for 8082A	If the requirements for continuing calibration are not met, review standard preparation. Evaluate the instrument for any errors and perform reanalysis. If the second analysis does not meet criteria, recalibrate the instrument prior to sample analysis.

12. Procedure

12.1. Extraction

12.1.1. Refer to the Attachment section for each matrices extraction instruction sheet.

12.1.2. All data is recorded onto extraction sheets See Attachment I as an example.

12.2. Instrument Analysis

12.2.1. Agilent model 5890 or 6890 gas chromatograph is equipped with a dual electron capture detector. The following operating parameters are recommended, but may be adjusted to optimize the analytical run:

Parameter	Setting
Column Flow	0.32 id = 2-3 mL/min 0.53 id= 7-10 mL/min
Split Flow	70-80 mL/min
Make up	70-90 mL/min
Injector Temperature	250°C
Injection	Split/Splitless
Injection Volume	1 uL
Detector Temperature	300°C
Initial Temperature	160°C
Initial Time	0 min
Temperature Ramp	5°C/min to 280°, 30°C to 310°
Final Temperature	310°C
Final Hold Time	4 min

12.2.2. Inject aliquots of all sample extracts and QC into the GC under the same operating conditions as used for the initial calibration. Sample vials are loaded onto the autosampler which is programmed via Chemstation to inject the necessary volume.

12.2.3. Data is reported from the following column: DB-35MS or RTX-CLP: 0.32 micron x 30 meters with 0.5 micron film thickness.

12.2.4. A second column is used for confirmation: DB-XLB or RTX-CLP2: 0.32 micron x 30 meters with a 0.25 micron film thickness. Data generated from the second column is used for confirmation; data is reported from the primary column.

12.3. **Qualitative Analysis**

12.3.1. The surrogate TCMX uses a retention time window ±0.05 min. All aroclors use a retention time window ±0.07 min. The surrogate DCB uses a retention time window of ±0.10 min. RT time windows are set using the initial calibration and are updated daily with daily CCV's.

12.3.2. **Data Processing:** Raw data files from either front or back columns are to be processed using the Target software (or equivalent). Review peak integration and identification, edit if necessary, and generate quantitation reports. Perform the following procedures independently for data files obtained from each of the two analytical columns.

12.3.2.1. Data is processed using the particular processing methods developed for Aroclor analysis. The processing method contains calibration information, integration parameters and expected retention times of analytes to be determined. Integration parameters should be developed that minimize the need for manual integration. It is imperative that samples and standards be processed using the same integration parameters. A separate, yet similar method is required for data from each column.

12.3.2.2. Review the processed data by looking at the Result files generated by the Target Software. Inspect the peak integration to insure that all peaks of interest are properly integrated. Manual integrations may be performed, if necessary. Any manual integration performed on any sample, standard, or QC sample should be made and documented as described in the most recent manual integration SOP, S-ALL-Q-016.

12.3.3. Aroclor identification for Aroclors 1016 and 1260 are based on the six-point initial calibration. Quantitation of the other Aroclors is based on single-point calibration standards, unless further calibration is required by the client or regulation agency.

12.3.4. Identification of an Aroclor is made when then pattern seen in a sample chromatogram is matched with the pattern seen in the initial calibration. A book of reference chromatograms for each Aroclor on each column and each instrument (F-MN-O-346) is available to show the preferred peaks picked and the ratios between peaks that are characteristic of each Aroclor. When interferences are present, weathering/degradation has occurred, or multiple Aroclors are suspected, the tools listed below may help with proper identification.

12.3.4.1. Overlays of the sample chromatogram with chromatograms of Aroclor standards: The Target Software allows overlays of multiple files which can be useful in comparing the ratios between peaks, and the overall pattern of an Aroclor.

12.3.4.2. Comparison of characteristic peak retention times with Aroclor standards: The Target files of each injection show the retention time, expected retention time and the difference on the Target report (target.rp) located in each file. The retention time and expected retention time can also be evaluated in the Target viewer.

12.3.4.3. Comparison of the ratio between characteristic peaks with ratio of Aroclor standards: The ratio between peaks can be seen by pulling up an overlay of the sample and standard in the Target software or comparing with the sample chromatograms in F-MN-O-346.

12.3.4.4. Comparison with historical results, if available: All projects can be traced in the Horizon lims system and the equivalent Target files can be obtained. Consistent weathering/degradation on Aroclors may be seen throughout a project.

12.3.4.5. Analyst judgement and consultation with other experienced analysts: All Aroclor identification comes down to the analyst. When an analyst is questioned on an identification, there should be a clear technical justification behind the decision. An analyst may wish to confer with other analysts to make sense of a sample that has weathering/degradation or other interference.

12.3.5. The result of a single injection analysis must be confirmed on a second, dissimilar, GC column. The second column must demonstrate a sensitivity that is comparative to the first column. When using a dual-column system, target Aroclors must be identified and confirmed by identifying criteria on both columns. This second column confirmation must meet all QC criteria described for the first column including calibration, QC, and retention time criteria. Refer to F-MN-O-282 (Attachment V) to find the appropriate corrective action when either column is not meeting analytical criteria.

12.4. **Quantitative Analysis**

12.4.1. The quantitation of PCB residues as Aroclors is accomplished by comparing the sample pattern with known Aroclor standards and selecting the most similar Aroclor. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

12.4.2. Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the calibration standard for that Aroclor with the peaks observed in the sample extract. The Aroclor amount is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen and the calibration model established from the multi-point calibration of AR1660. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor. Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria.

12.4.3. When interferences are present or degradation has occurred, peaks yielding concentrations that are dissimilar to others may be excluded. When multiple Aroclors are suspected, quantitation of these Aroclors should be based on the best match with established Aroclor patterns as determined using the tools outlined in Section 12.3.4.

12.4.4. If the concentration of an analyte in a sample is less than the concentration of the laboratory generated reporting limit for that analyte, then the result for that analyte is reported as less than reporting limit or non-detect. Data for that analyte in the sample from the second column need not be evaluated. If the concentration of analyte determined exceeds the reporting limit for that analyte, then the data from the confirmation column must be evaluated for that analyte in that sample to confirm the presence of that analyte in the sample.

12.4.5. The ECD response for all analytes must be within the established calibration range in order for quantitative measurements to be made. Dilute and reanalyze the sample extracts

if the response of a target analyte in a sample extract exceeds the limits of the initial calibration range.

12.4.6. If the analyte in question is also detected in the sample on the second column, compare the concentration determined for the analyte on this column to the reporting limit for the analyte. If the concentration exceeds the reporting limit, then the presence of that analyte in the sample is confirmed. If the relative percent difference between the concentrations determined on both columns exceeds 40% for 8082 report the higher of the two concentrations, unless otherwise specified by client requirements. If the relative percent difference between the concentrations determined on both columns exceeds 40%, for 8082A report the lower of the two concentrations, unless otherwise specified by client requirements. If the relative percent difference between the concentrations determined on both columns does not exceed 40% report the data from the primary column.

12.4.6.1. For samples originating from the state of Wisconsin, if the relative percent difference between columns exceeds 40% report the higher of the two concentrations.

12.4.7. Quantitation of Analytes

12.4.7.1. Oil

$$mg/kg = \frac{Cx\ (\mu g/mL) \times Vt\ (mLs) \times 1000\ grams \times 1\ mg \times DF}{V\ (grams) \times 1\ kg \times 1000\ \mu g}$$

*Where Cx= Concentration of the peak for the compound to be measured in the sample extract (µg/mL)

Vt = Final volume of the sample extraction (mLs)

V = Initial weight or volume of the sample (grams or liters)

DF= Dilution factor, if necessary

*Applies to Sections 12.4.7.2 through 12.4.7.4

12.4.7.2. Soil

$$mg/kg = \frac{Cx\ (\mu g/mL) \times Vt\ (mLs) \times 1000\ grams \times 1\ mg \times DF \times [100]}{V\ (grams) \times 1\ kg \times 1000\ \mu g \times [100\text{-}M]}$$

12.4.7.3. Water

$$\mu g/L = \frac{Cx\ (\mu g/mL) \times Vt\ (mLs) \times DF}{V\ (liters)}$$

12.4.7.4. Wipe

$$\mu g = Cx\ (\mu g/mL) \times Vt\ (mLs) \times DF$$

12.4.7.5. The recovery of the surrogates is calculated according to the following equation:

$$\text{Surrogate Percent Recovery} = \frac{Qd}{Qa} \times 100\%$$

Where Qd = Concentration determined by analysis

Qa = Concentration added to the sample/blank

12.4.7.6. The following calculation can be used to calculate the LCS percent recovery (where SampleConc would be equal to 0):

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

13. Quality Control

13.1. Table 13.1 Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water; clean sand, oil or wipe depending on the matrix	One per 20 samples	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect See Attachment V for surrogate criteria.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result > 10x MB detects, report sample as not impacted by the blank contamination; If sample result is < 10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	Reagent water; clean sand, oil or wipe depending on the matrix spiked with all target compounds	One per 20 samples; LCS duplicate is performed if there is insufficient sample volume for matrix spike samples	Internally generated limits generated on an annual basis. If an LCSD is performed, relative percent difference (RPD) is < 20% See Attachment V for surrogate criteria.	Reanalyze the LCS to confirm results. If problem persists, check spike solution. Perform system maintenance prior to new LCS run if it was suspected that the instrument was the cause of failure. Re-extract the entire batch if the recoveries are biased low. If insufficient sample volume is available for reextraction, report the data with appropriate qualifiers indicating the biased. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. If the LCSD is within criteria, but the RPD fails treat like a biased high LCS recovery.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 20 samples	Internally generated limits generated on an annual basis. See Attachment V for surrogate criteria.	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences The sample should be investigated for matrix interference and narrated appropriately. MPCA requires full narrative and investigation prior to qualifying the data.

MSD / Duplicate	MS Duplicate *OR* *(alternative)* Sample Dup	One per 20 samples.	%RPD ≤ 30% See Attachment V for surrogate criteria.	Report results with an appropriate footnote. For Minnesota Admin Contract Clients - all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.

14. Data Analysis and Calculations

14.1. See Section 12.4.7.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-of-Control Data

16.1. See table in section 11 & 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 or equivalent replacement, Method Detection Limit Studies for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279, Training Employee and Orientation, or equivalent replacement.

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Results are stored in the QA office.

19. Method Modifications

19.1. TSCA Modifications

19.1.1. Multi-Phasic Samples - Multi-phasic samples will be separated into individual phases prior to PCB extraction and analysis. This will be accomplished only after contacting the client and discussing all options. A complete separation must be accomplished by the use of a centrifuge between oil and water.

19.1.2. The holding times for TSCA soil, oil and wipes samples will not be applied. The data will then be reported as SW8082 (modified/TSCA) or equivalent. Since the applicability of these holding times, which are a RCRA requirement, will not affect the usability of the data for TSCA samples, they will be disregarded.

19.1.3. All TSCA samples will be reported on a dry weight basis.

19.1.4. All the Aroclors including PCB 1262 and PCB1268 will be monitored. Prior to analysis standards of these PCBs will be analyzed and MDLs will be obtained

19.1.5. Wipe samples will be extracted utilizing 25-50 mL of hexane. The extracted will be filtered and concentrated to a final volume, which is dependent on the action level requested by the client. All surrogate and matrix spikes should be adjusted to ensure the final concentration will be at the midpoint of the analytical curve.

19.1.6. The 6th point in the calibration curve (10µg/mL) will not be used to quantitate the data.

19.1.7. Oil – Initial volumes may be increased depending on action level requested by the client. All surrogate and matrix spikes should be adjusted to ensure the final concentration will be at the midpoint of the analytical curve.

19.1.8. The following table represents the extraction amounts that should be utilized to achieve a specific action level. If the action level has not been specified on the chain of custody, the client must be contacted and this level noted. If an action level is not communicated, the extraction procedure for an action level of 1 ppm must be utilized with a full understanding by the client that the surrogates may be diluted out.

Action Level	Extract Amount	Final Volume	Surr conc|amt spiked	QC conc|amt spiked
1 ppm	10g	5 mL	2 ug/mL| 0.2mL	50 ug/mL | 0.2 mL
10 ppm	10g	50 mL	20 ug/mL| 0.2 mL	500 ug/mL | 0.2 mL
25 ppm	10g	125 mL	20 ug/mL| 0.5 mL	500 ug/mL| 0.5 mL
50 ppm	10g	250 mL	20 ug/mL | 1.0 mL	500 ug/mL | 1.0 mL

*** The final volume will be a combination of dilutions. The acid cleanup will be performed on an aliquot from the final dilution prior to finalization.**

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument manual for maintenance procedures performed by the lab. Additional information can be found in the Instrument Maintenance SOP S-MN-L-114, or equivalent replacement.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3. If calibrations fail to meet criteria, perform the following suggested maintenance or as suggested in Instrument Maintenance SOP S-MN-L-114, ore equivalent replacement.

20.3.1. Cleaning or replacing the gold seal.

20.3.2. Cleaning or replacing the liner.

20.3.3. Replacing the glass wool in the liner.

20.3.4. Replace the septa.

20.3.5. Cleaning split lines and weldment nut.

20.3.6. Clip a small amount of column or replace the column.

21. Troubleshooting

21.1. If sample emulsions appear during extraction after shaking, drain the emulsion into a clean 250mL vial and put in centrifuge. Then pour solvent from 250mL vial back into the separatory funnel and drain through the funnel.

21.2. Baking your instrument oven at 300°C after an analytical run or prior to running the daily ccv may be helpful to reduce contaminants in your analytical system. Regular cleaning of the injection port, liner, gold seal and the occasional split line cleaning will be needed in order for the analytical system to pass CCV's.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. USEPA Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Method 8082, Revision 0, December 1996.

25.5. USEPA Test Methods for Evaluating Solid Waste, SW-846, Online Edition, Method 8082A, Revision 1, February 2007.

25.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition and Update I, Method 3550B, "Ultrasonic Extraction" Revision 2, December 1996.

25.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510 "Separatory Funnel Liquid-Liquid Extraction", 1986.

25.8. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, Online Edition, Method 3541 "Automated Soxhlet Extraction" Revision 0, September 1994.

25.9. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3540 "Soxhlet Extraction", 1986.

25.10. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3580 "Waste Dilution", 1986.

25.11. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update V, Method 8000C, Revision 3, March 2003.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Water Extraction Procedure Sheet (example)

26.2. Attachment II: Sonication Extraction Procedure Sheet (example)

26.3. Attachment III: Wipe Extraction Procedure Sheet (example)

26.4. Attachment IV: Oil Extraction Procedure Sheet (example)

26.5. Attachment V: PCB Dual Column Corrective Action Guidelines

26.6. Attachment VI: Microwave Extraction Procedure (example)

27. Revisions

Document Number	Reason for Change	Date
S-MN-O-432-Rev.29	Removed attachments III/VI/VII Section 11 – Calibration and standardization instructions added Section 12 – Procedure updated Section 14 – Data Analysis and calculations updated	04Jan2017
S-MN-O-432-Rev.30	Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Changed contents of 11.2.3. 12.6.5 – Removed "Peaks common to…either compound." Added "separate peaks for each aroclors may be used." Added Attachment VIII. Added reference to Attachment VI (missing from previous revision). Updated revision of Attachment I added. Updated 6.2 to LLC. Added "Cleanup procedures may be used to remove such interferences. Refer to the appropriate cleanup SOPs if extract cleanup to remove interferences is required" To 6.3. Reworked all of Section 11. Table 11.13 – added Criteria for RL Standard of "±50% for non MN for each analyte set to linear or quadratic and ±30% of the true value for all levels of a linear or quadratic curve"; updated Comments for ICV and CCV adding "and perform reanalysis" and deleting "Only two…to back"; Updated Parameter for CCV to "running a CCV more frequently" instead of "a greater frequency of…ten injections." New section 12.2.2 and added 12.2.3. Changed 12.3 to Qualitative Analysis instead of Sample Analysis. Deleted 12.3.1. Deleted copyright for Target software from 12.3.2 and 12.3.2.2. Updated SOP reference in 12.3.2.2. 12.3.2.2. – deleted "Review peak identification…..not a target analyte." Deleted previous section 12.6 and all subsections except 12.6.2 (now 12.3.3.) Added new sections 12.3.4 through 12.4.7.6. Moved contents of Section 14 into new section 12.4.7. Deleted Attachments VII and VIII.	23Apr2018
S-MN-O-432-Rev.31	Added "For WI samples…" exception to Table 13.1, MB row, Corrective Action column. Added Section 12.4.6.1. New revision of Attachment I.	10May2018

Attachment I: Water Extraction Technique Sheet (example)

Pace Analytical®	Document Name: SW8082 Water Extraction Procedure	Document Revised: 14May2018 Page 1 of 1
	Document No.: F-MN-O-193-rev.11	Issuing Authority: Pace Minnesota Quality Office

Analysis Method: SW8082 – Water

Extraction Procedure – Method 3510

Holding Time:	Samples should be extracted within 1 year from sample collection.
QC Requirements:	A method blank (MB) and LCS/MS/MSD (if sufficient sample is available, LCSD if insufficient volume) must be performed each day or every 20 samples, whichever is more frequent.
Extraction Solvent:	Methylene Chloride ($MeCl_2$)
Extraction:	• Rinse each 2L separatory funnel with 30 mL MeCl2 and collection vessel (250 or 500 mL Erlenmeyer flasks) three times with MeCl2. Rinse with acetone as needed to remove any water from flasks. • Label the collection vessel with the sample number, analysis, and date extracted. Label separatory funnels with the sample ID. • Measure the initial volume and pH of each sample and record on the extraction sheet. The state of WI requires a set of MS/MSD in a batch if the sample is from WI. If there is insufficient sample volume for a MS/MSD, the extra container from the state of WI must be split into 3 portions (parent sample, MS, and MSD). A placeholder must be requested from the PM and used as the parent sample. The volume will then be brought up for extraction purposes. o If more than 5% sediment is present in the sample container, refer to SOP MN-L-142. o If the pH is not in the range of 5-9, adjust it lower with 1:1H2SO4 or raise it with 10 NaOH • Use 1L of DI water for the MB and LCS and pour directly into the funnel. • Add 100 uL of surrogate solution to each QC and sample container, and 50 uL of matrix spike solution to the LCS/MS/MSD. • Pour the samples into the rinsed and labeled separatory funnel. • Rinse each sample bottle with 60 mL of Methylene Chloride, transfer to the funnel and shake the funnel for 4 minutes on the tumblers, making sure to properly vent the funnel initially into a hood. Do not rinse the jars with more than 5% sediment with MeCl2. • After the samples have been shaken, wait 10 minutes before draining the solvent layer into the collection vessel. • Repeat 2 more times.
Finalization:	• Assemble a KD/concentrator tube apparatus. • Add sodium sulfate to each extract and transfer only the extract to the KD apparatus. • Concentrate on the waterbath to 10 mL at a temperature of approximately 95°C. • Add 10 mL of hexane through the top of the macrosnyder, and concentrate the extract to approx. 8 mL, shaking the macrosnyder periodically to ensure full conversion. • Remove the KD from the bath, disassemble, and transfer the extract to the nitrogen blow-down. Each extract should be concentrated to a 1.0 mL final volume. • Transfer extract in labeled 1mL autovial. • Add .75 mL of concentrated H_2SO_4 into the 1 mL final volume of the sample. • Vortex the vial for approximately 30 seconds. Then allow the layers to separate into an acid layer on the bottom and an extract layer on top. If the layers do not separate, centrifuge the vials for 3 minutes. • After the layers separate decant approximately 1 mL of top layer into a labeled auto vial. Be sure no acid gets into the vial as it will ruin the GC column. • Proceed with sulfur cleanup if necessary. • Sulfur Cleanup o Add enough copper powder to cover the bottom of the auto vial that contains approximately 1 mL of sample. o Vortex the sample for 30 seconds. o Shake the sample on the shaker table for approximately 20 minutes. o Let the sample settle. If sulfur is present, a black sediment will be seen on top of the copper powder. Decant the sample from the copper powder using a disposable Pasteur pipette, being careful to not suck up any copper powder. o Place in new labeled auto vial. Repeat as necessary.
Note:	• Assure crimptops are crimped tightly to the autovial • Each autovial should be double-checked to ensure there is no acid remaining in the sample extract before it is brought to the analytical area.
Final Volume:	1.0 mL
Final Solvent:	Hexane

Attachment II: Sonication Extraction Technique Sheet (example)

Pace Analytical	Document Name: SW8082 Soil Extraction Procedure	Document Revised: 09Dec2015 Page 1 of 1
	Document No.: F-MN-O-194-rev.07	Issuing Authority: Pace Minnesota Quality Office

Analysis Method: SW8082 – Soil

Extraction Procedure – Sonication Method 3550

Holding Time:	Samples should be extracted within 1 year from sample collection.
QC Requirements:	A method blank (MB) and LCS/MS/MSD (if sufficient sample is available, LCSD if insufficient volume) must be performed each day or every 20 samples, whichever is more frequent.
Extraction Solvent:	Hexane/Acetone 80:20
Extraction:	• Weigh out 30g of the sample. Add sodium sulfate (enough to have the sample free-flowing). Mix thoroughly. Allow the samples to dry and mix again if needed. • Add 1.0 mL of the surrogate spike to each sample. • Add 0.5 mL of matrix spike to the LCS and/or MS/MSD. • Add 60 mL (or enough solvent to completely submerge the soil, leaving at least ½ inch of solvent above the sediment layer to ensure that the probe is not coming into contact with the soil) of the solvent mixture immediately following the spiking procedure. • Place samples in the sonicator box and sonicate each sample three times, each at 3 minutes. • After each sonication, filter the sample, pouring the extract into a 500 mL round bottom flask.
Finalization:	• Samples are concentrated on mantles using a heat setting of 8. • Boiling chips are added to the round bottom flasks. • Add 3-ball Snyder columns to the flasks. Concentrate to approximately 9 mL final volume. • Label a 20 mL amber crimptop vial with the sample number, extraction date, and analysis. • Transfer the extract from the round bottom flask into the 20 mL vial by pouring and rinsing the flask with small amounts of hexane. • Bring the extract to a final volume of 10 mL and fully dispense all the extract from the pipet. If the extract is over 10 mL, place on N-Evap and blow it down to 10mL. • Using the same 10 mL pipette place approximately 2mL of sample extract into a 7mL vial. • Add 2 mL of concentrated H_2SO_4 to the vial containing the sample. • Vortex the vial for approximately 30 seconds. Then allow the layers to separate into an acid layer on the bottom and an extract layer on top. If the layers do not separate, centrifuge the vials for 3 mins or less until the layers can be seen. • After the layers separate decant approximately 1 mL of top layer into a labeled auto vial. Be sure no acid gets into the vial as it will ruin the GC column. • Proceed with sulfur cleanup if necessary. • Sulfur Cleanup o Add enough copper powder to cover the bottom of the auto vial that contains approximately 1 mL of sample. o Vortex the sample for 30 seconds. o Shake the sample on the shaker table for approximately 20 minutes. o Let the sample settle. If sulfur is present, a black sediment will be seen on top of the copper powder. Decant the sample from the copper powder using a disposable Pasteur pipette, being careful to not suck up any copper powder. o Place in new labeled auto vial. Repeat as necessary.
Note:	• Assure crimptops are crimped tightly to the autovial • Each autovial should be double-checked to ensure there is no acid remaining in the sample extract before it is brought to the analytical area.
Final Volume:	10.0 mL
Final Solvent:	Hexane

Attachment III: Wipe Extraction Technique Sheet (example)

Pace Analytical	Document Name: SW8082 Wipe Extraction Procedure	Document Revised: 21Feb2013 Page 1 of 1
	Document No.: F-MN-O-196-rev.03	Issuing Authority: Pace Minnesota Quality Office

Analysis Method: SW8082 – Wipes

Extraction Procedure – Method 3580

Holding Time:	Samples should be extracted within 14 days from sample collection.
QC Requirements:	A method blank (MB) and LCS/MS/MSD (if sufficient sample is available, LCSD if insufficient volume) must be performed each day or every 20 samples, whichever is more frequent.
Extraction Solvent:	Hexane
Extraction:	• Extract each wipe in the amber jar it was received by the laboratory. A single gauze pad can be used for each QC. • Add sufficient Hexane to each GN to extract to total. MBlk and samples should have a total volume of 9.5 mL hexane. • LCS, LCSD, MS and MSD should have a total volume of 9.25 mL hexane. Make sure each wipe is submersed into the hexane. • Inject 250 µL of matrix spike into the LCS/LCSD (MS/MSD if necessary), and 500 µL of surrogate spike into each sample.
Finalization:	• Add 5 mL of sulfuric acid to each jar shake vigorously for 2 minutes. If wipe still has not dissolved in acid, shake until it does. • Decant 1 mL of hexane layer into labeled autovial and cap vial. Additional sulfur cleanup may be needed. • Sulfur Cleanup o Add enough copper powder to cover the bottom of the auto vial that contains approximately 1 mL of sample. o Vortex the sample for 30 seconds. o Shake the sample on the shaker table for approximately 20 minutes. o Let the sample settle. If sulfur is present, a black sediment will be seen on top of the copper powder. Decant the sample from the copper powder using a disposable Pasteur pipette, being careful to not suck up any copper powder. o Place in new labeled auto vial. Repeat as necessary.
Note:	• Each autovial should be double-checked to ensure there is no acid remaining in the sample extract before it is brought to the analytical area.
Final Volume:	10.0 mL
Final Solvent:	Hexane

Attachment IV: Oil Extraction Technique Sheet (example)

Pace Analytical®	Document Name: SW8082 Oil Extraction Procedure	Document Revised: 21Feb2013 Page 1 of 1
	Document No.: F-MN-O-197-rev.03	Issuing Authority: Pace Minnesota Quality Office

Analysis Method: SW8082 – Oil

Extraction Procedure – Method 3580

Holding Time:	Samples should be extracted within 14 days from sample collection.
QC Requirements:	A method blank (MB) and LCS/MS/MSD (if sufficient sample is available, LCSD if insufficient volume) must be performed each day or every 20 samples, whichever is more frequent.
Extraction Solvent:	Hexane
Extraction:	• Weigh out 0.1g of sample into a tared 7 mL vial. • Use two significant figures on the bench sheet. MB/LCS should be standard PCB-free cooking oil. • Add 4.5 mL of hexane to the MB and samples. The LCS should have 4.0mls of Hexane. • Spike 0.5 mL of surrogate into all samples. • Add 0.5 mL of matrix spike to the LCS/MS/MSD. • The final volume of each sample should TOTAL 5.0 mL. • Vortex each extract ~10 seconds after spikes and hexane have been added.
Finalization:	• Aliquot two mL of the hexane extract, along with two mL of sulfuric acid into a 7 mL screwtop autovial. • Vortex this mixture for ~30 seconds. A two layer separation should occur. If not, then extract should be centrifuged until separation occurs. • Additional sulfur cleanup may be needed. • Sulfur cleanup o Add enough copper powder to cover the bottom of the auto vial that contains approximately 1 mL of sample. o Vortex the sample for 30 seconds. o Shake the sample on the shaker table for approximately 20 minutes. o Let the sample settle. If sulfur is present, a black sediment will be seen on top of the copper powder. Decant the sample from the copper powder using a disposable Pasteur pipette, being careful to not suck up any copper powder. o Place in new labeled auto vial. Repeat as necessary.
Note:	• Each autovial should be double-checked to ensure there is no acid remaining in the sample extract before it is brought to the analytical area.
Final Volume:	5.0 mL
Final Solvent:	Hexane

Attachment V: PCB Dual Column Corrective Action Guidelines

Pace Analytical	Document Name: PCB Dual Column Corrective Action Guidelines [redacted]	Document Revised: 09July2014 Page 1 of 1
	Document No.: F-MN-O-282-rev.00	Issuing Authority: Pace Minnesota Quality Office

PCB Dual Column Corrective Action Guidelines [redacted]

Primary Column	Confirmation Column	Corrective Action
TCX and DCB recoveries low	Not Evaluated	RR
TCX and DCB recoveries high	Not Evaluated	Report NDs, RR others
One surrogate outside control limits but CCAL okay	Same as primary column	Report
One surrogate outside control limits but CCAL okay	The surrogate that fails in the sample fails in CCAL	Report
The surrogate that fails in sample fails in CCAL	Same as primary column	RR
The surrogate that fails in sample fails in CCAL	CCAL passes	RR
The surrogate that fails in sample is okay in CCAL	Same as primary column	Report
Surrogates pass control limits in sample but one fails in CCAL	Same as primary column	Report with CC footnote
Surrogates pass control limits in sample but one fails in CCAL	CCAL passes	Report with CC footnote
Surrogate in samples and CCAL pass control limits	Surrogates pass control limits in sample but one fails in CCAL	Report
CCAL target compounds fail high	CCAL target compounds pass	Reports NDs
CCAL target compounds fail high	CCAL target compounds fail high	Reports NDs
CCAL target compounds pass	CCAL target compounds fail high	Reports NDs
CCAL target compounds fail low	CCAL target compounds pass	RR all
CCAL target compounds fail low	CCAL target compounds fail low	RR all
CCAL target compounds pass	CCAL target compounds fail low	RR

Attachment VI: Microwave Extraction Procedure (example)

Pace Analytical®	Document Name: SW8082 Soil Extraction (Microwave) Procedure	Document Revised: 25Oct2016 Page 1 of 2
	Document No.: F-MN-O-317-rev.00	Issuing Authority: Pace Minnesota Quality Office

Analysis Method: SW8082 – Soil/Sediment

Extraction Procedure – Microwave Automated Extraction (MAE) 3546

Holding Time:	Samples should be extracted within 1 year from sample collection.
QC Requirements:	A method blank (MB) and LCS/MS/MSD (if sufficient sample is available, LCSD if insufficient volume) must be performed each day or every 20 samples, whichever is more frequent.
Extraction Solvent:	Hexane/Acetone – 80:20
Extraction:	• See S-MN-O-465 Glassware Cleaning, or equivalent replacement, on proper glassware and cleaning practices. Rinse and label all glassware appropriately • Group samples by fraction to a maximum of 20 paying samples per extraction batch. An extraction batch consists of a method blank, appropriate quality control samples, and associated paying samples • Homogenize sample according to Sample Homogenization and Sub-sampling SOP for homogenization process S-MN-L-147, or equivalent replacement. • All extraction glassware and vessels (250 mL Erlenmeyer flasks, microwave vessels, etc.) must be rinsed 3 times with Methylene Chloride (any residual water must be removed completely using Acetone and Methylene Chloride as necessary) Prepare samples for extraction • Weigh approximately 30 grams of each sample (5 grams for caulk sample) into a rinsed and labeled microwave vessel. Document the actual sample weight on the bench sheet (Attachment IV) • For laboratory control samples, prepare with each group of samples a method blank (MB), laboratory control spike (LCS) and/or laboratory control spike duplicate (LCSD), use 30 grams, 5 for caulk (to the nearest 0.1g) of Ottawa sand weighed out into a rinsed, and labeled microwave vessel • A matrix spike (MS) and matrix spike duplicate (MSD) should be weighed out to 30 g , 5 grams for caulk(record weight to the nearest 0.1 g, which should be to the same weight as the original sample, on extraction benchsheet. The client, supervisor, or project manager may designate the MS/MSD sample, but one set should be completed per batch. • If there is insufficient sample remaining to perform an MS/MSD or additional quality control information is needed (program specific), a laboratory control sample duplicate (LCSD) may be prepared • Add approximately 1 scoop of sodium sulfate to each sample (including QC samples). • To all quality control spike samples (MS/MSD/LCS/LCSD), 0.5 mL PCB matrix spiking solution (Table 10.2) should be added • Add 1 mL of TCMX/DCB surrogate working standard (Table 10.2) to all samples, blanks, and quality control spiked samples • All spiking should be verified by a second person and recorded on the extraction bench sheet (Attachment VI) according to SOP S-MN-O-497, or equivalent replacement. • Label the microwave vessels with a sharpie to note the sample IDs. Pour the sample into the microwave vessel using a powder funnel. Organize vessels in the vessel rack. • Dispense 25 mL of 80/20 Hexane/Acetone into the microwave vessel using the powder funnel, rinsing the funnel as the solvent is added • Stopper and cap each vessel. Start the cap by hand to make sure the threads are aligned correctly and finish using the Vessel Capping Station, which will result in all vessels being capped uniformly. Shake each vessel well to mix the contents • Put the vessels in the turntable, labeling the turntable position of each on the bench sheet • Load the turntable containing the microwave vessels into the microwave. The turntable will lock in place when aligned correctly • Select the method in the CEM Main menu for organic extraction based on the number of vessels that will be processed together. For 6 to 20 vessels, select the method "SIM SOIL 800", and for more than 20 vessels use "SIM SOIL 1600". The methods are identical except for the power (in watts) • The required parameters for the extraction of organic compounds from soil samples are listed in Table 1 below

Document Information

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STANDARD OPERATING PROCEDURE

DETERMINATION OF DIESEL RANGE ORGANICS

Reference Methods: Wisconsin Modified DRO (WIDRO)

Local SOP Number:	S-MN-O-466-rev.26
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APPROVALS

Laboratory General Manager — Date: 23 May 2018

Laboratory Quality Manager — Date: 23 May 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-466-rev.26

TABLE OF CONTENTS

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. The purpose of this Standard Operating Procedure (SOP) is to define the process to determine diesel range organics (DRO) by Method WIDRO.

2. SUMMARY OF METHOD

2.1. Water samples are extracted at a pH of ≤ 2 with methylene chloride and analyzed using a gas chromatograph equipped with a flame ionization detector (FID). Soil samples are extracted with methylene chloride and analyzed using a gas chromatograph equipped with a flame ionization detector (FID).

3. SCOPE AND APPLICATION

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method process.

3.2. **Parameters**: This SOP applies to the measurement of the concentration of petroleum products such as diesel or fuel oil in water and soil. This relates to a hydrocarbon range of C10-C36. The standard WDRO carbon range is C10-C28'.

3.3. A 10 component standard is used for initial calibration and continuing calibration checks for routine WDRO work. The following analytes are found in the standard mix.

Components	
Decane	Eicosane
Dodecane	Decosane
Tetradecane	Tetracosane
Hexadecane	Hexacosane
Octadecane	Octacosane

3.4. For Extended WDRO or TEH screening a standard containing the following compounds will be used:

Components		
Decane – C10	Eicosane – C20	Triacontane – C30
Dodecane – C12	Decosane – C22	Dotriacontane – C32
Tetradecane –C14	Tetracosane – C24	Tetratriacontane – C34
Hexadecane – C16	Hexacosane – C26	Hexatriacontane – C36
Octadecane – C18	Octacosane – C28	

4. APPLICABLE MATRICES

4.1. This SOP is applicable to groundwater and soil.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. The reporting limit (LOQ) for DRO waters is 0.1 mg/L for groundwater and 10 mg/kg for soil. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. INTERFERENCES

6.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Interferences by phthalate esters can pose a major problem in organic analysis. These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. Phthalates are easily extracted or leached from materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are

handled. Avoiding the use of plastics in the laboratory can best minimize interferences from phthalates. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

6.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the site being sampled.

6.4. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

6.5. Other organic compounds, which include chlorinated hydrocarbons and phthalate esters, are measurable. As defined in method, the DRO results include these compounds.

6.6. Method interferences are regulated by washing all glassware with hot soapy water and then rinsing with tap water, deionized water, acetone, and methylene chloride.

6.7. DRO samples may be complicated by biogenic interferences which includes materials such as naturally occurring organics. These concentrations may occur at levels well above the regulatory limit. Silica gel clean up is a well established procedure used to separate analytes of different polarity. The majority of "fresh" or non-biodegraded petroleum hydrocarbons are considered non-polar compounds. Depending upon the soil makeup, the majority of the biogenic compounds may be polar or semi-polar in nature. The silica gel clean up procedure will preferentially remove polar and semi-polar compounds, thus leaving the non-polar or petroleum hydrocarbons behind. See Section 12.5 for the Silica Gel Cleanup Process.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Table 7.1 – Sample Collection, Preservation and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	1 L or 250 mL amber glass containers	Acidified with 5 mL for 1L containers and 2 mL for 250 mL containers of 1:1 Hydrochloric acid (HCl) to pH<2	<6°C but above freezing	Must be extracted within 7 days of collection and analyzed within 40 days of extraction
Soils	4 oz pre-tared jars; collect 25-70 grams	NA	<6°C but above freezing	Must be extracted within 10 days of collection and analyzed within 40 days of extraction

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Separatory funnels	2L separatory funnel with Teflon® lined cap	Fisher Scientific or equivalent replacement
Stopper	Teflon®	Fisher Scientific or equivalent replacement
Graduated cylinder	1 L	Fisher Scientific Cat.# 08-548-207
Erlenmeyer flask	250 mL	Fisher Scientific Cat.# 07250090
Ringstand	Fisher Scientific Cat.#	Fisher Scientific Cat.# S47815
pH paper	Wide range pH strips	Fisher Scientific Cat.# 09-876-17
Concentrating glassware	Kuderna-Danish concentrating setup, including concentrator tube, 500 mL evaporative flask, and three ball macro Snyder	Fisher Scientific Cat.# 192002AP2

Balance	Top-loading balance, capable of weighing to the nearest 0.1 g (samples)	Denver Instruments MXX-612 or A&D EK-410i or equivalent replacement
Stainless steel stirring utensil	butter knives	Purchased at local dollar store
Sonicating bath	Ultrasonic Bath for extraction	UltraSonicator Branson Model 8510 or equivalent replacement
Water bath	Heated with concentric ring cover, capable of temperature control (±5°C)	In house counter water bath
Buchi Concentrator	Concentrator with F-108 recirculating chiller, concentrator vessels with V-855 vacuum controller	Buchi, or equivalent replacement
Jars	60 mL or 120 mL wide mouth jars with Teflon-lined caps	C&G containers or equivalent replacement
Syringes	Microsyringes	Hamilton or equivalent replacement
Filter paper	Fluted filter paper	Whatman or equivalent replacement
Evaporation system	N-EVAP, nitrogen evaporator with high purity nitrogen gas	Fisher Scientific or equivalent replacement
Pipets	Disposable	Fisher Scientific or equivalent replacement
Boiling Chips		Fisher Scientific or equivalent replacement Fisher Scientific Cat.#02215521
Vials	2 mL autovials with Teflon® lined crimp seals	Fisher Scientific or equivalent replacement
Aluminum foil		Fisher Scientific or equivalent replacement
Funnel	Powder funnel	Fisher Scientific or equivalent replacement
Glass wool		Fisher Scientific Cat.#11-388
Silica gel	Purchased pre-activated silica gel 60-100 mesh	Supelco Cat.#236799-1KG
Gas Chromatograph (GC)	Hewlett Packard 5890 Series II, 6890, or 7890 LTM, equipped with a Hewlett Packard 7673, 7683, or 7693 autosampler interfaced to HP Chemstation which transfers the data to a Target Server for data processing	Hewlett Packard, or equivalent replacement
Column	DB-5MS or comparable column bonded phase (0.5 µm film thickness), 30 meter by 0.25 mm ID column for non LTM/MACH door	Fisher Scientific or equivalent replacement
Data Acquisition Software	Used to acquire raw data and transferred to Target for final processing	Chemstation, or equivalent replacement
Processing software	Target, see master list for current version	Target
Data Reporting software	Laboratory Information Management System (LIMS)	Horizon, or equivalent replacement
Data Review and Package software	Used to generate paperless packages for review and L4 data packages	Gandalf, or equivalent replacement

10. REAGENTS AND STANDARDS

10.1. Table 10.1 – Reagent and Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
Sodium Sulfate	Na_2SO_4 – granular, anhydrous (see SOP S-MN-O-500, or equivalent replacement, for preparation instructions)	Fisher Scientific Cat.#S-415-200
Methylene Chloride	CH_2Cl_2 – Optima Grade or equivalent	Fisher Scientific Cat.#D151-4
Sodium Chloride	NaCl	Fisher Scientific Cat.#S-271-10
Hydrochloric Acid	Concentrated HCl	Fischer Scientific or equivalent

(HCl)		replacement
Organic free sand	Rinsed and baked sand used for soil method blanks	Fisher Scientific Cat.#S-23-50
Initial Calibration Stock Standard (ICAL)	1,000 µg/mL each alkane (peak), used for the WDRO. Extended WIDRO, and TEH screen	Accustandard CT ETPH Alkane Standard Cat# DRH-009S-PAK
Surrogate Spike Solution(1L extractions) ntcs-SS GCSV	n-Triacontane-d62, 5,000 µg/mL	Accustandard DRH-SS
ICV standard	Custom CT EPH Standard 1,000 ug/mL of each even alkane C10-C36 C10-C28 = 10,000 ug/mL total C10-C32 = 12,000 ug/mL total C10-C36 = 14,000 gu/mL total	Phenova AL0-130190

10.2. Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Calibration standard CCV	1.25 mL DRO ICAL Verification Stock and 0.20 mL Surrogate solution	MeCl2	8.55 mL	10.0 mL	2500 µg/mL and 100 µg/mL
LL-ICV Stock	0.010 mL of ntcs-SS GCSV 0.125 mL of Custom CT EPH Standard	MeCl2	0.865 mL	1.0 mL	50/1250 ug/mL
DRO-ICV	0.020 mL of ntcs-SS GCSV 0.250 mL of Custom CT EPH Standard	MeCl2	0.730 mL	1.0 mL	100/2500 ug/mL
Extended WDRO Ical Stock	2.00 mL of ICAL Stock and 0.160 mL of Surrogate Solution	MeCl2	1.84	4.0 mL	5000 ug/mL and 200 ug/mL
Calibration Std 1 WIDRO (C10-28)	0.010 mL of Extended WDRO Ical Stock	MeCl2	0.990 mL	1.000 mL	50 µg/mL and 2 µg/mL
Calibration Std 2 WIDRO (C10-28)	0.020 mL of Extended WDRO Ical Stock	MeCl2	0.980 mL	1.000 mL	100 µg/mL and 4 µg/mL
Calibration Std 3 WIDRO (C10-28)	0.050 mL of Extended WDRO Ical Stock	MeCl2	0.950 mL	1.000 mL	250 µg/mL and 10 µg/mL
Calibration Std 4 WIDRO (C10-28)	0.100 mL of Extended WDRO Ical Stock	MeCl2	0.900 mL	1.000 mL	500 µg/mL and 20 µg/mL
Calibration Std 5 WIDRO (C10-28)	0.200 mL of Extended WDRO Ical Stock	MeCl2	0.800 mL	1.000 mL	1000 µg/mL and 40 µg/mL
Calibration Std 6 WIDRO (C10-28)	0.500 mL of Extended WDRO Ical Stock	MeCl2	0.500 mL	1.000 mL	2500 µg/mL and 100 µg/mL
Calibration Std 7 WIDRO (C10-28)	1.000 mL of Extended WDRO Ical Stock	MeCl2	Not Applicable	1.000 mL	5000 µg/mL and 200 µg/mL
Spiking Standard (for LCS and MS) Water 1 Liter and Soil DRO-SPK GCSV	Initial Calibration Verification Stock (ICV) 100 uL for water and for soil	MeCl2	Not Applicable	Not Applicable	2.0 mg/L for waters and 80 mg/Kg for soils
Spiking Standard (for LCS and MS) Water 250 mL DROLL-SPK GCSV	1.0 mL of Intial Calibration Verification Stock(ICV)	MeCl2	9.0 mL	10.0 mL	2000 ug/mL
Surrogate Standard for 1L waters and soils	10 uL for 1 liter waters, 5 uL for 250 mL waters and 25 uL for soils of Surrogate Spike Solution	MeCl2	Not Applicable	Not Applicable	50 ug/mL waters and 125 ug/mL soils on column
Calibration Std 1 WIDRO Extended (C10-32)	0.010 mL of Extended WDRO Ical Stock	MeCl2	0.990 mL	1.000 mL	60 µg/mL
Calibration Std 2 WIDRO Extended (C10-32)	0.020 mL of Extended WDRO Ical Stock	MeCl2	0.980 mL	1.000 mL	120 µg/mL

Calibration Std 3 WIDRO Extended (C10-32)	0.050 mL of Extended WDRO Ical Stock	MeCl2	0.950 mL	1.000 mL	300 µg/mL
Calibration Std 4 WIDRO Extended (C10-32)	0.100 mL of Extended WDRO Ical Stock	MeCl2	0.900 mL	1.000 mL	600 µg/mL
Calibration Std 5 WIDRO Extended (C10-32)	0.200 mL of Extended WDRO Ical Stock	MeCl2	0.800 mL	1.000 mL	1200 µg/mL
Calibration Std 6 WIDRO Extended (C10-32)	0.500 mL of Extended WDRO Ical Stock	MeCl2	0.500 mL	1.000 mL	3000 µg/mL
Calibration Std 7 WIDRO Extended (C10-32)	1.000 mL of Extended WDRO Ical Stock	MeCl2	Not Applicable	1.000 mL	6000 µg/mL
Calibration Std 1 WDRO, Extended(C10-36)	0.010 mL of Extended WDRO Ical Stock	MeCl2	0.990 mL	1.000 mL	70 µg/mL
Calibration Std 2 WDRO, Extended (C10-36)	0.020 mL of Extended WDRO Ical Stock	MeCl2	0.980 mL	1.000 mL	140 µg/mL
Calibration Std 3 WDRO, Extended (C10-36)	0.050 mL of Extended WDRO Ical Stock	MeCl2	0.950 mL	1.000 mL	350 µg/mL
Calibration Std 4 WDRO, Extended (C10-36)	0.100 mL of Extended WDRO Ical Stock	MeCl2	0.900 mL	1.000 mL	700 µg/mL
Calibration Std 5 (C10-36)	0.200 mL of Extended WDRO Ical Stock	MeCl2	0.800 mL	1.000 mL	1400 µg/mL
Calibration Std 6 WDRO, Extended(C10-36)	0.500 mL of Extended WDRO Ical Stock	MeCl2	0.500 mL	1.000 mL	3500 µg/mL
Calibration Std 7 WDRO, Extended (C10-36)	1.000 mL of Extended WDRO Ical Stock	MeCl2	Not Applicable	1.000 mL	7000 µg/mL
DROLL-CAL7	.25 mL of Extended WDRO Ical Stock	MeCl2	0.750 mL	1.000 mL	1250/50
DROLL-CAL 6	.125 mL of Extended WDRO Ical Stock	MeCl2	0.875 mL	1.000 mL	625/25 µg/mL
DROLL-CAL 5	.050 mL of Extended WDRO Ical Stock	MeCl2	0.950 mL	1.000 mL	250/10 µg/mL
DROLL-CAL 4	.025 mL of Extended WDRO Ical Stock	MeCl2	0.975 mL	1.000 mL	125/5 µg/mL
DROL- CAL 3	.0125 mL of Extended WDRO Ical Stock	MeCl2	0.9875 mL	1.000 mL	62.5/2.5 µg/mL
DROL- CAL 2 (make 2 used to make CAL 1)	.005 mL of Extended WDRO Ical Stock	MeCl2	0.995 mL	1.000 mL	25/1 µg/mL
DROLL-CAL 1	.5 mL of DROLL-CAL1	MeCl2	0.500 mL	1.000 mL	12.5/.5 µg/mL
DROLL-ICV	0.500 mL of LL-ICV-Stock	MeCl2	0.500 mL	1.000 mL	625/25 ug/mL
DROLL-CCV	.3125 mL of DRO Mix (Restek) .050 mL of n-triacontane d62 (Accustandard)	MeCl2	9.6375 mL	10.0 mL	625/25 ug/mL

11. CALIBRATION AND STANDARDIZATION

11.1. Table 11.1 – Calibration requirements

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression	$r \geq 0.99$	If not met, try non-linear regression fit. If still not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ±20%	If the requirements for initial calibration verification are not met, check for instrument error, standard preparation errors, calculation errors. Remake standards if it is suspected and reanalyze the standard. Only two injections of the same standard are permitted back to back.

Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 20 samples thereafter. Samples must be bracketed with a closing CCV standard.	% Diff ±20%	If the requirements for continuing calibration are not met, check for instrument error, standard preparation errors, calculation errors. Remake standards if it is suspected and reanalyze the standard. Only two injections of the same standard are permitted back to back.

11.2. For all calibration calculations see the most current quality manual.

11.3. See Attachment I: Recommended Chromatographic Conditions in Attachment I for suggested conditions for the operating system with or without a MACH Door unit.

11.4. Gas Chromatograph (GC) Calibration

11.4.1. The GC is calibrated using the external standard procedure.

11.4.2. Initial calibration standards are prepared in methylene chloride at a minimum of 5 concentration levels. One of the calibration standards should be at a concentration near the quantitation limit, and the other concentrations should define the working range of the method. See Table 10.2.

11.4.3. A summation of peak area for all peaks eluting between n-decane and n-octacosane is used for calibration purposes.

11.4.4. The retention time window is defined as beginning approximately 0.1 min. before the retention time of n-decane and ending 0.1 min after the retention time of n-octacosane is the calibration run. The retention time of the DRO pattern elutes much faster using a Gerstel MACH or Agilent LTM door while not sacrificing sensitivity of the system. For this reason, the retention time window for WDRO was shortened to 0.05 minutes before the n-decane to 0.05 minutes after the n-octacosane for analysis with a MACH door for normal WDRO work, defined as prior to the C10 peak to after the C32 peak for the extended WDRO (not including the area for the surrogate), and defined as prior to the C10 peak to after the C36 peak for the TEH screen value (not including the area for the surrogate).

11.4.5. Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of the chromatogram that includes all responses within the retention time window. The correct baseline is placed at the lowest point in the chromatogram before the end of the window. Typically, the baseline is taken from before the solvent peak (integrators are set up this way) as this is the lowest point in the chromatogram. However, the lowest point may be within the window, before the window, or before the solvent front so make sure you evaluate each injection. Baseline to baseline integration does not include the solvent peak.

11.4.6. See Table 11.1 for calibration criteria.

11.4.7. Every 20 samples/24 hours, the mid level calibration standard (continuing calibration standard) will be analyzed (at a minimum). The retention time (RT) for the analytes must be within the windows determined from the initial calibration sequence. The calculated concentrations for DRO must be within ±20% difference of the true value of the mid-point standard. If the concentration is not within ± 20% difference of the initial calibration, maintenance should be performed and samples re-analyzed – unless the CCV is biased high and the associated samples were non-detect.

12. PROCEDURE

12.1. Water Sample Extraction Process (separatory funnels)

12.1.1. Sample IDs should be verified by another lab personnel.

12.1.2. Measure the initial volume and record the amount of sample in the 1L or 250 mL amber glass container on the extraction sheet.

12.1.2.1. If more than 5% of the entire sample volume is sediment present in the bottom of the glass container, follow the procedure in S-MN-L-142 (or equivalent replacement).

12.1.3. Using pH paper, pipet a small amount of the sample onto a pH strips and record the pH of each sample on the extraction sheet.

12.2. For 1 liter extraction blanks and LCS/LCSDs, pour 1.0 L of deionized water into the separatory funnel. For 250 mL extraction blanks and LCS/LCSDs, pour 250 mL of deionized water into the separatory funnel. All records should be written on the extraction bench sheet.

12.2.1. Add 100g NaCl to all 1 liter QC, blanks, and samples. Add 25g NaCl to all 250 mL QC, blanks, and samples. Cap and shake each separatory funnel to dissolve the NaCl.

12.2.2. Add 2 mL of concentrated HCl to 1 liter blanks and LCS/LCSDs to ensure the pH is <2. Add .5 mL of concentrated HCl to 250 mL blanks and LCS/LCSDs to ensure the pH is <2. Record on Comment portion of the extraction sheet any samples which have a pH >2. If sample pH is not <2, pH is recorded on the extraction sheet and data is flagged by the analyst.

12.2.3. Add 100 µL of DRO spiking solution (Table 10.2) using DRO-SPK GCSV for 1 liter extractions and DROLL-SPK GCSV for 250 mL extractions to all quality control samples and MS/MSD. All spiking should be verified by a second person and recorded on the extraction bench sheet.

12.2.4. For 1L extractions add 10 µL DRO surrogate (ntcs-SS GCSV – see 10.1) to all QC, blanks and sample containers. For 250 mL extractions add 5 µL DRO surrogate (ntcs-SS GCSV – see 10.1) to all QC, blanks and sample containers. Pour each sample into individual 2L separatory funnels that have been rinsed with methylene chloride.

12.2.5. For 1 liter extractions add 60 mL of methylene chloride to each sample jar and for 250 mL extractions add 30 mL of methylene chloride to each sample jar (unless the sediment is greater than 5% of the sample volume- if so, see section 12.1.2), cap, shake and decant rinsate into the appropriate separatory funnel. Load sep funnels into tumbler and tumble for about 5 rotations. Stop the tumbler with the seps in the upside-down position and vent each separatory funnel to release the pressure. Set timer; funnels are tumbled for 4 minutes at a speed of 55 RPM. Alternatively, shake the samples vigorously by hand for two minutes.

12.2.6. Allow the layers to separate for 10 minutes. Drain the solvent layer through a stainless steel funnel that contains a glass wool plug and sodium sulfate all having been rinsed with methylene chloride, into an Erlenmeyer flask that has also been rinsed with methylene chloride. Rinse funnel with Methylene Chloride after sample has been allowed to drain.

12.2.7. If an emulsion has formed, make a note on the extraction sheet. See Section 21 for troubleshooting tips.

12.2.8. Repeat the extraction once more using a second 60-mL aliquot of methylene chloride for 1 liter extractions and 30 mL methylene chloride for 250 mL extractions. Collect the solvent in the same flask. Rinse funnel again after all extract is through the sulfate.

12.2.9. Cover flask with foil and store in cooler above freezing but below 6 °C until the concentration procedure (12.3 or 12.4).

12.3. Soil Sample Extraction

12.3.1. Weigh the tared sample jar (4 oz jar) to determine the actual sample weight. If the sample arrives in the 60-mL vial and weighs more than 35.4 g, or if the sample arrives in the 120-mL vial and weighs more than 70.0 grams, the project manager must be contacted followed by a client contact.

12.3.1.1. If the sample is overweight and was received unpreserved from the field, the sample can be homogenized and reweighed into a separate container to the proper method specified weight. Qualify the final data indicating that this procedure was performed to obtain the correct weight.

12.3.1.2. The entire sample can be transferred to a larger container to allow for the addition of solvent to maintain the 1:1 solvent/sample ratio. Qualify the final data indicating that this procedure was performed to obtain the correct weight.

12.3.1.3. Mix the sample to ensure homogeneity, and remove sample amount until what remains in the container is within method specified limits. Dispose of the removed amount properly. Qualify the final data indicating that this procedure was performed to obtain the correct weight.

12.3.2. Record the sample weight on the Soil Extraction bench sheet.

12.3.3. To each soil sample, add enough sodium sulfate to make sample free flowing, and stir with a stainless steel stirring utensil.

12.3.4. Prepare a method blank (MB) and LCS/LCSD by adding 25.0 grams of DRO-free baked and rinsed sand and 25 mL of methylene chloride into a 60 or 120 mL vial/jar.

12.3.5. Sample IDs must be verified by another lab personnel before being weighed out.

12.3.6. Spike100 µL DRO matrix spike solution (Table 10.2) to all quality control samples (LCS/LCSD).

12.3.7. Spike 25 µL DRO surrogate to all QC, blanks and samples. See Table 10.2.

12.3.8. All spiking should be verified by a second person and recorded on the Soil Extraction bench sheet.

12.3.9. The solvent, methylene chloride, is added in a 1:1 ratio (e.g. 25 grams of soil, 25 mL of solvent should be added.) A minimum of 25 mL of solvent must be added by removing the cap of the sample jar and dispensing the solvent carefully. More solvent may be added to cover the sample. Quickly replace the cap.

12.3.10. Tighten the caps of the sample jars to ensure no water from the bath leaks into the samples and no solvent leaks out.

12.3.11. Vigorously shake the jars for 2 minutes to ensure all contents are thoroughly mixed by hand or a mechanical shaker.

12.3.12. Place the sample jars in the ultrasonic bath, turn the sonicator on, and sonicate for 20 minutes.

12.3.13. Carefully pour the extract into a clean, rinsed 250-500mL collection flask through filter paper and funnel. Rinse filter paper with Methylene Chloride after pouring extract through.

12.3.14. Add another portion of solvent, approximately 25 mL to each sample, blank, and quality control sample.

12.3.15. Shake vigorously for two minutes and sonicate again for 20 minutes.

12.3.16. After the second sonication carefully pour the extract into the same flask.

12.3.17. Thoroughly rinse the filter paper with methylene chloride.

12.3.18. Cover tightly with foil and store at <6°C or follow with concentration in 12.3 or 12.4.

12.4. K-D Extract Drying and Concentration

12.4.1. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500- mL evaporative flask (KD apparatus). Add a clean boiling chip to the evaporative flask.

12.4.2. Add enough sodium sulfate to the extract to ensure all water is absorbed. Mix by swirling in Erlenmeyer flask. Pour into a solvent rinsed KD apparatus.

12.4.3. Rinse the sodium sulfate in the flask with methylene chloride two more times and transfer the solvent into the same KD apparatus.

12.4.4. Attach a three-ball Snyder column.

12.4.5. Place the K-D apparatus on a hot water bath (80°-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches ~4-6 mL, remove the K-D apparatus. Allow it to drain and cool. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

12.4.6. Rinse the joints on the KD apparatus with a small amount of methylene chloride as it is disassembled. Leaving the extract in the concentrator tube, carefully concentrate the extract to 1.0 mL under a gentle stream of nitrogen using the N-evap apparatus.

12.4.7. If the extract forms a precipitate or stops evaporating, the final volume should be larger than 1 mL; bring volume to the nearest whole number. Record the actual final volume on the

extraction bench sheet. Remove a 1-mL aliquot from the actual final volume and transfer to a 2.0 mL autovial.

12.4.8. Transfer the extract to a 2.0-mL autovial with a Teflon® lined crimp seal.

12.4.9. Each autovial is labeled with the extraction date, method, and laboratory sample ID. The sample extract is ready for analysis.

12.5. Buchi Sample Concentration

12.5.1. Make sure the receiving flask and secondary receiving flask are free of solvent before use.

12.5.2. Turn on the F-108 recirculating chiller.

12.5.2.1. Set the temperature to 10 °C and press the start button. Wait approximately 5 minutes for the chiller to reach the set temperature.

12.5.3. Turn on the Syncore® platform.

12.5.3.1. Set the temperature to 50°C and press the start button. Allow approximately 20 minutes for the platforms to reach the set temperature.

12.5.4. Add approximately 10 mL of DI water to each cell of the rack to help with heat transfer. The platform will not concentrate correctly without water in the cells. Make sure there is enough water causing overflow when the vessel is placed in the cell.

12.5.5. All Buchi concentration vessels must be cleaned by soap wash. Refer to SOP S-MN-O-465 for glassware cleaning procedure section 12.3.

12.5.6. Add baked sodium sulfate to all extracts to remove any water.

12.5.7. Label the vessels with batch prep labels.

12.5.8. Transfer the extract from the Erlenmeyer to the concentration vessel. Rinse the Erlenmeyer with approximately 10 mLs of methylene chloride and transfer to the vessel. Make sure no sodium sulfate is transferred into the concentration vessel.

12.5.9. Insert the vessels into the cells and evenly tighten down the vacuum cover.

12.5.10. Turn the rotation dial and set the speed to 250 RPM.

12.5.11. Select the desired gradient program that is set in the V-855 vacuum controller. See Attachment IV for desired programs. (Use 120 DCM program for WIDRO soil.)

12.5.11.1. To select desired program select "Menu".

12.5.11.2. Arrow down and select "program".

12.5.11.3. Arrow down to "open" and turn the dial to select desired program. Once program is highlighted select "ok".

12.5.11.4. Press "start" to run the program.

12.5.12. Once the program is finished select "Stop" and allow the vacuum to return to 1000 mbar. Remove the vacuum cover and take out the vessels. If a sample is higher than 1 mL place it on the N-EVAP and bring down to 1 mL.

12.5.13. Finalize using a calibrated disposable pipet into a 2 mL vial.

12.6. Sample Analysis

12.6.1. After calibration (Section 11), load MB and LCS samples onto the instrument first, followed by samples. The LCSD is generally run after the last sample in the batch but does not necessarily need to run at the end.

12.6.2. Run solvent blanks after samples that are discolored or are suspected to have a high DRO concentration. In addition, renew extraction sheet comments to check for viscous emulsion, etc. This may indicate further dilutions are needed.

12.6.3. Samples are quantified by summing all peak areas eluting between n-decane and n-octacosane. Linear regression is used to quantitate each sample. All peaks are integrated baseline to baseline.

12.6.3.1. WIDRO extended (C10-C32) is quantified from n-decane to n-dotriacontane.

12.6.3.2. TEH (C10-C36) is quantified from n-decane to n-hexatriacontane.

12.6.4. If the responses for the peaks of interest in the sample exceed the working range of the calibration curve, the extract will be diluted and reanalyzed.

12.6.5. Significant peaks and baseline rises outside of the DRO window are reported. Peaks eluding before the window are labeled as low boilers. Peaks eluding after the window will be labeled as high boilers.

12.6.6. All samples and standards should be integrated in the same manner as calibration standards.

12.6.7. Samples following high standards or over-range samples are to be monitored for carryover.

12.6.8. All data reported above the highest calibration level (see Table 10.2) must be diluted. All data quantitated below the PQL should not be reported.

12.7. DRO Silica Gel Cleanup Process

12.7.1. The cleanup procedure typically takes place after the initial DRO extract has been analyzed and the client evaluates the results. It is important that the original extract be used to minimize the variability of non-homogeneous samples.

12.7.2. The original extract is taken back to the preparation laboratory for cleanup and reinjection along with the batch QC samples (blank, LCS/LCSD, MS/MSD). Both "before and after" results are reported, unless the client only requires the result with the silica cleanup. The silica cleanup can be used on both soil and water samples.

12.7.3. A micro column is created by filling a Pasteur pipet ¾ full with activated silica gel. Soak with MeCl until the column is fully absorbed.

12.7.4. The extract is transferred to the top of the column with a disposable pipet. Rinse the extract vial with MeCl rinses. Rinse and transfer the rinsate to the top of the column a total of three times.

12.7.5. Elute with additional MeCl to obtain a total of 7 mL of solvent collected into the collection vessel.

12.7.6. Concentrate down to a final volume of 1 mL on the N-Evap.

12.7.7. Reanalyze on the GC/FID as outlined in 12.5.

13. QUALITY CONTROL

13.1. Table 13.1 Quality Control Criteria.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than ½ reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.

Laboratory Control Sample (LCS)	DI water spiked with all target compounds	One per 20 samples	75-115% for waters 70-120% for soils	Analyze a new LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Laboratory Control Sample Duplicate (LCSD)	DI water spiked with all target compounds	One per 20 samples	75-115% for waters 70-120% for soils RPD ≤ 20%	Analyze a new LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run ***Exceptions:*** If LCSD recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. Treat RPD failures like a high biased LCSD.
Matrix Spike (MS)	Client sample spiked with all target compounds	Only upon client request	75-115% for waters 70-120% for soils	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Reanalyze to confirm if it is suspect of instrument error.
MSD / Duplicate	MS Duplicate *OR* *(alternative)* Sample Dup	Only upon client request	75-115% for waters 70-120% for soils %RPD ≤ 20%	Report results with an appropriate footnote.
Surrogate	Labeled analyte used to indicate extraction efficiency	In all samples and QC	50-150%	If the surrogate recovery is outside of established limits, check for errors in calculations, sample and standards preparation and spiking of the surrogate solution, or problems with the instrument performance. Correct errors found, if the results fall within limits data may be reported. Narrate in checklist. If matrix interferences are believed to be the cause of surrogate exceedances, report the data with an appropriate qualifier. Note any sample characteristics that illustrate the reasoning behind matrix interference, i.e. large amounts of sediment, emulsions, heavy hydrocarbon content, etc in the associated runlog or checklist. Reanalyze the sample extract once if the above steps failed to reveal or correct the problem. If the surrogate is within limits, report the data without qualifier. If the outlier is confirmed, report the data with the appropriate data qualifier If an error is discovered which cannot be corrected, re-prepare and reanalyze the sample when sufficient sample is available. All samples impacted by the error should be re-extracted. If the reextract confirms, report the original data with a data qualifier indicating confirmed by second analysis.

14. DATA ANALYSIS AND CALCULATIONS

14.1. Calculations can be found in the most current version of the Quality Manual.

14.2. Percent Difference Calculation

$$\text{Percent Difference} = \frac{\text{Cnom} - \text{Ccalc}}{\text{Cnom}} \times 100\%$$

Cnom = True value of the continuing calibration standard
Ccalc = Calculated value of the continuing calibration standard

14.3. Sample Concentration

$$C_s = [(mR_s + b)(V_E)(D)]/V$$

Where:

C_s = Concentration of sample in (µg/L for waters, mg/kg as a dry weight basis for soils)
m = slope of the calibration curve
R_s = GC response of sample in the DRO retention time window
b = intercept of calibration curve
V_E = total volume of sample extract (after concentration) in mL
D = dilution factor if extract was diluted
V = Initial volume or weight of sample

14.4. The following calculation can be used to calculate the LCS percent recovery (where SampleConc would be equal to 0) or MS recovery where SampleConc is the parent concentration:

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. See tables in section 11 & 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. See tables in section 11 & 13.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. If not specifically listed in the table in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. METHOD PERFORMANCE

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269, Method Detection Limit Studies, or equivalent replacement, for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279, Training and Employee Orientation, or equivalent replacement.

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Results are stored in the QA office.

19. METHOD MODIFICATIONS

19.1. Per WIDNR Release News February 1996, the holding time was extended to 10 days from the date of collection prior to solvent addition for soil extraction.

19.2. DRO Silica Gel Cleanup is a modification of the method.

19.2.1. Internally generated control limits are used for acceptance criteria of silica gel cleanup QC. The most current limits can be found in Epic Pro.

19.3. The lab has established an extraction procedure using 250 mL sample volume for water versus the method defined 1 L sample volume extractions based on an adjusted ratio of sample, reagents and standards according to the following:

19.3.1. 25g NaCl is used for 250 mL initial volumes.

19.3.2. 2 mL of 1:1 Hydrochloric acid (HCl) is preserve bottles prior to sampling.

19.3.3. Samples are extracted using two 15 mL aliquots of methylene chloride.

19.3.4. MPCA has approved this modification for use on all water matrices.

20. INSTRUMENT/EQUIPM ENT MAINTENANCE

20.1. Please refer to the GC instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. TROUBLESHOOTING

21.1. If sample emulsions appear handle in one of the following ways:

21.1.1. Allow the emulsion to stand for a longer period of time.

21.1.2. Vigorously shake the separatory funnel containing only the solvent layer, venting frequently.

21.1.3. Centrifuge the emulsion in 250-mL centrifuge tubes with Teflon stoppers for 5 minutes at 1500 rpm.

21.1.4. Add the emulsion to a 150-mL beaker containing a small amount of sodium sulfate and mix with a glass rod.

21.2. If the ICAL or ICV fails, some or all of the following maintenance may need to be performed:

21.2.1. Make a new curve or ICV standard.

21.2.2. Clip column, change gold seal, clean liner, and replace glass wool.

21.2.3. Replace column and guard column.

21.3. CCV failures may be due to the residue that can accumulate from dark extracts. The matrices of these thick dark samples can cause surrogate loss in the injection port resulting in a low bias. Clean the injection port and rerun CCVs if failures occur.

22. SAFETY

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. POLLUTION PREVENTION

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846, 43rd Edition, Final Update III, Method 8000B Gas Chromatography and Chapter One.

25.5. Wisconsin DNR Method For Determining Diesel Range Organics, Method MDRO (Modified DRO); PUBL SW-141, 1995.

25.6. Solvent Addition Holding Time Extended for DRO Analysis. (1996, February). *Wisconsin DNR Release News,* p. 4.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1. Attachment I: Recommended Operating Conditions.

26.2. Attachment II: Buchi Programs

27. REVISIONS

Revision Number	Reason for Change	Date
S-MN-O-466-Rev.24	Table 10.1 – updated ICV Standard Description to Custom CT EPH Standard, Vendor to Phenova AL0-130190. Table 10.2 – updated LL-ICV Stock and DRO-ICV rows Standard Amount column to Custom CT EPH Standard from Accustandard Calibration window defining hydrocarbon. Replaced reference to training SOP with new local SOP number in 18.3. Added NOTE to section 12.2 for state of WI. Added "For WI samples…" exception to Table 13.1, MB row, Corrective Action column.	15Jan2018
S-MN-O-466-Rev.25	Added "(4 oz jar)" to section 12.3.1. 12.3.9 – added "More solvent may be added to cover the sample."	17May2018
S-MN-O-466-Rev.26	Removed NOTE from 12.2, not a method requirement should not have been added previously.	23May2018

ATTACHMENT I: Recommended Operating Conditions

RECOMMENDED CHROMATOGRAPHIC CONDITIONS

Note: Instrument programs may be modified or changed based on analysts experience with elution orders, chromatography seen, or necessary bake times for samples analyzed (if carryover is noticed, etc.)

Gas Chromatograph: Hewlett Packard 5890 Series II, 6890, or 7890, equipped with a Hewlett Packard 7673, 7683, or 7693 autosampler interfaced to HP Chemstation which transfers the data to a Target Server for data processing.

Column Conditions: DB-5MS or comparable column bonded phase (0.5 µm film thickness), 30 meter by 0.25 mm ID column for non LTM/MACH door applications.

Column Conditions for LTM/MACH door: 10m X 0.32 mm X 0.5 um

Injection Port: Injection temperature = 250°C, sample injection volume = 1 µL, splitless injection, purge time off 0.00 minute; purge time on 2.0 minute (purge time on at appx 0.2 min for LTM/MACH door). Head pressure ≈6.0 psi. Split flow ≈35 mL/minute.

Oven Temperature Program: Initial temperature @ 60°C for 3 minutes, 20.0/minute. to 320°C; final hold 12 minutes.

Oven Temperature Program for LTM/MACH door: Init temp @ 50°C for 60 seconds, 250°C per minute to 320°C hold 176 sec. GC Oven is held isothermal at 280°C

Detector: Flame Ionization Detection (FID) - Detector temperature 300°C. Air flow ≈350 mL/minute. FID Hydrogen flow 6-8 mL/minute. Makeup flow ≈20 mL/minute. Column flow ≈7.0 mL/minute. H_2 flow ≈30 mL/minute.

ATTACHMENT II: BUCHI PROGRAMS

SIM soil 80/20 Program to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	50 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	29
3	475	475	33
4	400	400	4
5	325	325	3
6	275	275	4
		Total Time	**74**

40 mL of DCM to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	50 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	9
3	475	475	11
4	400	400	2
5	300	300	4
6	225	225	3
		Total Time	**30**

60 mL of DCM to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	60 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	15
3	475	475	12
4	400	400	2
5	300	300	4
6	225	225	3
		Total Time	**37**

ATTACHMENT II: BUCHI PROGRAMS (cont.)

80 mL of DCM to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	50 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	21
3	475	475	15
4	400	400	2
5	300	300	4
6	225	225	3
		Total Time	**46**

100 mL of DCM to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	50 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	24
3	475	475	22
4	400	400	2
5	300	300	4
6	225	225	3
		Total Time	**56**

120 mL of DCM to exactly 1 mL (-0.3 mL)

Platform Temperature	50 °C
Coolant Temperature	10 °C
Vacuum Cover Temperature	50 °C
RPM	250

Gradient Program

Step	P_1 (mbar)	P_2 (mbar)	T (min)
1	1000	520	1
2	520	520	29
3	475	475	33
4	400	400	2
5	300	300	4
6	225	225	3
		Total Time	**72**

Document Information

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SPECIFIC AROMATIC COMPOUNDS AND GASOLINE RANGE ORGANICS IN WATERS AND SOILS

Reference Methods: EPA SW846 Method 8021B/Wisconsin Modified GRO

Local SOP Number:	S-MN-O-427-rev.23
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-427-rev.22

APPROVALS

Laboratory Assistant General Manager — Date: 12 Jul 2018

Laboratory Quality Manager — Date: 12 Jul 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-427-Rev.23

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to define a purge and trap gas chromatographic method for analysis of 10 individual compounds and gasoline range organics (GRO) in drinking water, ground water, wastewater, soils, and solids as delineated in EPA method SW846 8021B and WI GRO.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline, stoddard solvents, and mineral spirits. Samples are analyzed utilizing purge and trap (P&T) sample concentration. Volatile organic compounds are volatilized by purging an inert gas, nitrogen or helium, through a 5 mL water sample. The vapor is then swept through a sorbent tube where the volatiles are trapped. When the purging is complete, the trap is heated and backflushed with inert gas to desorb the volatiles onto a chromatographic column. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved by a flame ionization detector (FID) for the GRO range. Detection for PVOCs is achieved by a photoionization detector (PID) which is in series with the FID detector. Quantitation is based on PID/FID tandem detector response to a gasoline component standard.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to measuring the concentration of gasoline range organics and the individual components in water and soil. This corresponds to a hydrocarbon range of C6-C10 and a boiling point range between approximately 60°C to 220°C. As defined in the method, other organic compounds, including chlorinated solvents, ketones, ethers, mineral spirits, Stoddard solvents, and napthas are measurable. GRO results include these compounds/products. This method can be used to determine GRO and petroleum volatile organic compounds (PVOCs) concurrently.

4. Applicable Matrices

4.1. This SOP is applicable to drinking water, ground water, wastewater, soils, and solids.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) ranges from 1 ug/L to 100 ug/L for waters and 0.05 mg/Kg to 10 mg/Kg for these methods: see Table 2 in the Attachment section for the individual analyte breakdown. All current MDLs are listed in the Laboratory Information Management System (LIMS) and are available upon request from the Quality Assurance Office.

6. Interferences

6.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants.

6.2. Samples can be contaminated by diffusion of volatile organic compounds through the sample vial septum or between the vial and septum interface A trip blank is prepared using HPLC grade, organic-free, water (or pre-tested, boiled DI water) and carried through the sampling and handling protocol or pre tested, boiled, deionized water can serve as a check on such contamination. Trip blanks may also be purchased premade, refer to the Bottle Preparation SOP, S-MN-C-003, or equivalent replacement.

Laboratory contamination is monitored by analyzing cooler blanks per SOP S-MN-Q-263, or equivalent replacement.

6.3. Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing of the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more method blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with methanol, rinse it with organic-free reagent water, and then dry the purging device in an oven less than 120°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning, typically a methanol back flush followed by a DI water back flush. Screening the sample prior to analysis is recommended to prevent system contamination. This is especially true for soil and waste samples.

6.4. The retention time window definition (methyl-tertiary-butyl ether to naphthalene) introduces a negative bias of approximately 25%. This bias may be greater for weathered samples particularly, low level samples. The lab needs to report peaks detected outside the window so contamination outside the window is not missed. Note that gasoline blends often contain 10% ethanol which could be responsible for a portion of the negative bias.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Three 40mL capped vials (actual volume equals 42 mL with no headspace). The size of any bubble should be less than 6mm. Even though a minimal bubble is allowed, these vials should not be utilized unless no vials without headspace exist. If a vial is used that does have headspace, this should be footnoted in Horizon.	Preserved at pH < 2. For volatiles, pH is measured after analysis and recorded in the daily sequence log. If the pH is greater than two, sample results must be flagged.	Above freezing but below 6°C	If samples are properly preserved at pH < 2, they must be analyzed within 14 days from collection. If the pH > 2, sample results must be analyzed within 7 days for unpreserved samples for 8021. For WI GRO, if the pH > 2, sample results must be flagged indicating pH > 2.
Solid/Soil Samples	Tared VOA vials and must be preserved immediately in the field with methanol.	Methanol preservation is mandatory for the Modified WI GRO method. See section 7.1.1 if they are not preserved.	Above freezing but below 6°C	For EPA 8021B/WI GRO soils is 14 days from collection. If WI GRO only, the holding time is defined by the WIGRO method as 21 days. If samples analyzed for outside recommend holding time the data must be footnoted appropriately.

7.1.1. GRO Soil samples that arrive without methanol should be rejected.

7.1.1.1. Notify the project manager using Lab Track or email so the client may be notified.

7.1.1.2. If the client indicates to proceed with analysis, the data must be flagged.

7.1.2. Samples cannot be analyzed if the amount of soil in the vial exceeds the weight maxima listed in Table 3 for WIGRO soil samples.

7.1.2.1. If the client requests analysis on soil samples which exceed the maxima, the data must be flagged accordingly.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Autosampler	The purging chamber is designed to accept 5mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap should be minimized. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3mm at the origin. The purge gas must be introduced no more than 5mm from the base of the water column. WIGRO requires that samples be purged for 11 minutes.	Varian Archon 5100, EST Archon 8100, or Centurion (or equivalent)
Sample Concentrator	Purge-and-trap device	Tekmar (Lab Sample Concentrator) LSC 3100, LSC 3000 or equivalent
Gas chromatographs	With temperature programming. Equivalent gas chromatographs may be used as long as method performance objectives are met	Hewlett Packard 5890 or 6890
Chemstation	Data Acquisition Software	See master list for current version
Target	Data processing software	See master list for current version
Horizon	Data reporting software	See master list for current version
Gandalf	Data packaging software	See master list for current version
Detector	Detector temperature is 220°C. Detector ranges and lamp intensity are adjusted to provide appropriate sensitivity to achieve the MDL and linearity through the calibration range.	O.I. 4450 PID/FID Tandem detectors (or equivalent)
Column	Restek Rtx-VGC - 30 m x 0.25 mm ID, 1.4 µm film thickness (or equivalent), or DB-624 – 30m x .53mm ID, 1 mm film thickness (or equivalent)	Restek
Microsyringes	10, 25, 50, 100, 250, 500, and 1000 µL	Fisher Scientific or equivalent
Syringes	5, 10, 25mL or 50mL, gas-tight with shutoff valve	Fisher Scientific or equivalent
Eppendorf Pippetter	1000 µL	Fisher Scientific or equivalent
Balance	Analytical, 0.0001g, and top-loading, 0.01g	Fisher Scientific or equivalent
Clear glass vials	2mL, with Teflon lined screw-caps	Fisher Scientific C40131500 or equivalent replacement
Disposable pipettes	Pasteur	Fisher Scientific or equivalent
Volumetric flasks	Class A - 5mL, 10mL, 25mL, 50mL, and 100mL, 200mL, 250mL, 500 mL, and 1000mL with ground-glass stoppers	Fisher Scientific or equivalent
pH paper	Wide Range	Whatman 2613-991
Spatula	Stainless Steel or equivalent	Fisher Scientific or equivalent
Desorber	Should be capable of rapidly heating the trap to the manufacturer's recommended temperature for desorption, typically 180°C to 260°C, depending on the trap chosen.	Tekmar LSC-3100, LSC 3000 or equivalent
Trap packing	A variety of traps are available from manufacturers. Any of these traps may be used if	Some traps used include, but are not limited to a Tenax/silica

	the trap packing materials do not introduce contaminants into the analysis and the data generated using the trap meets the initial and continuing calibration technical acceptance criteria of this method.	gel/carbon trap, a Tenax/silica gel/carbon/OV-1 trap, and a Vocarb 3000 trap
VOA Vials	40 mL VOA Vials (actual volume = 42 mL)	C&G Unpreserved Vials NC9879693 or equivalent replacement

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
Organic-free Water (OFW)	De-ionized water (DI water may be boiled or purged to further remove volatile contaminants)	Verify that background levels of volatile compounds are acceptable by analysis
GRO free sand		
Methanol, CH_3OH	CH_3OH - Fisher Purge and Trap grade or equivalent, demonstrated to be free of analytes. Store apart from other solvents.	Fisher Scientific A453-1 or equivalent replacement
Custom Modified GRO Component Standard (Stock ICAL standard)	A typical vendor stock solution will have each of the PVOC compounds found in Table 2 and which will compose the total GRO concentration. 10,000 mg/L	O_2Si or equivalent, 122014-02-02
Custom Modified GRO Second Source (Stock ICV Standard)	2,000 mg/L	O_2Si or equivalent, 122014-03-02-SS
α,α,α -Trifluorotoluene(TFT)	2,500 mg/L	O_2Si or equivalent, 020133-05
1-Chloro-3-fluorobenzene	5,000 mg/L	O_2Si or equivalent, 020912-05-02

10.1.1. After a portion of stock standard has been used, transfer any remaining stock standard solution into a clear bottle with a Teflon lined screw-cap or crimp cap vial or mininert valves. Store, with minimal headspace, at manufacturers listed conditions and protect from light. This unused portion is only good for 6 months from the date that the ampule is opened.

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Surrogate Standard.	α,α,α – Trifluorotoluene (TFT) Stock	1 mL	Methanol	24 mL	25 mL	100 µg/mL
Internal Standard.	1-Chloro-3-fluorobenzene	0.5 mL		24.5 mL	25 mL	100 µg/mL
ICAL/CCV Working Standard	Custom Modified GRO stock standard	0.5 mL		4.5 mL	5 mL	*100/1000 µg/mL *individual components
ICV standard intermediate	Custom Modified GRO second Source stock standard	0.5 mL		9.5 mL	10 mL	100 µg/mL
Calibration Std 1	GRO/PVOC Working Standard at 100 ug/mL	0.4 µL		99.9996 mL	100 mL	0.4 µg/L for single components/4 µg/L for GRO
Calibration Std 2	GRO/PVOC Working Standard at 100 ug/mL	1.0 µL		99.999 mL	100 mL	1.0 µg/L for single components/ 10 µg/L for GRO

Calibration Std 3	GRO/PVOC Working Standard at 100 ug/mL	5.0 µL	Water	99.995 mL	100 mL	5.0 µg/L for single components/ 50 µg/L for GRO
Calibration Std 4	GRO/PVOC Working Standard at 100 ug/mL	10 µL		99.9 mL	100 mL	10 µg/L for single componenets/ 100 µg/L for GRO
Calibration Std 5	GRO/PVOC Working Standard at 100 ug/mL	50 µL		99.95 mL	100 mL	50 µg/L for single components/ 500 µg/L for GRO
Calibration Std 6	GRO/PVOC Working Standard at 100 ug/mL	100 µL		99.9 mL	100 mL	100 µg/L for single components/ 1000 µg/L for GRO
Calibration Std 7	GRO/PVOC Working Standard at 100 ug/mL	300 µL		99.7 mL	100 mL	300 µg/L for single components/ 3000 µg/L for GRO
Calibration Std 8	GRO/PVOC Working Standard at 100 ug/mL	500 µL		99.5 mL	100 mL	500 µg/L for single components/ 5000 µg/L for GRO
Initial Calibration Verification Standard (ICV) (Second Source)	ICV Intermediate Standard	100 µL		99.9 mL	100 mL	100 µg/L for single components/ 1000 µg/L for GRO
Continuing Calibration Verification Standard and LCS	ICAL/CCV Working Standard	250 µL		249.75 mL	250 mL	100 µg/L for single components/ 1000 µg/L for GRO
Spiking Standard for MS/MSD	ICAL/CCV Working Standard	42 µL	Parent sample vial (~42 mL VOA vial collected in the field)	42 mL	42 mL	100 µg/L for single components/ 1000 µg/L for GRO

10.2.1. Intermediate or working standards - Using stock standard solutions, prepare in purge and trap grade methanol working standards containing the compounds of interest, either singly or mixed together. Working standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Internal standards and surrogate solution should be prepared every three months or sooner.

10.2.2. If multiple mixes are used, the expiration date for the final solution MUST NOT exceed the earliest expiration date of any of the parents, or constituents.

10.2.3. The surrogate standard and internal standard may be combined into one mix for spiking samples if desired. Note the centurion autosampler is set to spike for 30ms and the concentration of the working internal standard solution and surrogate solution need to be made at a lower concentration due to the fact that the centurion autosampler adds for 30ms.

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Calibration Curve Fit	Average	≤20% RSD	A minimum of a 5-point-curve is required, 6 points

	Linear Regression Quadratic	≥0.995 ≥0.990	are required if a quadratic curve fit is utilized. One level must be at or below the reporting limit.
Retention time	Evaluate daily utilizing the continuing calibration verification	GRO retention time window is 0.1 minute prior to MTBE and ending 0.1 minute after Naphthalene. The retention time shift of the internal standards is verified to be within ±30 seconds	Account for drift by adjusting the windows. See 11.2.5 and 11.2.6
Second Source Verification Standard (ICV)	A second source standard must be analyzed to verify an initial calibration curve which contains all the analytes of interest. The spike level of the ICV should be near the midpoint level of the calibration curve.	The second source standard must meet the criteria specified in S-All-Q-025 (or equivalent replacement), Standard Traceability SOP, for the initial calibration to be verified.	If the first ICV fails, the analyst should try to determine the root cause for the failure. Verify the standard preparation. The ICV can be reanalyzed one additional time. If it still fails the system must be recalibrated and verified prior to sample analysis.
Reporting Limit	The State of Minnesota requires the reporting limit to be verified upon initial calibration and every 30 days.	± 40% of the true value for samples originating in MN	Evaluate the curve fit. Evaluate the data quality objectives of the projects reporting. If possible adjust the reporting limit to the next standard that meets criteria or recalibrate to pass the criteria.
Continuing Calibration Verification (CCV). See section 11.2.9.	The initial calibration curve for each compound of interest must be checked and verified before samples are to be run. This is accomplished by analyzing a continuing calibration verification (CCV) standard near a midpoint level of calibration curve. At a minimum a CCV must be ran at the beginning of each batch and a bracketing CCV must run at the end. Additional CCVs may be run throughout the 12 hour shift. If a quadratic curve is utilized, WI originating samples must be bracketed by two different CCV concentrations to show that there is not bias due to the quadratic curve fit.	The CCV for PVOCs must have a % difference less than 15% of the known concentration and GRO must be less than 20%. All CCVs must pass the required criteria, unless the CCV was above the acceptable criteria and samples were non-detected for the analyte which exceeded the criteria.	If the first CCV fails, the analyst should try to determine the root cause for the failure. Some possible reasons for failures may include but not limited to: bad CCV solution, bad spike of CCV standard, standard mix degradation, internal standard fluctuation/change, analytical system not conditioned, active sites and/or cold sites in the trap or concentrator, contaminated system due to dirty samples, or analytical conditions changed over time. Corrective action for a failed CCV will be a case – by-case depending on the root cause. The analyst's expertise in determining the root cause will help determine the corrective action. Some common

			corrective actions may include but not limited to: making a new CCV solution, running a different vial of a CCV solution, using a different standard to make the CCV solution, making new standards and new CCVs, baking out the concentrator, or replacing transfer lines on concentrator. For volatiles there is generally no major maintenance performed on a daily basis to maintain calibration. If major maintenance is required, the system then needs to be recalibrated. If the 2nd CCV fails to meet the criteria, a new initial calibration curve must be performed

11.2. Initial calibration for purge-and-trap procedure

11.2.1. All standards, blanks, spikes, and samples must be analyzed using the same conditions. A set of at least five calibration standards containing the method analytes and surrogates is needed (six standards are necessary for quadratic curve fits). One calibration standard should contain each analyte at a concentration at or below the reporting limit for that compound; the other calibration standards should contain analytes at concentration that define the range of the method.

11.2.2. To prepare a calibration standard, add an appropriate volume of standard solution to organic-free reagent water in a volumetric flask. Using a microsyringe, rapidly inject the standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times. Transfer the standard to a 40 mL VOA vial and load into the Autosampler. If ICAL or CCVs are not used immediately they made to stored at 0 to 6 degrees Celsius in a cooler which does not house samples for one day from the day they were made.

11.2.3. The ICAL defined in Table 10.1 is an example of standard preparation for an initial calibration for 8021B. Standard preparation is determined by client and project requirements. A "soil" curve to reflect the chromatography conditions of medium level soils (1:50 ratio of MeOH:Water) is prepared by adding 2mL of methanol into the calibration standards and reducing the volume of reagent water accordingly. The 0.4 ug/L or 0.02 mg/Kg standard is only required when the reporting limit is required for a client data quality objective.

NOTE: As long as there is separation between the methanol peak and the start of the GRO range for methanol diluted samples, the ICAL does not need methanol added.

11.2.4. The calibration curves for the individual compounds on the PID are constructed utilizing the Internal Standard calculation procedure as shown in Section 14 at the concentration listed in Table 10.2.

11.2.5. The retention time windows for PID compounds are determined as follows:

11.2.5.1. The retention time shift of the internal standards is verified. The retention time shift between the initial and subsequent standards must be $< \pm 30$ seconds. If this is not met, continue injecting replicated standards to meet this criterion until the criteria is met.

11.2.5.2. The standard deviation of the absolute retention times is calculated for each analyte of interest.

11.2.5.3. The standard deviations determined in 11.2.5.2 shall be used to determine the retention time windows for a particular run sequence. Plus or minus three times the standard deviations in 11.2.5.2 is applied to the retention times of each analyte of interest (from the daily calibration check). This range of retention time defines the retention time window for the compound of interest.

11.2.5.4. In cases where the retention time window is less than 0.01 minutes, use ± 1.0% of the retention time of the daily calibration check standard to define the retention time window.

11.2.6. Retention Time Window and Quantitation for GRO

11.2.6.1. Quantitation of GRO is performed by the external standard method. The concentration of Gasoline Range Organics in the sample is determined from a summation of the total response within the retention time window for GRO. No area may be subtracted from the GRO retention time window in calculating GRO results.

11.2.6.2. The retention time window for GRO analysis is defined as starting 0.1 minutes prior to the methyl-tert-butyl ether (MTBE) peak and ending 0.1 minutes after the Naphthalene peak (see Figure 1).

11.2.6.3. Evaluate continuing calibrations daily for any retention time drift and account for any drift by adjusting the window.

11.2.6.4. Integration must be "baseline to baseline" and is defined as a flat baseline drawn parallel to the x-axis of the chromatogram that includes all responses within the retention time window. The correct baseline placement would be a horizontal line drawn through the lowest point in the chromatogram. See Figure 1 for examples.

11.2.7. Refer to the Pace Analytical Quality Manual for initial calibration curve formulas.

11.2.8. When calibrating for WIGRO water and soil samples, a constant concentration of surrogate (not to exceed 20 µg/L) must be added to all samples and all calibration standards

11.2.9. Continuing Calibration Verification (CCV)

11.2.9.1. For waters, the CCV and LCS/LCSD solutions are the same solution (prepared the same, using the same standards, etc.). The CCV and LCS/LCSD analyses are interchangeable and can be used for both sample types (the CCV can also be used as the LCS and the LCS can also be used as the CCV) provided that 2 CCV's didn't already fail in a row (which would trigger an initial calibration). When these analytical runs are used as the same file, they should be named as 2 separate files to distinguish the sample types for reporting the appropriate reports and so that there is a unique file name associated with each sample type.

11.2.9.2. For medium level soils, LCS/LCSD's are not interchangeable with the CCV's in the run sequences as the LCS/LCSD's are prepared and extracted with the associated sample on the day of preparation while the CCV's and initial calibration solutions are prepared by using a ratio of 1 mL methanol into 50 mL of DI water (to matrix match the calibrations to the sample matrix) and are not extracted.

11.2.10. Internal standards should be monitored against the internal standard responses and retention times from the daily continuing calibration verification. If the retention time for any internal standard changes by more than 30 seconds and the responses changes by a factor of two (-50% to +100%), the chromatographic system should be inspected for malfunctions and corrections should be made. If, in the interpretation of an experienced analyst, an outlier is due to matrix or other contributing issue, data may be utilized.

11.2.10.1. Internal standard recoveries out low (high bias) – if compounds associated with the internal standard(s) that are outside the control limits are non-detect, the sample can be

reported without re-analysis, however, if the outlier is not indicative of a system drift (i.e. If only one sample has internal standard drift, which is dissimilar from other samples around the injection time), re-analysis should be performed to rule out matrix effects.

11.2.10.2. Internal standard recoveries out high (low bias) – re-analysis should be performed assuming there is sufficient sample volume remaining. Appropriate footnoting practices are also observed.

11.3. INITIAL CALIBRATION VERFICATION (ICV)

11.3.1. To ensure internal standard recoveries from samples that run after the Ical under the same folder go off the Ical and not the ICV, process the ICV as a sample. To see if the ICV meets passing requirements pull the ICV file into a batch.b along with the method and requant the ICV as a continuing calibration.

11.4. CONTINUING CALIBRATION VERIFICATION (CCV)

11.4.1. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition.

11.4.2. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration, the analytical system must be inspected for malfunctions and corrections must be made.

11.4.3. If the EICP area for any of the internal standards changes by a factor of two, (-50% to +100%) from the last daily calibration standard check, the system must be inspected for malfunctions and corrections must be made.

12. Procedure

12.1. GC analysis

12.1.1. Water samples

12.1.1.1. Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Screening can be accomplished by using a headspace GC PID or by analyzing the sample at a dilution by GC.

12.1.1.2. The original zero headspace sample vial is placed into the autosampler tray and the Archon 5100 or EST 8100, or equivalent Autosampler spikes the internal standard (IS) and surrogate (SS) mix. If a dilution is required, the necessary sample volume is manually diluted and placed into the Archon 5100 or EST 8100 Autosampler.

12.1.1.3. The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample vial is sealed.

12.1.1.3.1. Dilutions may be made in volumetric flasks of various sizes. Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

12.1.1.3.2. Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask. See table 5 for common dilution factors.

12.1.1.3.3. Inject the proper aliquot of sample into the flask. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert three times. Once sample dilution is completed, the pH of the un-diluted sample must be taken with pH paper. If the pH is greater than 2 the sample must be footnoted. Repeat above procedure for additional dilutions.

12.1.1.3.4. Fill the vial with diluted sample and load onto the autosampler.

12.1.1.3.5. The autosampler adds the internal standard spiking solution and the surrogate spiking solution to the 5mL sample aliquot. The amount added by the autosampler should be equivalent to the concentration of 20 µg/L of the surrogate and the internal standard. Note the centurion autosampler is set to spike for 30ms and the concentration of the working internal standard solution and surrogate solution need to be made at a lower concentration due to the fact that the centurion autosampler adds for 30ms.

12.1.1.3.6. Analyze the samples using the same autosampler and GC conditions used to pass initial calibration, CCV standard, and blank criteria.

12.1.1.4. If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated peaks, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. Alternately, samples loaded on an autosampler can be accepted after a subsequent sample is shown to be free of carry-over contamination or if the detection is 10x greater than the carryover detection. Carryover in p&t systems can vary for instrument to instrument depending on the condition of the equipment. Analysts review the carryover after the upper level of the initial calibrations and after the ICV, in addition they monitor the carryover daily on the system blanks ran which is generally ran after QC samples. It is common for the laboratory to run multiple blanks after an initial calibration to monitor the carryover and ensure the ICV does not have carryover affecting the % recoveries. Daily, it is common for the laboratory to run system blank before the method blank. This is to help determine if there is a contamination coming from the system itself or if contamination occurred during the sample preparation phase.

12.1.1.5. All samples must be thoroughly reviewed when sample concentrations exceed GRO detection of 500µg/L and 50ug/L for PVOC to ensure low-level carryover is not occurring into subsequent analyses.

12.1.1.5.1. For matrix spike analysis, add 42µL of a 100ug/mL working standard solution to the aqueous sample vial (42 mL actual volume). Disregarding any dilutions, this is equivalent to a concentration of 100 µg/L of each matrix spike standard. Add the spiking solution through the septa of the vial as the vial should not be opened to maintain sample integrity.

12.1.1.5.2. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the calibration range.

12.1.1.5.3. Once sample analysis is completed, the pH of the sample must be taken with pH paper and recorded in the instrument run logbook. If the pH is greater than 2 the sample must be footnoted.

12.1.1.5.4. In addition to reporting the GRO range the chromatogram must be evaluated for detections previous to or following the GRO range. Any area response attributed to analyte detections must result in qualifying the data with either low boiler detection (before the GRO range) or high boiler detection (after the GRO range).

12.1.2. Sediment/soil and waste samples - For medium level soils (MLS) the Pace label is placed on all containers during the log-in process which is performed by sample receiving. The weight of the label is subtracted from all soil samples to reflect the weight of the soil weight. The weight of the label is determined annually (unless specified) by weighing out 10 labels and determining an average weight. This subtracting of the label weight is performed in the soil prep logbook. It is recommended that all samples of this type be screened prior to analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and trap system, and require extensive cleanup and instrument downtime.

12.1.3. A sample is either extracted or diluted with methanol, depending on its solubility. An aliquot of the extract is added to organic-free reagent water. This is purged at ambient temperature.

12.1.4. NOTE: The following steps must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

12.1.5. To prepare the laboratory method blank, laboratory control sample and duplicate weigh out 10 grams of Ottawa sand and add 10mL of methanol to a 40 mL VOA vial (42 mL actual volume). They should be uniquely labeled by QC batch numbers to ensure they are analyzed with the correct batch of samples. The weight of the BLK, LCS,LCSD is record as 10 grams so long as the actual weight is 10+/-0.1grams. The LCS/LCSD are also spiked with the 50µL of 1000 µg/mL of the UST Modified GRO stock standard to achieve a final concentration of 100 µg/L after the 1:50 dilution.

12.1.6. To prepare the matrix spike and matrix spike duplicate, weigh the vial and record the weight to the nearest 0.01g. Subtract the vial weight prior to sampling and record the final weight. If the weight is greater than the expected 5g, 10g, or 25g weight, the addition of methanol is necessary in order to maintain the 1:1 sample to solvent ratio. Add the appropriate amount of 1000 µg/mL stock standard to achieve a final concentration of 100 µg/L after the 1:50 dilution. If insufficient sample volume was received to prepare the MS/MSD, the project must be footnoted and the project manager should be notified.

12.1.7. To prepare samples that arrive preserved in methanol, weigh the vial and record to the nearest 0.01g. Subtract the vial weight prior to sampling and record the final weight. If the weight is greater than the expected 5g, 10g, or 25g weight, the addition of methanol is necessary in order to maintain the 1:1 sample to solvent ratio. If the weight is less than expected 5g, 10g or 25g weight, record the difference and in the soil prep logbook and Horizon prep batch.

12.1.8. To prepare samples that are not preserved in methanol the sample consists of entire contents of sample container. Using a top-loading balance, weigh 10 grams (wet weight) of the sample into a tared 40 mL vial. Record the weight to 0.01g. Quickly add 10 mL of methanol. Samples not field preserved should be preserved within 48 hours of collection. Client, QAPP, or state requirements may supersede this requirement.

12.1.9. Oily, solid waste or product samples are generally not field preserved due to the unknown solubility. If the sample is not soluble in water, a waste dilution will be performed by weighing out 1gram of the sample into a tared 40mL VOA vial. Record the weight to 0.01 grams. Quickly add 10 mL of methanol and 10 µL of 2500 µg/mL surrogate standard may be added to achieve a final concentration of 50 µg/mL after the 1:50 dilution.

12.1.10. The LCS/LCSD, MS/MSD, method blank, and all associated samples within the batch must be shaken for two minutes, then sonicated for 20 minutes. After sonicating, prepare the samples by adding 1000 µL either by syringe or Eppendorf pipette of the methanol extract to a 50 mL volumetric flask containing DI water. Dilute to a final volume of 1:50 using DI water. Fill a 40 mL VOA vial with the prepared sample for analysis.

12.1.11. NOTE: Samples analyzed for 8021B and AK101 should not be sonicated, AK101 analysis is outlined in S-MN-O-556, or equivalent replacement.

12.1.12. The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample vial is sealed.

12.1.12.1. Dilutions may be made in volumetric flasks of various sizes. Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

12.1.12.2. Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask. See tables 5 and 6 for common dilution factors.

12.1.12.3. Inject the proper aliquot of sample extract into the flask and the proper amount of P&T methanol, so that the same amount of methanol is added to all samples and QC. Dilute

the sample to the mark with organic-free reagent water. Cap the flask and invert three times.

12.1.12.4. A single mix of internal standard and surrogate may be spiked by the autosampler if the surrogate is not spiked prior to extraction.

12.1.13. The extracts must be stored above freezing but below 6°C.

12.1.14. Methanol Correction: This will only be performed if specifically requested by a regulatory agency or by the client. When methanol correction is requested, the volume needs to be adjusted for the amount of soil moisture present in the solid sample. This is done through Horizon once the % moisture analysis has been performed. The analyst enters the sample weight and volume into the prep batch in Horizon. The condition code needs to be changed to "mc" for methanol corrected instead of the standard "ok". Once this is done, Horizon will perform the calculation. The final volume will change in Horizon to reflect the methanol correction. Note: the lab will spike the surrogate solution (if applicable) based on the known actual volume methanol in the sample, not the methanol corrected volume. Depending on the % moisture in the sample(s) the lab has seen circumstances where the methanol corrected volume is artificially higher than it visually appears. It is not uncommon to have the surrogate recoveries fail once moisture is corrected. The lab will not re-run for surrogate confirmation if non-methanol corrected surrogate recoveries are within control limits.

12.1.14.1 Methanol Correction Formula:
Methanol Corrected volume = Volume of methanol (mL) + ((% moisture* sample weight (g))/100)
The methanol corrected volume obtained becomes Vt in the calculation in section 14.6.

12.1.15 When SW8021 is requested without WIGRO, the detections found for 8021 require confirmation. For 8021 requested analysis, all positive detections will be confirmed by 8260.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Free of analytes of interest. Corrective action is taken when the concentration of any target analytes is detected above the reporting limit and is greater than 1/10 of the amount of the analyte found in any associated sample. Due to client requirements, the lab may have to evaluate to ½ RL or MDL based on those data quality objectives. DoD requires the evaluation to ½ RL and WI DNR to the MDL. No corrective action is taken for detections found below ½ RL beyond data qualification.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.

Laboratory Control Sample (LCS) and Laboratory Control Spike Duplicate (LCSD)	DI water spiked with all target compounds	One per 20 samples for 8021; WI GRO requires LCS and LCSD	80-120% %RPD: 20%	Analyze a new LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run. For WI GRO both the LCS and LCSD must pass criteria, failing RPD criteria should be investigated the same as a recovery failure. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS/MSD)	Client sample spiked with all target compounds Matrix spikes will be performed when more than three containers (except soil received in packed jars) have been received to ensure that sufficient sample remains for any re-analysis (i.e., dilutions, instrument problems). An MS and sample duplicate (DUP) will be analyzed if insufficient sample is received.	One per 20 samples if sufficient sample volume Batches with WI samples must have MS/MSD – see 13.2.	80-120% RPD: 20%	If there is an outlier, the corresponding laboratory control spikes must be evaluated. If the same outlier occurs in the LCS, a system problem may be assumed. In this instance, samples may have to be reanalyzed. If the LCS reports an acceptable recovery, matrix interferences are assumed. In either instance, the data on the final report must be footnoted with the outliers and possible reason if known. When contamination is suspected in the sample provided for the OQS, a MS and a DUP may be performed instead of a MS/MSD to result in a more representative determination of method precision. Client, QAPP, or state requirements may supersede this. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately
Surrogate	α,α,α – Trifluorotoluene (TFT) For WI GRO samples, no more than one surrogate and one internal standard can be used to avoid co-elution interferences within the GRO	All samples and QC samples must be evaluated for surrogate % recoveries.	80-150%; The laboratory updates surrogate recovery limits on a matrix-by-matrix basis, annually	Check the calculations, surrogate solutions or internal standards to be sure there are no errors. Recalculate accordingly if error exists. Check instrument performance. If problem is identified, correct the problem and reanalyze the sample. If no problem is found, re-analyze the sample. If surrogate recovery is high due to matrix, determine if the sample needs

	window, and a constant surrogate concentration must be used which is not to exceed 20 µg/L.			to be re-run. If still out of limit, report the initial run and footnote appropriately. If the recovery is now within limits, report the second run (if within holding time). If the methanol preserved soil is methanol corrected see section 12.1.4 for outliers details. If surrogate recovery is high and all associated analytes of interest are non-detect (a positive bias), the sample does not need to be re-run. Flag the data with the appropriate footnote.

13.2. If there is insufficient sample volume for a batch containing WI samples for MS/MSD then the analyst will have to ask the PM that has the WI sample in the batch for a QC replacement holder. The analyst will add this to the batch and pair a MS/MSD with it. The analyst can use any sample in the cooler that has extra volume. The one 40 mL VOA vial sample will be split three ways. Raised reporting limits are acceptable for the QC replacement holder and MS/MSD.

14. Data Analysis and Calculations

14.1 The Pace Quality Manual contains several example calculations in addition to those expressed below.

14.2 Calculations for the PVOC compounds are performed by the internal standard procedure utilizing 1-Chloro-3-fluorobenzene as the internal standard for the individual aromatics analyses.

14.3 The equations used to calculate the absolute amount of a component (y) are:

14.4 For a single level calibration the equations are of the form:

$$RRF(y) = \frac{\text{Area (y)} * \text{Amount (I)}}{\text{Amount (y)} * \text{Area (I)}}$$

Where:

y = Any calibrant peak or group of peaks

I = An Internal Standard peak

RRF = The relative response factor for peak y.

Area = The peak area of calibrant or Internal Standard I in the standard.

14.5 Quantitative Analysis: When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used should be the one nearest the retention time of that of a given analyte or as specified in the method.

14.6 Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste: Equation 6

$$\text{Concentration(mg/L)} = \frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)}$$

Where:

$\mathbf{A_x}$ = Area of characteristic ion for compound being measured.
$\mathbf{I_s}$ = Amount of internal standard injected (mg/L).
$\mathbf{A_{is}}$ = Area of characteristic ion for the internal standard.
RRF = Average Relative Response factor for compound being measured.

DF = Dilution Factor

Sediment/Soil, Sludge, and Waste: Equation 7

$$High\ \text{Conc.}\ (\mu\text{g/kg}) = \left[\frac{(A_x)(I_s)(DF)(V_t)}{(A_{is})(RRF)(W_s)}\right] \bullet 50$$

Equation 8

$$Low\ \text{Conc.}\ (\mu\text{g/kg}) = \frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)}$$

Where:

A_x, I_s, A_{is}, RRF = See Equation 6 (above) for definitions
V_t = Volume of total extract (mL)
V_i = Volume of extract added (mL) for purging
W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.
DF = Dilution factor
Note: All methanol extracts are diluted 1:50

14.7 The actual concentrations of sample components are calculated by the data system using the equations of the best fit of straight line through the points of the initial calibration.

14.8 The calibration plot for GRO is constructed by external standard calibration, using the area sum for all of the components of the GRO standard from the FID. The GRO concentration is a comparison of the total peak area from the sample between the MTBE and naphthalene retention times to the linear plot of the GRO calibration standards. This concentration is calculated by the data system.

14.9 The concentration of gasoline in aqueous samples is calculated utilizing the following equation:

$$Amount_{(y)} = \frac{\left(Area_{(y)}\right)(DF)}{\left(RF_{(y)}\right)}$$

where:

y = Any calibrant peak or group of peaks
$Area_{(y)}$ = peak area of y in the sample
DF = Dilution factor
$RF_{(y)}$ = The response factor for peak y
Amount = Concentration of *y* in the sample (µg/L)

Medium Level Soil Concentration

$$\text{Medium Level Conc(y)}\ (mg/kg) = \left[\frac{(A_y)(DF)(V_t)}{(RF_{(y)})(W_s)}\right] \bullet 50$$

Where:

y = Any calibrant peak or group of peaks
$A_{(y)}$ = Peak area of y in the sample
DF = Dilution factor
$RF_{(y)}$ = The response factor for peak y
V_t = Volume of total extract (mL)
W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See table in section 11 & 13.

16 Corrective Actions for Out-of-Control Data

16.1 See table in section 11 & 13.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in the table in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18 Method Performance

18.1 All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2 **Method Detection Limit (MDL)** Study: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4 **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19 Method Modifications

19.1 As this SOP is written for both EPA Method 8021 and Modified WI GRO, PVOC analytes are allowed to be reported under WI GRO without second column confirmation per the method. Only the samples that are received requesting 8021 that will require confirmation as indicated in 12.1.15.

20 Instrument/Equipment Maintenance

20.1 Please refer to the 6890 or 5890 gas chromatograph instrument manual for maintenance procedures performed by the lab.

20.2 All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21 Troubleshooting

21.1 The purge and trap concentrator must be leak free in order to ensure properly sample purge efficiency and desorbtion. If analyst notices significant decrease in response and suspects a possible leak, one can leak check the concentrator to ensure the p&t concentrator is free for leaks. This can be done through the software or manually by capping the vent valve of the concentrator and purging a blank.

22 Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24 Pollution Prevention

24.1 The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25 References

25.1 Wisconsin DNR, Modified GRO Method for Determining Gasoline Range Organics, September 1995; PUBL-SW-140 and subsequent updates as posted in the WI-DNR newsletters

25.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, Method 8021B-95

25.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 8260B-95.

25.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 5035-95.

25.5 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 5030B-95.

25.6 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, Method 8000B-96.

25.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4rd Edition, Final Update IV, Method 8260C-06.

25.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4rd Edition, Final Update IV, Method 5035A-02.

25.9 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4rd Edition, Final Update IV, Method 5030C-03.

25.10 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, Method 8000C-03.

25.11 Method 524.3 Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Rev. 1.0" Technical Support Center Office of Ground Water and Drinking water, U.S. Environmental Protection Agency, Cincinnati, Ohio, June 2009

25.12 Pace Quality Assurance Manual- most current version.

25.13 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.14 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

26 Tables, Diagrams, Flowcharts, and Validation Data

26.1 Table 1: Order, CAS Numbers, Quantitation Limit

27 Revisions

Document Number	Reason for Change	Date
	Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Added Section 12.1.1.5.4. Table 13.1: MB row Acceptance Criteria column- added "or MDL" and "and WI DNR to the MDL", MS/MSD row Frequency column added "Batches with WI samples must have MS/MSD – see 13.2" Added "For WI samples…" exception to Table 13.1, MB row, Corrective Action column. Added Section 13.2.	
S-MN-O-427-Rev.22	6.2 – updated to local SOP number instead of corp SOT number.	23Apr2018
S-MN-O-427-Rev.23	Added sections 11.3 and 11.4 and all subsections.	12Jul2018

TABLE 1: Order, CAS numbers, Quantitation Limit

Parameter (in Elution order)	Detector	CAS Number	Quant. Limit ug/L	Quant. Limit mg/Kg
Methyl-tert-butyl-ether	PID	1634-04-4	5.0	0.25
Benzene	PID	71-43-2	1.0	0.05
α,α,α-Trifluorotoluene (SS)	PID	98-08-8		
Toluene	PID	108-88-3	1.0	0.05
1-Chloro-3-Fluorobenzene (IS)	PID	N/A		
Ethylbenzene	PID	100-41-4	1.0	0.05
m-Xylene (coelute) p-Xylene	PID	108-38-3 106-42-6	1.0 1.0	0.1
o-Xylene	PID	95-47-6	1.0	0.05
Xylene, (total)	PID	1330-20-7	3.0	0.15
1,3,5-Trimethylbenzene	PID	108-67-8	1.0	0.05
1,2,4-Trimethylbenzene	PID	95-63-6	1.0	0.05
Napthalene	PID	91-20-3	N/A	N/A
Gasoline Range Organics	FID	N/A	100	10

TABLE 2: Standard Preparation Concentration

Compound	Final Conc. ug/mL
ICAL/CCV Working Standard Concentration	
MTBE	100
Benzene	100
Toluene	100
Ethylbenzene	100
m-xylene	100
p-xylene	100
o-xylene	100
1,3,5-TMB	100
1,2,4-TMB	100
Naphthalene	100
GRO	1000

TABLE 3: Weight Maxima

Vial Size	Target Sample Weight	Actual Sample Weight	Volume of Methanol	Action
40 mls (GRO only)	10 gms	<8 gms	10 mls	Flag
		8-11 gms	10 mls	None
		>11 gms<20 gms	10 mls	Add Methanol
		>20 gms	for any amount	Reject
60 mls	10 gms	<8 gms	10 mls	Flag
		8-11 gms	10 mls	None
		>11 gms<35 gms	10 mls	Add Methanol
	25 gms	<20 gms	25 mls	Flag
		20-26 gms	25 mls	None
		>26 gms<35 gms	25 mls	Add Methanol
		>35 gms	for any amount	Reject
120 mls	10 gms	<8 gms	10 mls	Flag
		8-11 gms	10 mls	None
		>11 gms<70 gms	10 mls	Add Methanol
	25 gms	<20 gms	25 mls	Flag
		20-26 gms	25 mls	None
		>26 gms<70 gms	25 mls	Add Methanol
	50 gms	<40 gms	50 mls	Flag
		40-51 gms	50 mls	None
		>51 gms<70 gms	50 mls	Add Methanol
		>70 gms	for any amount	Reject

Laboratories should use standard rounding rules to determine compliance with the maximum weight requirement. Sample weights should be rounded to the nearest whole number. This means that a sample weighing between 34.5-35.4 is rounded to 35.0 gms, and a sample weighing between 69.5-70.4 gms is rounded to 70.0 gms. There will be NO allowances given past these tolerances.

Table 5: Common Dilution Factors for Waters

Water Dilution Factors		
Dilution	**Into 50 mL**	**Into 100 mL**
2x	25 mL	n/a
5x	10 mL	20 mL
10x	5 mL	10 mL
20x	2.5 mL	5 mL
25x	2 mL	4 mL
50x	1000 uL	2 mL
100x	500 uL	1000 uL
200x	250 uL	500 uL
500x	100 uL	200 uL
1000x	50 uL	100 uL
10000x	5 uL	10 uL

Table 6: Common Dilution Factors for Soils

Soil Dilutions into 50mLVolumetric		
Dilution Factor	Volume of Soil Extract (uL)	Volume of P&T Methanol (uL)
1	1000	0
2	500	500
5	200	800
10	100	900
20	50	950
25	40	960
50	20	980
100	10	990
200	5	995
500	2	998
1000	1	999
Beyond 1000x Serial Dilutions are performed.		

Soil Dilutions into 100mLVolumetric		
Dilution Factor	Volume of Soil Extract (uL)	Volume of P&T Methanol (uL)
1	2000	0
2	1000	1000
5	400	1600
10	200	1800
20	100	1900
25	80	1920
50	40	1960
100	20	1980
200	10	1990
500	4	1996
1000	2	1998
Beyond 1000x Serial Dilutions are performed.		

Figure 1A: Wisconsin GRO Integration Samples

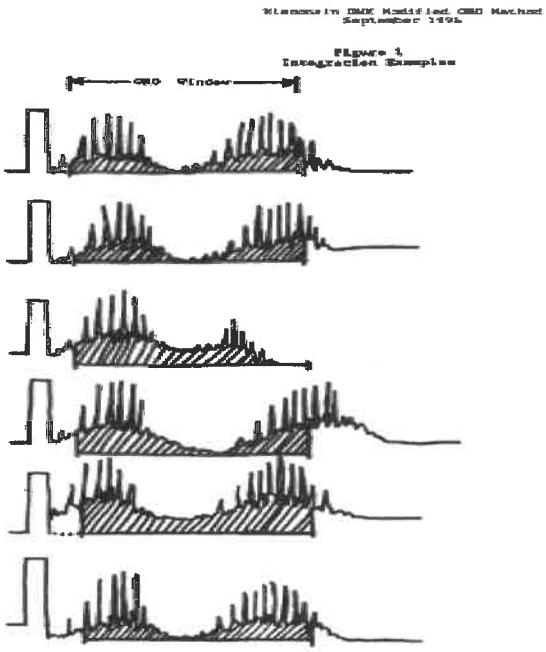
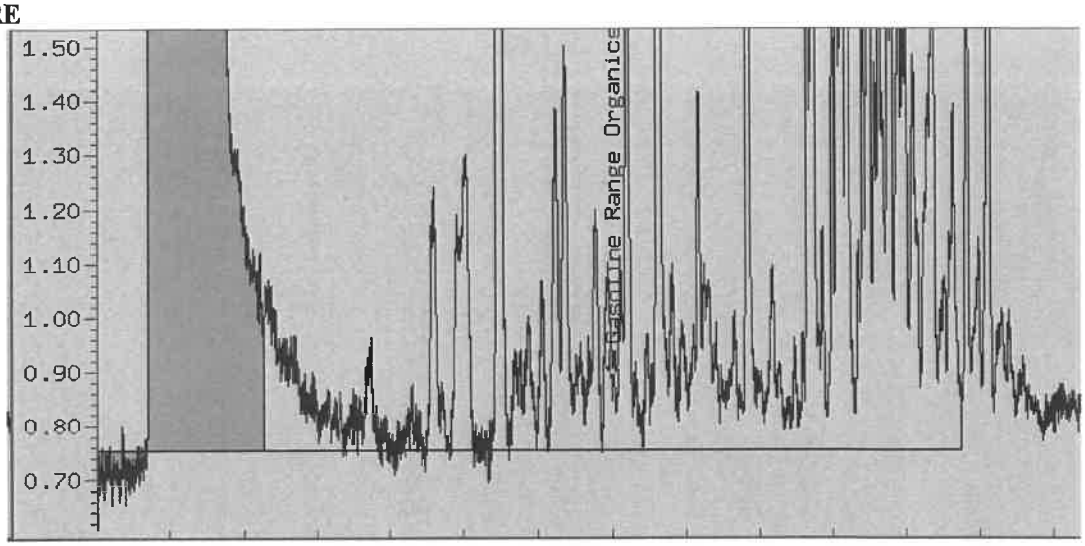


Figure 1A: Example From WIGRO Method

Figure 1B: Laboratory Example of Proper Integration Technique

BEFORE



AFTER

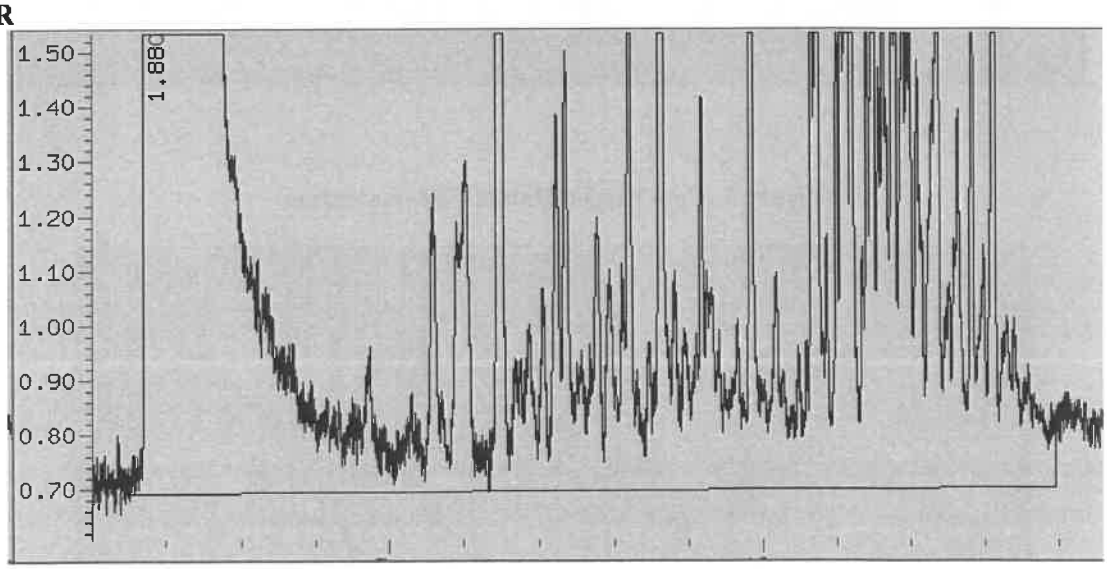


Figure 2: WIGRO Example Chromatogram

2a LCS

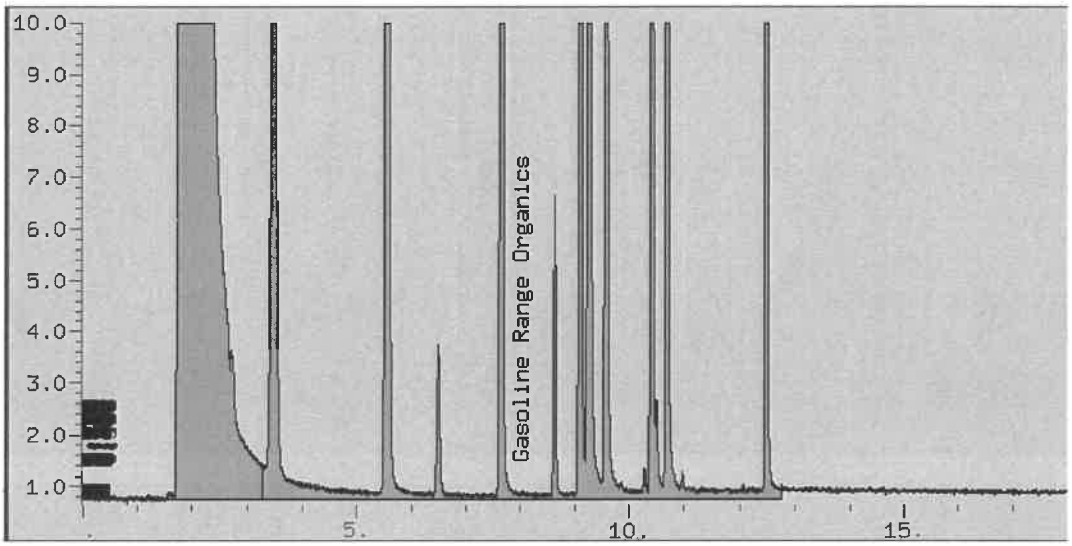
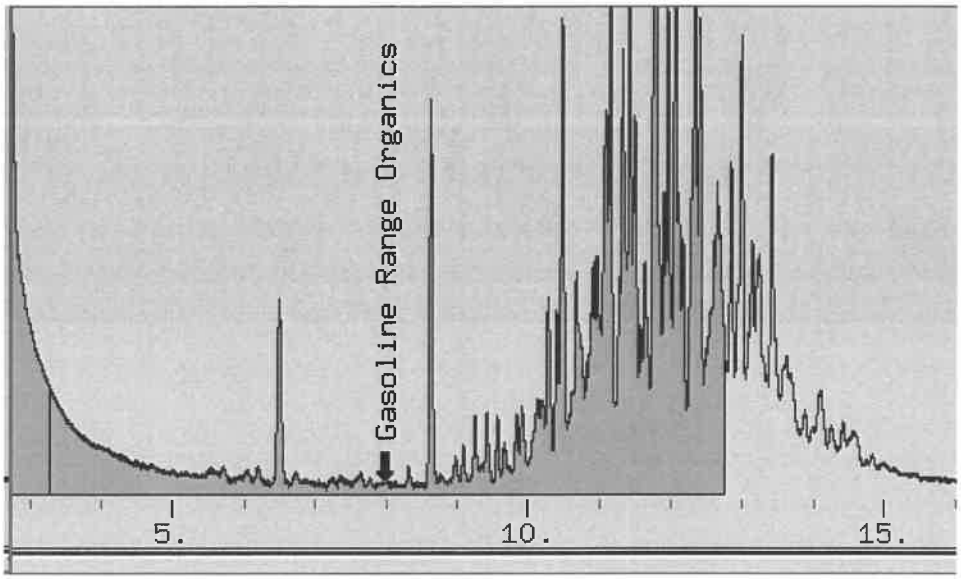


Figure 3: Non-Target Matrix Chromatogram



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STANDARD OPERATING PROCEDURE

PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS BY ICP

Reference Methods: EPA 200.7 and EPA SW-846 3010A

Local SOP Number:	S-MN-I-458-rev.25
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-458-rev.24

APPROVALS

Laboratory General Manager — Date: 16 Mar 2018

Laboratory Quality Manager — Date: 28 Feb 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-458-rev.25

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the digestion of aqueous samples to be analyzed by ICP-AES as described in EPA Method 200.7 and EPA SW-846 Method 3010A.

2. Summary of Method

2.1. Aqueous samples are digested in concentrated nitric acid and hydrochloric acid at 95°C ± 2°C. Samples requiring dissolved metal analysis must be filtered through a 0.45 micron filter prior to preservation.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to the digestion of aqueous sample to be analyzed by ICP as described in EPA Method 3010A.

4. Applicable Matrices

4.1. This SOP is applicable to aqueous samples, mobility-procedure extracts, and liquid waste.

5. Limits of Detection and Quantitation

5.1. Refer to the appropriate analytical SOP. In addition, the reporting limits (LOQ) and current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Liquid	Plastic or glass containers; however, plastic is preferable. Pre-cleaned containers are purchased from a supplier	HNO_3 to pH < 2 See section 7.2	Store at room temperature	Analyzed within 6 months of collection

7.2. If samples are received at pH >2, the samples need to have additional preservative added. This is generally performed in sample receiving upon receipt. The samples are required to equilibrate for 24 hours before conducting digestion. The time of addition of the acid and the time of digestion are documented. The pH is re-verified after the 24 hour time limit. If the sample is still > 2 pH, contact the PM for client notification on how to proceed. If the digestion is conducted, the samples are to be qualified for the pH discrepancy.

7.3. Samples requiring dissolved metals analysis should be filtered in the field through a 0.45 um filter. If the samples are requesting lab filtration, perform the filtration through a 0.45 um filter in the lab and acidify or filter into a nitric preserved container. Check the pH to ensure it is less than 2.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Mechanical pipettes	various sizes	Fisher Scientific or equivalent
Digestion Cups	50mL	Environmental Express or equivalent
Filters	filter mates	Environmental Express, # SC0401
Filters	0.45 µm	Celltreat
Hot Block ™	54 place	Environmental Express
Plastic ribbed watch glasses		Environmental Express
Horizon	Laboratory Information System	See master list for current version
Electronic Prep Log	Pace created software to track preparation	

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized water	ASTM Type II DI (> 18 MegaOhm)	House DI Water
Concentrated nitric acid (HNO3)	Trace Metal grade. Store the bottle at room temperature, expires as specified by manufacturer.	Fisher Scientific or equivalent
Concentrated hydrochloric acid (HCl)	Trace Metal grade Store the bottle at room temperature, expires as specified by manufacturer.	Fisher Scientific or equivalent
Stock solution standards for LCS and MS/MSD samples	The solution identifications are PA-STD-1B, PA-STD-2B and PA-STD-3B. Store stock standards at room temperature. Standards expire as specified by manufacturer. See Table 10.2 for the contents of each standard.	Inorganic Ventures (or equivalent)

10.2. Table 10.2 – Standards Table

PA-STD-1B		PA-STD-2B		PA-STD-3B	
cmpd	mg/L	cmpd	mg/L	cmpd	mg/L
As	200	Ag*	100	Al	2000
Ba	200	B*	200	Ca	2000
Be	200	Mo	200	Fe	2000
Cd	200	Sb*	200	K	2000
Co	200	Sn*	200	Mg	2000
Cr	200	Ti*	200	Na	2000
Cu	200	Zr*	200		
Li*	200	Si*	1000		
Mn	200				
Ni	200				
P*	200				
Pb	200				
Se	200				
Sr*	200				
Tl	200				
V	200				
Zn	200				

* See section 19.1

10.3. Table 10.3 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Working LCS/MS/MSD Spike Solution	PA-STD-1B, PA-STD-2B, PA-STD-3B	50 mL of each standard	DI water	50 mL	200 mL	Varies by element. Refer to appropriate analytical SOP.

10.4 Store at room temperature. Expires in 3 months.

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes and thermometers as specified in SOP S-MN-Q-264 (or equivalent replacement).

11.2. Temperature Logbook

11.2.1. Record the temperature of each hot block daily in the temperature logbook.

11.2.2. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature is checked in different wells of the Hot Blocks such that all wells are evaluated over a period of time.

12. Procedure

12.1. Sample Preparation

12.1.1. Transfer a 50 mL representative aliquot of the well-mixed sample to a labeled digestion vessel. Record the volume used in the prep log Alternate volumes may be used as long as the acid and spike additions are adjusted accordingly.

12.1.1.1. Create a method blank and a laboratory control sample (LCS) using DI water.

12.1.1.2. If the samples are filtered in the lab for dissolved metals, an associated filter blank must be performed and be digested with the batch of samples filtered. The filter blank is not in substitution of the method blank but in addition to.

12.1.2. Spike the LCS, MS/MSD samples with the appropriate amount of metals spike.

12.1.3. Add 1.0 mL of concentrated nitric acid (HNO_3) and 2.5 mL of concentrated hydrochloric acid (HCl), if a lesser volume of sample is digested due to a lack of sample; adjust acids accordingly. If samples originate from WI, perform according to Attachment II.

12.1.4. Place in a Hot Block at 95 ± 2 ° C. Document the block temperature.

12.1.5. Cover each sample with a plastic ribbed watch glass.

12.1.6. Gently reflux for 4 hours, the volume will be approximately 20 mL at this time. Do not allow the samples to boil or to go to dryness.

12.1.7. Remove samples from the digest block and allow to cool.

12.1.8. Bring samples to a 50 mL final volume with DI water, unless a different final volume is required. Cap, mix and label the samples. Record the final volume in the prep log.

12.1.9. Filter the samples if needed – filtration is to be done only if there is concern that insoluble materials may clog the nebulizer. If any sample is filtered, the method blank and LCS must also be filtered.

12.1.9.1. Use the filter mates to plunge-filter the sample in the existing cup.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI water	Prepared and analyzed with each batch of samples digested or for every 20 samples, whichever is more frequent	Refer to appropriate analytical SOP	Refer to appropriate analytical SOP
Filter Blank	DI water filtered through the same lot of filters used to perform lab dissolved metals filtration	Prepared only with batches of lab filtered dissolved metals, one per batch of 20 or less.	This blank is used to demonstrate the cleanliness of the filters used in the process. The filter blank is required to be less than the RL unless otherwise specified by client or QAPP data quality objectives.	Reanalyze to confirm the detection. If the detection is confirmed, refilter DI water using the same filter lot. If the detection is present then the samples are all in question that were filtered with the same lot. Review the detections in the associated samples and perform the corrective actions as defined for a method blank. Refer to the appropriate analytical SOP.
Laboratory Control Sample (LCS)	Prepare an LCS by adding 1.0mL of the working LCS/MS Spike Solution to 50mL of DI water.	An LCS will be digested for each SDG, batch, or 20 samples prepared, whichever is more frequent	Refer to appropriate analytical SOP	Refer to appropriate analytical SOP
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Prepare an MS/MSD by adding 1.0mL of the working LCS/MS Spike Solution to a 50mL aliquot of sample.	An MS and MSD will be digested and analyzed for each SDG or batch of twenty samples or less. For 200.7, batches containing 11 samples or more, a second MS must be prepared.	Refer to appropriate analytical SOP	Refer to appropriate analytical SOP

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section13.

16. Corrective Actions for Out-of-Control Data

16.1. See table in section13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Instrument Detection Limit (IDL) Study**: An IDL study must be conducted quarterly per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement).

18.4. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.5. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. The analytes noted in section 10.2 are not outlined in method 3010A. Annual verification by PT, DOC, and MDL are conducted to document the verification and suitability of this method for these analytes. Records are maintained in the QA Office and are available for review.

19.2. The scope of the method has been expanded to include analytes that have better solubility and stability in hydrochloric acid. Because of this hydrochloric acid is added immediately in the digestion. This is consistent with EPA Method 200.7.

19.3. Pace procedure uses a final nitric acid concentration of 2% and a final hydrochloric acid concentration of 5%. This is more hydrochloric than what is called for in 200.7 due to the concentration of the silver spike at 500 ug/L. For 200.7 samples, Pace will re-prep if results are greater than 100 ug/L at a 50x dilution. Pace can demonstrate the ability to keep silver in solution up to 500 ug/L based on the dilution process. This is the recommendation in SW-846.

19.4. The procedure is consistent with EPA 200.7 because the scope of analytes are similar to the analysis for EPA SW-846 6010B and 6010C.

19.5. The method specifies a 100 mL representative aliquot of sample. Pace utilizes a 50 mL aliquot of sample, employing the Hot Block digestion system rather than glassware.

19.6. The 4 hour digestion time is a modification of the 2 hours indicated in 200.8 due to the difference in the amount of time it takes to reach ~20 mL using the digestion tubes and watch glasses with the hot block.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Method 200.7 Revision 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-atomic Emission Spectrometry.

25.5. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3005A.

25.6. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3010A.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. ATTACHMENT I: WIDNR DIGESTION PROCESS.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-458-rev.24	Added 200.7 Method reference to Section 25 12.1.6 – Clarification on not allowing it to boil or go to dryness Removed 12.2 Section 13 – added Filter blank Added clarification for 200.7 needing the additional MS Added clarification on the filter blank needed in 12.1.1.2 Removed outdated attachments Added 19.6 Added 7.2 Added storage conditions to Table 10.1 10.4 – Replaced this section with "Store at room temperature. Expires in 3 months."	03Feb2016
S-MN-I-458-rev.25	Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Updated reference to training SOP in 18.3 with new local SOP Q-279.	18Jan2018

ATTACHMENT I: WIDNR DIGESTION PROCESS

Pace Analytical	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2016 Page 1 of 1
	Document No.: F-MN-I-411-Rev.02	Issuing Authority: Pace Minnesota Quality Office

For Wisconsin solid samples only

ICPMS/ ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

Document Information

Document Number: ENV-SOP-MIN4-0043 **Revision:** 00

Document Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

Department(s): Metals

Previous Document Number: S-MN-I-492-rev.29

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

METALS ANALYSIS BY ICP/MS

Reference Methods: EPA Methods 6020/6020A/6020B/200.8

Local SOP Number:	S-MN-I-492-rev.29
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-492-Rev.28

APPROVALS

Laboratory General Manager — Date: 26 Jun 2018

Laboratory Quality Manager — Date: 03 May 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-492-rev.29

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. This Standard Operating Procedure provides a detailed description based on EPA Methods 200.8, 6020, 6020A and 6020B for analysis determining dissolved and total recoverable metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) in environmental samples.

2. Summary of Method

2.1. An aliquot of a well-mixed, homogeneous sample is accurately measured for sample processing. For total recoverable analysis of solids and liquids or dissolved liquid analysis requiring digestion, analytes are first solubilized by gently refluxing with nitric and hydrochloric acids. After cooling, the sample is brought to volume, mixed and allowed to settle overnight prior to analysis. For the determination of dissolved analytes in a filtered aqueous sample by 200.8, or for the "direct analysis" total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, and then mixed and allowed to set for the required time prior to analysis.

2.2. The method describes the determination of trace elements in aqueous solutions by ICP-MS. Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards. Additional corrections are applied as necessary to correct for isobaric and poly atomic elemental interferences (Section 6).

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. **Parameters**: This SOP applies to the determination of dissolved and total recoverable elements by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) in environmental samples. Elements are listed in Attachments III and IV.

4. Applicable Matrices

4.1. This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

4.2. Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

4.3. Where this method is approved for the determination of metal and metalloid contaminants in drinking water, samples are analyzed directly by pneumatic nebulization without acid digestion if the samples have been properly acid-preserved and have turbidity of < 1 NTU at the time of analysis, this total recoverable determination procedure is referred to as "direct analysis". Direct Analysis is only applicable to samples being analyzed by 200.8.

4.4. For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes ranges from 0.08 to 53.5 ug/L for waters and 0.08 to 50 mg/Kg for soils depending on the element for this method. All current method detection limits (MDLs) and LOQs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instrument's spectrometer. One way to solve this problem is to measure a different isotope for which there is no interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

6.1.1. All analytes listed in section 2.1 have at least one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table III), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. Such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

6.2. Abundance Sensitivity Interference – Abundance sensitivity interference refers to the degree of peak overlap that can occur between adjacent peaks. Interference can occur when the shoulder of a large peak significantly overlaps the peak of a neighboring minor peak, thereby contributing to its intensity. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

6.3. Isobaric Polyatomic Interference – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. Examples include ArCl+ (mass 75) which interferes with As, ClO+ (mass 51) which interferes with V and must be corrected by measuring ClO+ at mass 53. These interferences are highly dependent on the matrix of the samples and day-to-day plasma conditions, so correction factors must be determined on the day of analysis. When possible, one should choose an interference-free isotope to measure.

6.4. Physical Interferences – Physical interferences result from the physical processes associated with the transport of sample to the plasma, sample behavior within the plasma, and transmission through the interface region between the plasma and the mass spectrometer. Viscosity and surface tension differences can affect results, as can deposits on the sampler and skimmer cones caused by large quantities of dissolved solids in the sample. The interferences can be compensated for by the use of internal standards that approximate the analytical behavior of the elements being determined. Additionally, it is recommended that dissolved solids in samples be kept below 0.2% (w/v).

6.5. Memory Interference – Memory interferences are related to sample transport and result when there is carryover from one sample to the next. Sample carryover can result from sample disposition on the sample and the skimmer cones and from incomplete rinsing of the sample solution from the plasma torch and the spray chamber between samples. These memory effects are dependent upon both the analyte being measured and sample matrix and can be minimized through the use of suitable rinse times.

6.5.1. The rinse times necessary for a particular analyte should be estimated prior to analysis. This may be achieved by aspirating a standard containing the analyte at a concentration ten times the LDR for the normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce the analyte signal to less than ten times the method detection limit should be noted. The minimum rinse time between samples should be set to this time. Memory interferences may also be assessed within an analytical run by using three or more replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should check for the possibility

of a memory effect. If the analyte concentration in the previous sample is high enough to suspect analyte carryover, the sample should be re-analyzed after a long rinse period.

6.6. Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in samples, fortified sample matrices and even fortified blanks if determined as a dissolved analyte or by "direct analysis" where the sample has not been processed using the total recoverable mixed acid digestion. For this reason samples are digested prior to the determination of silver. The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

6.7. The total recoverable sample digestion procedure given in this method will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Collection, Preservation and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Plastic or glass, 250 mL minimum.	Acidified with nitric acid to pH<2; if received unpreserved, add 1.5 mL nitric for each liter received. Mix and confirm that the pH has been adjusted to <2 and allow the samples to sit for 24 hours[1] prior to analysis.	Ambient if preserved properly with nitric acid	Must be prepped and analyzed within 180 days of collection for all other metals than mercury.
Solid	8 oz glass jar	None	<6°C, but above freezing	Must be prepped and analyzed within 180 days of collection.

[1]EPA Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies.

Supply	Description	Vendor/ Item # / Description
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700 and Thermo XII ICPMS instrumentation equipped with interference reduction technology via collision cells. Cetac ASX-520 Autosampler, Niagra valve system and PC3 spray chamber chiller/valve system. Thermo or Agilent recirculating chiller.	Thermo Fisher Scientific XSeries 2 ICP-MS Agilent 7700 series ICPMS
Argon gas	High purity grade, 99.99%	Praxair or equivalent replacement
Collision Gas	High purity 7%H/93% He mix Ultra high purity He, Ultra high purity H_2	Praxair (Oxygen Services) or equivalent replacement
Autosampler tubes	15 mL metals free autosampler tubes	Mold Pro, MP100 or equivalent replacement

Rough pump oil	CMP-19	Fisher Scientific or equivalent replacement
Peristaltic pump tubing	Various sizes	Fisher Scientific or equivalent replacement
Analytical Balance	With the capability to measure to 0.1 mg	Sartorius BP 110S, A&D EK-610i, A&D FX1200, or Sartoriius LC612S-00MS or equivalent replacement
Mechanical pipettors	Capable of delivering volumes ranging from 10 to 5000 µL, and associated metal –free disposable pipet tips	Eppendorf, Fisher brand or equivalent replacement
Glassware	Class A volumetric flasks, graduated cylinders, funnels (glass and/or metal-free plastic	Fisher Scientific or equivalent replacement
Digestion cups	50 mL disposable digestion cups	Environmental Express SC475or equivalent replacement
Digestate Filters	6µm PTFE-faced polypropylene filter	Environmental Express, # SC0408
Narrow mouth storage bottles	FEP (fluorinated ethylene propylene) with screw top closure, 135 mL to 1 L capacity	C&G Containers or equivalent replacement
Data Reporting Software		Horizon (also referenced Epic Pro)
Data Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS	Limslink
Instrument software	PlasmaLab and Mass Hunter ICPMS software	Thermo Fisher and Agilent

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards.

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Hydrochloric acid (HCl)	Trace metals grade or better	Fisher Scientific, A-508-P212 or equivalent replacement
Nitric Acid (HNO_3)	Trace metals grade or better	Fisher Scientific, A-509-P212 or equivalent replacement
Deionized water (DI)	Reagent grade (DI) water – (18 MOhm resistivity)	Barnstead Epure System or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.	n/a
Calibration Stock Standard solutions	Custom blend of elements. See Table 10.2 for the standard preparation information Table IV contains Stock Standard Concentrations.	Spex Certiprep, XFSPA-221-250, XFSPA-656-250, XFSPA-220-250, XFSMN-26-250A, XFSMN-27-250A, XFSMN-28-250A,Inorganic Ventures PACE-28; or equivalent replacement
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend from of elements from a different source than the Calibration Stock Standard Solutions. See Table 10.2 for standard preparation information. Table IV contains Stock Standard Concentrations.	Inorganic Ventures, PACE-5, PACE-4B , Hg: 4400-1000331 or equivalent replacement.
Internal Standard Stock Solution	Can be a purchased multi-element blend or single element standards to be mixed prior to use with concentrations of 10,000 ug/mL.	Peak Performance; In: S4400-1000241, Y: S4400-1000671, Tb: S4400-1000571; Inorganic Ventures; Ge: CGGe1

		Th: CGTH1, Sc: CGSC-1
Tuning Stock Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.	Spex Claritas PPT, CL-Tune-1
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Prepared in 1G increments utilizing Nalgene bottles. This is measured by mixing 76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.	n/a

10.2. Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration (ug/L)
Standard 1	Inorganic Ventures, PACE-28	0.1	2% Nitric/1% HCl acid solution	9.9	10	varied
Standard 2	Refer to Table 10.1, See Calibration Stock Standard Solutions	0.500		9.5	10	10/125
Standard 3		1.000		9	10	20/250
Standard 4		0.1		9.9	10	200/2500
Standard 4		0.1		9.9	10	200/2500
Standard 4		0.1		9.9	10	200
Standard 5		0.05		9.95	10	500
Standard 5		0.05		9.95	10	250/500/2500
Standard 5		0.25		9.75	10	25000
CRDL	Inorganic Ventures, PACE-28	0.1		9.9	10	varied
ICS-A	Inorganic Ventures 6020 ISC-OA	0.25		9.75	10	25000
ICS-AB	Inorganic Ventures, 6020 ISC-OA Spex, XFSPA-221-250, XFSPA-656-250, XFSPA-220-250	0.25		9.60	10	100/1250/26250
		0.05				
		0.05				
		0.05				
ICV	Inorganic Ventures, PACE-5, PACE-4B	0.2		49.6	50	80/1000
		0.2				
CCV	Inorganic Ventures, PACE-5, PACE-4B	0.2		49.6	50	80/1000
		0.2				

11. Calibration and Standardization

11.1. Table 11.1 Calibration and Standardization.

Calibration Metric	Parameter / Frequency	Criteria	Comments

Tune	Daily prior to any calibration	EPA performance reports - print and file all reports, note failures and corrective actions taken. Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Note: The tuning criteria utilized are based on the 200.8 method. The mass criteria for 200.8 (0.75 amu at 5% peak height) is more stringent than the criteria for 6020A/B (0.9 amu at 10% peak height) therefore applicable for both methods.	The tune criteria must pass before instrument is calibrated and any samples are analyzed. Follow manufacturer guidelines for troubleshooting and maintenance for tune failures. Document all maintenance performed and return to compliance.
Calibration Curve Fit	Linear Regression	$r \geq 0.998$	Instruments are setup with the following calibration curve fit options; linear regression, linear regression through the blank, and weighted least squares. Analysis is conducted utilizing the calibration curve fit method recommended by the instrument manufacturer. If not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ± 10% of the true value %RSD between multiple integrations must be ≤ 5%	Review the standard preparation. Remake the standard accordingly if that is the cause. Re-inject the ICV one more time, if it fails stop all analysis. Perform all necessary instrument maintenance and recalibrate the instrument. Only two injections are allowed back to back, then the system must be recalibrated.
Initial Calibration Blank (ICB)	Immediately after the initial calibration verification	Evaluate the blank to the MDL depending on the data quality objective of the associated samples, a blank with detections less than the RL or have levels at least $1/10^{th}$ of that in the associated samples to be acceptable. 6020B ICB and NC samples are required to be < ½ RL for target analytes. Per client QAPP/Technical Specifications the blanks may require to be evaluated and clean to the MDL or ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	If there is a detection, stop analysis and determine the source of the contamination. Perform any necessary maintenance and recalibrate the instrument accordingly.

		The blanks must be clean to the data quality objectives.	
Contract Required Detection Limit Sample (CRDL) or Low Level Initial/ Continuing Calibration Verification (LLICV/LL CCV)	6020/6020B/200.8 - The CRDL must be analyzed at the beginning of each run for every analyte of interest. 6020A - must be analyzed at the beginning of each run, and once at the end of each analytical batch. Per client QAPP/Technical Specifications a closing CRDL may be required every 8 hours or close of the sequence of client samples, whichever more frequent.	The CRDL/LLICV/LLCCV is at or below the RL. For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (or specified by the client).	Evaluate standard preparation, re-prepare and analyze if suspected. -If the CRDL fails high, samples that are non-detect below the reporting limit can be reported and sample concentrations exceeding the CCV may be reported provided the CCV passes all criteria, as there would be no impact from the high bias. -If the CRDL is biased low, no data can be reported for the target elements failing criteria including J-flagged data. The system must be stopped. Perform any necessary maintenance and recalibrate accordingly.
Interference Check Solutions (ICSA/AB)	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining analyzed elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter. Per client QAPP/Technical Specifications a closing sequence ICSA/AB may be required at a frequency of every 8 hours or end of sequence, whichever is more frequent.	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value. Client QAPPs/Technical Specifications by provided alternate criteria for non-spiked elements and must be followed accordingly.	If the ICSA or AB fail criteria, stop analysis. Review the standard preparation, remake accordingly. Perform any necessary maintenance and recalibrate prior to sample analysis. Adjust the equations accordingly or recalibrate as needed to meet specified requirements. Note that monitoring the interference source does not necessarily require monitoring the interference itself, but that a molecular species may be monitored to indicate the presence of the interference Exception: If the minerals are high bias and the analytes of interest are not impacted, passing all criteria, the data may be reported.

Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.	% Diff ± 10% of the true value %RSD between multiple integrations must be ≤ 5%	If the requirements for continuing calibration are not met, review for preparation error or instrument malfunction. A CCV may be repeated, but a second failure requires the system to be recalibrated prior to further analysis. If the samples bracketed are non-detect and the CCV is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/MS) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples. If the CCVs are biased low, reanalyze any samples impacted since the last passing CCV.
Continuing Calibration Blank (CCB)	Following every CCV injection	Evaluate the blank to the MDL depending on the data quality objective of the associated samples, a blank with detections less than the RL or have levels at least 1/10th of that in the associated samples to be acceptable. NC samples are required to be < ½ RL for target analytes. Per client QAPP/Technical Specifications the blanks may require to be evaluated and clean to the MDL or ½ RL. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	If there is detection, stop analysis and determine the source of the contamination. Perform any necessary maintenance and recalibrate the instrument accordingly. Reanalyze any samples impacted since the last passing CCB.
Internal Standard Response	Monitor the signal intensity for the internal standard masses throughout the analytical run. This information is useful in detecting instrument drift, sensitivity shifts, and inherent internal standard (i.e., a natural constituent in a	For method 6020, the absolute intensity of any one internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The absolute intensity of any one internal standard in the samples and remaining QC must not deviate more than 30-120% from its original intensity in the associated calibration blank. For method 6020A/B, the	If deviations greater than these are observed, flush the instrument with the rinse blank and re-analyze the calibration blank and examine the internal standard intensities with the following actions; If the intensities of the internal standards are acceptable, dilute a fresh aliquot of the sample and re-analyze. A 5X dilution or

	sample).	intensity of the internal standards must not fall below 70% from its original intensity in the associated calibration blank for any standard or sample. An upper limit of 125% from its original intensity in the associated calibration blank will be applied to the internal standard recoveries for analyses. For Method 200.8 the absolute intensity of any one internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	larger factor or multiple dilutions may be required to achieve acceptable results. If the internal standards are still out-of-limits, terminate the analysis and determine the cause of the drift. Routine maintenance of the sampling interface or re-tuning the mass spectrometer may be required. The system must be calibrated and any samples not bordered by acceptable ICV/CCV samples re-analyzed.

12. Procedure

12.1. Perform all initial system set up procedures

12.1.1. Check waste jug. If more than half full, empty it into the acid waste stream.

12.1.2. Check the chiller temperature and water level. Add additional water as needed to return the instrument to specification. The system will not operate if the water is too low.

12.1.3. Check rough pump oil color. It should be amber or light yellow; if it is the color of coffee it should be changed.

12.1.4. Check pressure of collision gas cylinder, change if needed.

12.1.5. Open the torch chamber and visually inspect the cones if the instrument performance is not acceptable. If there are significant deposits, remove both cones and clean or replace them.

12.1.6. Check torch position and fittings. Adjust the position of the torch accordingly, reattach the fittings as necessary.

12.1.7. Check the peristaltic pump tubing. If it has flattened or plugged, replace it.

12.1.8. Turn plasma on by switching the instrument into the operating state. Place the probes in 2% nitric acid solution and allow the instrument to warm up for 20-30 minutes.

12.1.9. Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

12.1.10. Perform daily tuning and optimization of the instrument in the following order:

12.1.10.1. Torchbox alignment, if torch chamber was opened.

12.1.10.2. Perform the autotune function for all modes of analysis.

12.1.10.3. Generate performance reports, print and file all reports. Note failures and corrective actions taken.

12.1.11. See Attachment I for an example of the Daily Operational Checklist Form F-MN-I-212, or equivalent replacement.

12.2. Analysis

12.2.1. Select masses carefully to avoid and/or minimize interferences. Make sure appropriate measures are in place to deal with interferences. Use an internal standard within 50 mass units of the analyte if possible. Table I outlines Internal standards recommended for use with this method. Alternative elements for the appropriate mass range were selected for use based on performance, these include ge and th.

12.2.2. Run analytical samples, appropriate batch and quality control samples.

12.2.3. Each sample preparation set will typically include a digestion blank, a laboratory control spike, matrix spike and sample duplicate, as defined in the Glossary Section of the Pace

Quality Manual. More quality control samples may be necessary. In particular, it is suggested that the matrix spike be added in a form that tests the digestion efficiency.

12.2.4. CCV/CCB required every 10 samples using the same solution and control limits as the ICV and ICB standard and blank.

12.2.5. Review results of quality control samples for PASS/FAIL criteria.

12.2.6. Review the sample in-solution concentrations. Samples that have concentrations outside the linear range for each element will be diluted until they fall within range.

12.2.7. At the conclusion of the experiment, set the instrument to go into vacuum status. Selecting shut down will turn the vacuum off. Selecting or leaving the instrument at none will leave the plasma on.

12.3. Daily File (in hardcopy format; if electronic assembly is available the same images will be captured in pdf accordingly)

12.3.1. Gather all daily printouts.

12.3.2. Print instrument raw data to PDF paperless printer.

12.3.3. Include the calibration summaries and runlogs.

12.3.4. Update and include necessary standard preparation logbooks.

12.3.5. Label the daily folder with the date and instrument information.

13. Quality Control

13.1. Table 13.1 Quality Control and Corrective Action.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water for water samples or Teflon chips/or nonmetal containing solid matrix for soil batches	One per 20 samples	Target analytes must be less than reporting limit. 6020B – Target analytes must be less than ½ reporting limit for 6020B. If results are reported to MDL, target analytes in MB should be non-detect. NC samples are required to be < ½ RL for target analytes. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	DI water spiked with all target compounds or a spiked Teflon chip/or nonmetal containing matrix for soils.	One per 20 samples	6020/6020A/6020B: 80-120% 200.8: 85-115%	The analyses should be terminated, the problem corrected, and the samples associated with that LCS re-analyzed. If reanalysis of the samples fail, the samples affected by the failing LCS elements need to be re-digested and re-analyzed. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported

				with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 20 samples for 6020 and 6020A One per 10 samples for 200.8	6020/6020A/6020B : 75-125% 200.8: 70-130%	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Perform a PDS on any elements that failed to meet criteria. For Minnesota Admin Contract clients – all MS/MSD failures require analysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
MSD / Duplicate	MS Duplicate *OR* *(alternative)* Sample Dup	One for every 20 samples for 6020 and 6020A.	%Diff ≤ 20%	Report results with an appropriate footnote.
Serial Dilution	A 5-fold dilution of a digested sample. See section 13.1.1.	One per batch of 20 samples or less	6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	If criteria is not met, original sample and dilution shall be reanalyzed.
Post Digestion Spike (PDS)	An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. Spiked with 20 uL of 20/250 mg/L stock concentration.	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, then the dilution test on the PDS must be performed. If the dilution test fails, it is determined to be matrix interference,
Serial Dilution of Post Digestion Spike	The above PDS is diluted 1:5 and treated as a serial dilution	Required by certain client QAPPs. This is not a standard practice	fivefold dilution must agree within ± 10% of the original determination	If this fails data is qualified
Laboratory Filter Blank (FB)	A filtered aliquot of reagent water treated and prepared exactly as a sample when lab filtration is requested.	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. 6020B – Target analytes must be less than ½ reporting limit for 6020B. NC samples are required to be < ½ RL for target	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample

			analytes. If results are reported to MDL, target analytes in MB should be non-detect. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

13.1.1. To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of 2% HNO3 / 1% HCl DI water diluents. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.

14. Data Analysis and Calculations

14.1. Percent Recovery Equation:

$$\% \text{ Recovery} = \frac{(SSR-SR) \times 100}{ST}$$

Where:
SSR = Spike sample result, µg/L or mg/kg dry
SR = Sample result, µg/L or mg/kg dry
ST = Spike target, µg/L or mg/kg dry

14.2 Relative Percent Difference Calculation

$$RPD = \frac{|(S-D)| \times (100)}{(S+D)/2}$$

Where:
RPD = Relative Percent Difference
S = Original Spiked Sample Value, µg/L or mg/kg dry
D = Second Spiked Sample Value, µg/L or mg/kg dry

14.3 Concentration Calculation for Soils

$$(W) \times (1- (M/100)) = C$$

$$\frac{(K) \times (D) \times (F)}{(C \times 1000)} = X$$

Where:
W = original sample weight (g)
M = percent moisture
K = on column analyte concentration (ug/L)
D = digested volume (mL)
F = dilution factor
C = moisture corrected weight (g)

14.4 Linear Regression Equation

$$y = mx + b$$

Where:
y = instrument response
m = slope of calibration function
x = analyte concentration (ug/L)
b = blank concentration (ug/L)

14.5 The Weighted Least Squares Regression of each point (w_i) is calculated as follows:

$$w_i = S_i^{-2} / (\sum_i S_i^{-2} / n)$$

Where:
w_i = weight of each point
S_i = standard deviation of each point
n = number of point

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See tables in section 11 % 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269, Method Detection Limit Studies for each matrix per instrument. For 200.8, a new MDL must be performed with each new analyst begins work as well.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279, Training and Employee Orientation.

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Results are stored in the QA office.

18.5. An Instrument Detection Limit (IDL) Study is required quarterly. This consists of three runs on three consecutive days from the analysis of a reagent blank with seven consecutive measurements per day. The data will be maintained in the metals department.

19. Method Modifications

19.1. The tuning criteria utilized is based on the 200.8 method. The mass criteria for 200.8 (0.75 amu at 5% peak height) is more stringent than the criteria for 6020A (0.9 amu at 10% peak height) therefore applicable for both methods

19.2. Sample digestates are allowed to settle overnight. If undissolved material does not settle out, the sample digestate, method blank, and laboratory control sample are filtered prior to analysis on the instrument. The laboratory does not centrifuge samples as suggested in the method.

19.3. LRB results are not subtracted from LFB results for calculation of LFB recoveries. The level of contamination in the LRB should be minimal when compared to the LFB spiking levels.

19.4. Instruments utilize dual detector scanning, analog and pulse counting. The detector cross calibration is used to convert the data acquired using analog detector mode into the equivalent pulse counting data. During detector cross calibration, a polynomial fit line is applied by comparing masses found on the y-axis against x-axis. External calibration is completed each day by utilizing 100/1250 ug/L solution. Internal instrument calibrations are also obtained by the instrument during pauses in operation, utilizing obtained integrated counts per second across the mass range and creating a separate polynomial fit line.

19.5. Calibration

19.5.1. Linear Regression

19.5.1.1. Utilizing a standard linear regression format (See Equation 14.4). All points of the calibration curve including the blank are plotted as regular fitted points.

19.5.2. Weighted Least Squares Regression

19.5.2.1. Utilizing Weighted Least Squares Regression in Section 14.5. When data with more than one repetition is used to create a calibration curve, weighted regression can be selected. The count error for data collected in this manner is represented as the standard deviation of the counts. Usually the count error for higher concentrations is larger than that for lower concentrations. When weighted regression is selected, lower concentrations are given more weight because it is more desirable that the curve pass through points having lower error than points having higher error.

19.6. Analyte List in Attachments III and IV

19.6.1. The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, and uranium-238. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

19.6.2. The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

19.6.3. The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium-238. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

19.7. Kinetic Energy Discrimination (KED)

19.7.1. Interference reduction technologies, such as collision cells or reaction cells, are designed to reduce the effect of spectroscopic interferences that may bias results for the element of interest. The use of interference reduction technologies is allowed, provided the method performance specifications relevant to ICP-MS measurements are met. Collision Mode is utilized for all 6020/200.8 methods that have undergone PT, MDL, IDL, and LDR studies, with the exception of drinking water samples that cannot utilize this mode.

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument manual for maintenance procedures performed by the lab.

20.2. Routine Maintenance (Document all routine maintenance in the Routine Maintenance Logbook at the time of performing the maintenance)

20.2.1. Weekly Maintenance.

20.2.1.1. Replace peristaltic pump tubing for sample, internal standard and waste.

20.2.1.2. Clean spray chamber and nebulizer.

20.2.1.3. Clean and/or replace torch (optional or as needed).

20.2.1.4. Check and clean air filters id necessary.

20.2.1.5. Check multiplier voltages.

20.2.2. Monthly Maintenance

20.2.2.1. Check rotary pump oil.

20.2.2.2. Check oil mist filters.

20.2.2.3. Replace sample uptake tubing.

20.2.2.4. Check chiller water level in reservoir

20.2.2.5. Bi-Annual Maintenance

20.2.2.6. Examine lens system and clean, if necessary.

20.2.2.7. Examine penning gauge and clean, if necessary.

20.2.2.8. Change rotary pump oil (perform more frequently based on sample type and load).

20.2.2.9. Annual Maintenance

20.2.2.10. Replace worn o-rings.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

25.5. Fisons –VG Genesis Users Manual.

25.6. Region 9 Laboratory Standard Operating Procedure 130, Glassware Cleaning Procedures.

25.7. Region 9 Laboratory Standard Operating Procedure 462, Analysis of Total Suspended Solids By EPA Method 160.2.

25.8. U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

25.9. U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

25.10. U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

25.11. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

25.12. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: ICPMS Daily Operational Checklist

26.2. Attachment II: IS Reference ICPMS.

26.3. Attachment III: Method 6020/6020A/6020B/200.8 Analyte List and Reporting Limits

26.4. Attachment IV: Method 200.8 (Drinking Water) Analyte List and Reporting Limits

26.5. Table I: Internal Standards and Limitation of Use.

26.6. Table II: Recommended Elemental Equations for Data Calculations.

26.7. Table III: Recommended Analytical Isotopes and Additional Masses.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-492-rev.28	Remove "Uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Section 12.2.3 reference to PASI changed to Pace. Removed extra space from Section 1.1. Deleted "<6 degrees C….otherwise" from Table 7.1, aqueous row and storage column. Fixed typos in Preservation column in Table 7.1, aqueous row. In Table 9.1, digestate filters row: edited Description to 6 um from 5, edited Item # to SC0408. Table 10.1, added Inorganic Ventures PACE-28 to Calibration Stock Standard Solutions for Item #s. In Table 10.2: edited all columns except Final Volume for Standard 1 row, removed the rows Hg Intermediate, Standards 6-9, and ICV/CCV/Hg*. Edited 12.3.2 to print to PDF paperless printer instead of hard copy. Deleted Section 19.3.	02Oct2017

S-MN-I-492-rev.29	3.2 – added "Elements are listed in Attachments III and IV." To replace old section 3.3 (now deleted). Table 7.1 – Added note below table for superscript 1 in Aqueous Preservation. Table 9.1 – changed ICPMS Description to ASX-520 instead of 530. Table 11.1 – Updated Tune Criteria to "reports – print" 12.1.10.2 – replaced with "Perform the autotune function for all modes of analysis." 12.1.10.3 – Updated to "Generate performance reports" instead of EPA Deleted sections 12.1.10.4 through 12.1.10.8 12.2.1 – deleted "General method development steps are outlined in the Training and Operations manuals from Thermo Scientific" and added "Table I outlines…include ge and th." 18.3 – updated to reference new local training SOP number. 19.6 – moved analyte list to Attachments III and IV instead of 3.3. Deleted "All analytes listed in section 3.3 are part of the current TNI certification" from Sections 19.6.1 and 19.6.2. 26.3 – added 6020B and edited to Reporting Limits instead of PRL Added 26.4, attachment IV. Updated Attachment I. Attachment III – Added decimal place, updated to correct CAS #s for Fe, K, Na, and U, edited Silica numbers, added (PRL) to Note. Reformatted Tables I, II, and III.	20Apr2018

Attachment I – ICPMS Daily Operational Checklist

Pace Analytical®	Document Name: ICPMS Daily Checklist	Document Revised: 24Feb2014 Page 1 of 25
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ICPMS Daily Operational Checklist

Date	Analyst	Sample Tube Lot #	Waste Jug	Chiller Temp (°C)	Default Corrections Equations changed?	Vacuum Pressure (torr)	Peristaltic Tubing Changed?	Manual shutdown?	Comments & Maintenance
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no If yes, please provide analyte and multiplier value:____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	

Reviewed by:____________________Date:__________

Attachment II - IS Reference ICPMS

Pace Analytical®	Document Name: IS Reference ICPMS	Document Revised: 27Jan2014 Page 1 of 1
	Document Number: F-MN-I-260-rev.01	Issuing Authority: Pace Minnesota Quality Office

Instrument: Thermo XII

IS	45 Sc-CCT	45 Sc	72 Ge	115 In	159 Tb	232 Th
	7 Li	23 Na	59 Co	88 Sr	205 Tl	238 U
	9 Be	25 Mg	60 Ni	95 Mo	208 Pb	
	10 B	27 Al	63 Cu	107 Ag	209 Bi	
		28 Si	66 Zn	111 Cd		
		39 K	75 As	118 Sn		
		43 Ca	78 Se	121 Sb		
		47 Ti		137 Ba		
		51 V				
		52 Cr				
		54 Fe				
		55 Mn				

Instrument: Agilent 7700

IS	45 Sc -H_2	45 Sc	72 Ge-H_2	72 Ge	115 In	159 Tb	232 Th
	7 Li	23 Na	78 Se	59 Co	95 Mo	205 Tl	238 U
	9 Be	24 Mg		60Ni	107 Ag	208 Pb	
	10 B	27 Al		63 Cu	111 Cd	209 Bi	
	28 Si	39 K		66 Zn	118 Sn		
		43 Ca		75 As	121 Sb		
		47 Ti		88 Sr	138 Ba		
		51 V					
		52 Cr					
		55 Mn					
		56 Fe					

Attachment III: – Method 6020/6020A/6020B/200.8 Analyte List and Reporting Limits

Analyte	CAS#	Non-Potable Water PRL(ug/L)	Soil PRL (mg/kg)
Aluminum	7429-90-5	10.00	10.00
Antimony	7440-36-0	0.50	0.50
Arsenic	7440-38-2	0.50	0.50
Barium	7440-39-3	0.30	0.30
Beryllium	7440-41-7	0.20	0.20
Bismuth	7440-69-9	0.50	0.50
Boron	7440-42-8	5.00	5.00
Cadmium	7440-43-9	0.08	0.08
Calcium	7740-70-2	40.00	40.00
Chromium	7440-47-3	0.50	0.50
Cobalt	7440-48-4	0.50	0.50
Copper	7440-50-8	1.00	1.00
Iron	7439-89-6	50.00	50.00
Lead	7439-92-1	0.10	0.10
Lithium	7439-93-2	0.50	0.50
Magnesium	7439-95-4	10.00	10.00
Manganese	7439-96-5	0.50	0.50
Molybdenum	7439-98-7	0.50	0.50
Nickel	7440-02-0	0.50	0.50
Palladium	7440-05-3	0.50	-
Platinum	7440-06-4	0.50	-
Potassium	7440-09-7	50.00	50.00
Selenium	7782-49-2	0.50	0.50
Silica	7631-86-9	107.00	107.00
Silicon	7740-21-3	50.00	50.00
Silver	7440-22-4	0.50	0.50
Sodium	7440-23-5	50.00	50.00
Strontium	7440-24-6	0.50	0.50
Thallium	7440-28-0	0.10	0.10
Tin	7440-31-5	0.50	2.000
Titanium	7440-32-6	1.00	1.00
Vanadium	7440-62-2	1.00	1.00
Uranium-238	7440-61-1	0.50	0.50
Zinc	7440-66-6	5.00	5.00

NOTE: Reporting Limits (PRL) are current at the time of issuing this SOP. For the most current reporting limits, refer to LIMS system.

Attachment IV: – Method 200.8 (Drinking Water) Analyte List and Reporting Limits

Analyte	CAS#	PRL (ug/L)
Aluminum	7429-90-5	10.0
Antimony	7440-36-0	0.50
Arsenic	7440-38-2	0.50
Barium	7440-39-3	0.30
Beryllium	7440-41-7	0.20
Bismuth	7440-69-9	0.50
Boron	7440-42-8	5.00
Cadmium	7440-43-9	0.08
Calcium	7740-70-2	40.00
Chromium	7440-47-3	0.50
Cobalt	7440-48-4	0.50
Copper	7440-50-8	1.00
Iron	7439-89-6	50.00
Lead	7439-92-1	0.10
Lithium	7439-93-2	0.50
Magnesium	7439-95-4	10.0
Manganese	7439-96-5	0.50
Molybdenum	7439-98-7	0.50
Nickel	7440-02-0	0.50
Potassium	7440-09-7	50.00
Selenium	7782-49-2	0.50
Silica	7631-86-9	106.98
Silicon	7740-21-3	50.00
Silver	7440-22-4	0.50
Sodium	7740-23-5	50.00
Strontium	7440-24-6	0.50
Thallium	7440-28-0	0.10
Tin	7440-31-5	0.50
Titanium	7440-32-6	1.00
Vanadium	7440-62-2	1.00
Uranium-238	7440-61-1	0.50
Zinc	7440-66-6	5.00

NOTE: Reporting Limits (PRL) are current at the time of issuing this SOP. For the most current reporting limits, refer to LIMS system.

Table I - Internal Standards and Limitations of Use

Internal Standard	Mass	Possible Limitations
Lithium	6	a
Scandium	45	polyatomic ion interference
Yttrium	89	a, b
Rhodium	103	
Indium	115	isobaric interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	
Bismuth	209	

a – May be present in environmental samples
b – In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

NOTE: Internal standards recommended for use with this method are underlined.

Table II - Recommended Elemental Equations for Data Calculations

Element	Elemental Equation	Note
Al	(1.000)(^{27}C)	
Sb	(1.000)(^{123}C)	
As	1.000(^{75}C) - 3.1278(^{77}C) + 0.815(^{82}C)	1
Ba	1.000(^{137}C)	
Be	1.000(^{9}C)	
Cd	1.000(^{111}C) - 1.073^{108}C) + 0.76398(^{106}C)	2
Cr	1.000(^{52}C)	3
Co	1.000(^{59}C)	
Cu	1.000(^{63}C)	
Pb	1.000(^{206}C) + 1.000 (^{207}C) + 1.000(^{208}C)	4
Mn	1.000(^{55}C)	
Mo	1.000(^{98}C) - 0.146(^{99}C)	5
Ni	1.000(^{60}C)	
Se	1.000(^{82}C)	6
Ag	1.000(^{107}C)	
Tl	1.000(^{205}C)	
Th	1.000(^{232}C)	
U	1.000(^{238}C)	
V	1.000(^{51}C) - 3.1081(^{53}C) + 0.353351(^{52}C)	7
Zn	1.000(^{66}C)	
Bi	1.000(^{209}C)	
Sc	1.000(^{45}C)	
Tb	1.000(^{159}C)	
Y	1.000(^{89}C)	
Hg	1.000(^{202}C)	

C – Calibration blank subtracted counts at specified mass.
1 – Correction equation for as taken from EPA method 6020. Isobaric correction for ArCl.
2 – Correction for MoO, Sn.
3 – The background for ClOH will normally be small and can be estimated from the reagent blank.
4 – Allowance for isobaric variability of lead isotopes.
5 – Isobaric elemental correction for Ru.
6 – Some Ar supplies contain Kr as an impurity. Se is corrected for Kr by background subtraction.
7 – Correction for chloride interference with adjustment for Cr. ClO 51/53 ratio may be determined from the reagent blank. Isobaric mass 52 must be from Cr only not ArC^{+}.

Table III - Recommended Analytical Isotopes

Element of Interest	Isotope
Aluminum	27
Antimony	121, 123
Arsenic	75
Barium	135, 137
Beryllium	9
Cadmium	106, 108, 111, 114
Chromium	52, 53
Cobalt	59
Copper	65
Lead	206, 207, 208
Manganese	55
Mercury	202
Molybdenum	95, 97, 98
Nickel	62
Selenium	77, 82
Silver	107, 109
Thallium	203, 205
Vanadium	51
Zinc	66, 67, 68
Ruthenium	99
Palladium	105
Tin	118, 120

NOTE: Isotopes recommended for analytical determination are underlined.

Table IV – Stock Standards Concentrations

Analyte	Stock Standard Concentrations									
	XFSPA-221-250	XFSPA-656-250	XFSPA-220-250	XFSMN-26-250A	XFSMN-27-250A,	XFSMN-28-250A	PACE-5	PACE-4B	PACE-28	6020 ISC-OA
Aluminum		20 ug/mL		1000ug/mL				20 ug/mL	1000 ug/L	1,000 ug/mL
Antimony	20 ug/mL				100 ug/mL		20 ug/mL		50ug/L	
Arsenic	20 ug/mL					100 ug/mL		20 ug/mL	50ug/L	
Barium		20 ug/mL				100 ug/mL		20 ug/mL	30ug/L	
Beryllium		20 ug/mL				100 ug/mL		20 ug/mL	20ug/L	
Bismuth		20 ug/mL						20 ug/mL	50ug/L	
Boron		20 ug/mL			100 ug/mL			20 ug/mL	500ug/L	
Cadmium		20 ug/mL				100 ug/mL		20 ug/mL	8ug/L	
Calcium		250 ug/mL		1000ug/mL				250 ug/mL	4000ug/L	1,000 ug/mL
Chromium		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Cobalt		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Copper		20 ug/mL				100 ug/mL		20 ug/mL	100ug/L	
Iron		250 ug/mL		1000ug/mL				250 ug/mL	5000ug/L	1,000 ug/mL
Lead		20 ug/mL				100 ug/mL		20 ug/mL	10ug/L	
Lithium		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Magnesium		250 ug/mL		1000ug/mL				250 ug/mL	1000ug/L	1,000 ug/mL
Manganese		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Molybdenum		20 ug/mL			100 ug/mL		20 ug/mL		50ug/L	20 ug/mL
Nickel		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Palladium			20 ug/mL				20 ug/mL		50ug/L	
Platinum			20 ug/mL				20 ug/mL		50ug/L	
Potassium		250 ug/mL		1000ug/mL				250 ug/mL	5000ug/L	1,000 ug/mL
Selenium		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Silicon	250 ug/mL				500 ug/mL		250 ug/mL		5000ug/L	
Silver		20 ug/mL			50 ug/mL			20 ug/mL	50ug/L	
Sodium		250 ug/mL		1000ug/mL				250 ug/mL	5000ug/L	1,000 ug/mL
Strontium		20 ug/mL				100 ug/mL		20 ug/mL	50ug/L	
Thallium	20 ug/mL					100 ug/mL		20 ug/mL	10ug/L	
Tin	20 ug/mL				100 ug/mL		20 ug/mL		50ug/L	
Titanium		20 ug/mL			100 ug/mL		20 ug/mL		100ug/L	20 ug/mL
Vanadium		20 ug/mL				100 ug/mL		20 ug/mL	100ug/L	
Zinc		20 ug/mL				100 ug/mL		20 ug/mL	500ug/L	
Uranium-238		20 ug/mL						20 ug/mL	50ug/L	

Document Information

Document Number: ENV-SOP-MIN4-0044 **Revision:** 00

Document Title: Preparation of Aqueous Samples for ICPMS Analysis

Department(s): Metals

Previous Document Number: S-MN-I-523-rev.14

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

PREPARATION OF AQUEOUS SAMPLES FOR ICPMS ANALYSIS

Reference Methods: EPA Method 200.8 and EPA SW-846 Method 3020A

Local SOP Number:	S-MN-I-523-rev.14
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-523-rev.13

APPROVALS

Laboratory General Manager — Date: 22 Nov 2017

Laboratory Quality Manager — Date: 24 Oct 2017

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-523-Rev.14

TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to establish a procedure for the digestion of aqueous samples to be analyzed by ICPMS as described in EPA Method 200.8 and EPA SW-846 3020A.

2. Summary of Method

2.1. Inductively coupled plasma mass spectrometry (ICP-MS) is utilized for the determination of metals in solution. The method is applicable to a large number of matrices. Matrices include ground water, aqueous samples, leachates, industrial wastes.

2.2. Aqueous samples are digested in nitric acid and hydrochloric acid at 95°C ± 2°C. Samples requiring dissolved metals analysis must be filtered through a 0.45 micron filter prior to preservation.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. Parameters: Not applicable to this SOP.

4. Applicable Matrices

4.1. This SOP is applicable to aqueous samples, mobility-procedure extracts, and liquid wastes.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. In sample preparation, contamination is of prime concern. The work area, including bench top and fume hood, are regularly cleaned in order to eliminate environmental contamination.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Liquid	Plastic or glass. Pre-cleaned containers are purchased and provided.	Must be acid preserved with Nitric Acid to a pH <2 (5 mL HNO3/L) at time of collection. If samples are received unpreserved, add nitric acid (5 mL/L sample). Some samples require laboratory filtration. These samples must be received unpreserved, and are filtered through a 0.45 µm filter. Preserve samples after filtration.	Room temp	The samples have a shelf life of 6 months from the date of collection.

7.2 If samples are received at pH >2, the samples need to have additional preservative added. This is generally performed in sample receiving upon receipt. The samples are required to equilibrate for 24 hours before conducting digestion. The time of addition of the acid and the time of digestion are documented. The pH is re-verified after the 24 hour time limit. If the sample is still > 2 pH, contact the PM for client notification on how to proceed. If the digestion is conducted, the samples are to be qualified for the pH discrepancy.

7.3 Samples requiring dissolved metals analysis should be filtered in the field through a 0.45 um filter. If the samples are requesting lab filtration, perform the filtration through a 0.45 um filter in the lab and acidify or filter into a nitric preserved container. Check the pH to ensure it is less than 2.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Digestion cups	50 mL polypropylene	Environmental Express or equivalent
Ribbed plastic watch glasses	Ribbed watch glass	Environmental Express or equivalent
Filters	0.45 um	Celltreat or equivalent
Hot Block	Digestion system	Environmental Express or equivalent
Horizon	Laboratory Information System	See master list for current version
Electronic Prep Log	Pace created software to track preparation	

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II water	Verify that background levels of volatile compounds are acceptable by analysis
Concentrated Nitric acid (HNO3)	Trace Metal grade	Store the bottle at room temperature, expires as specified by manufacturer.
Concentrated hydrochloric acid (HCl)	Trace Metals grade	Store the bottle at room temperature, expires as specified by manufacturer.
Standard stock solutions	20 µg/mL	Can be purchased from a commercial supplier such as Spex or Inorganic Ventures. The list of required elements is provided in Section 2 of the ICP/MS Method 6020/200.8 SOP (S-MN-I-492, or equivalent replacement). Solutions are to be stored at room temperature and expire as per the manufacturer.

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP S-MN-Q-264 (or equivalent replacement), Support Equipment.

11.2. Calibrate the thermometer as specified in SOP S-MN-Q-264 (or equivalent replacement), Support Equipment.

11.3. Temperature Logbook

11.3.1. Record the temperature of each hot block daily in the temperature logbook.

11.3.2. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature is checked in different wells of the Hot Blocks such that all wells are evaluated over a period of time.

12. Procedure

12.1. Sample Preparation

12.1.1. Aqueous samples with a turbidity of <1 NTU being analyzed for 200.8 can be analyzed directly without digestion, with the exception of silver. All samples that are regulated under the Clean

Water Act for compliance monitoring purposes must be digested regardless of the turbidity. The preferred method is to digest samples; however, if samples are known to be particularly clean then the turbidity may be checked as an alternative to digestion.

12.1.2. Check the turbidity of each sample and record in the turbidity logbook. If the turbidity is <1 NTU, the sample is now ready for analysis. If the turbidity is > 1 NTU, the sample needs to be acid digested.

NOTE: If a precipitate is formed during acidification, transport, or storage, the sample aliquot must be digested using the following acid digestion procedure prior to analysis.

12.2. Acid digestion of aqueous samples

12.2.1. Transfer a well-mixed 50 mL acid preserved aliquot of the sample to a labeled digest cup. Document the initial volume used in the prep log.

NOTE: Alternate sample volumes may be used as long as the acid and spike additions are adjusted accordingly.

12.2.1.1. Create a method blank and laboratory control sample (LCS) using DI water.

12.2.1.2. If the samples are filtered in the lab for dissolved metals, an associated filter blank must be performed and be digested with the batch of samples filtered. The filter blank is not in substitution of the method blank but in addition to.

12.2.2. Spike the LCS and MS/MSD samples with the appropriate spiking standards.

12.2.3. Add 1.0 mL concentrated nitric acid to each sample. Adjust amount of acid if a smaller sample size was used. If samples originate from WI, starting at this step refer to Attachment I.

12.2.4. Add 0.5 mL concentrated hydrochloric acid to each sample. Adjust amount of acid if a smaller size was used. Cover each digest cup with a ribbed plastic watch glass.

12.2.5. Place samples in a Hot Block at 95 degrees C +/- 2 in the hot block. Document temperature of the Hot Block.

12.2.6. Samples will be gently refluxed for 4 hours and should be monitored to prevent boiling. The samples cannot go dry in this process. If it has gone dry, the sample needs to be removed from the batch and reprepared.

12.2.7. Remove from Hot Block . Document the temperature of the Hot Block.

12.2.8. Allow the digest to cool. Bring up to a final volume of 50 mL with reagent water, cap and mix. Record the final volume. Any deviation in initial or final volume must be recorded. Proper calculation for any dilutions performed during the digestion phase is managed in the LIMS.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	A blank consisting of 50 mL DI water processed through the sample preparation procedure.	Prepared and analyzed with each batch of samples digested or for every 20 samples, whichever is more frequent.	See appropriate analytical SOP.	See appropriate analytical SOP.
Filter Blank	DI water filtered through the same lot of filters used to perform lab dissolved metals filtration	Prepared only with batches of lab filtered dissolved metals, one per batch of 20 or less.	This blank is used to demonstrated the cleanliness of the filters used in the process. The filter blank is required to be less than the RL unless otherwise specified by client or QAPP data quality	Reanalyze to confirm the detection. If the detection is confirmed, refilter DI water using the same filter lot. If the detection is present then the samples are all in question that were filtered with the

			objectives.	same lot. Review the detections in the associated samples and perform the corrective actions as defined for a method blank. Refer to the appropriate analytical SOP.
Laboratory Control Sample (LCS)	Add 0.2 ml of each the ICPMS stock spiking solutions to 50 ml of DI.	A LCS will be digested and analyzed for each SDG, batch or 20 samples prepared, whichever is more frequent.	See appropriate analytical SOP.	See appropriate analytical SOP.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	Add 0.2 ml of each the ICPMS stock spiking solutions to a 50 ml aliquot of sample.	Digested and analyzed 1MS/MSD set for each batch of 20 samples or less prepared. For 200.8, o ne MS/MSD sample must be prepared for each batch. Batches of 11 or more samples need a second MS sample.	See appropriate analytical SOP.	See appropriate analytical SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Method Detection Limit (MDL) Study: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. Periodic performance evaluation (PE) samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. The scope of the method for 3020A has been expanded to include additional metals that require hydrochloric acid for best solubility and stability at the levels of analysis required by ICP-MS. Due to this requirement method 3020A has been modified to include the addition of hydrochloric acid to the digestion.

19.2. Method 3020A states a default nitric acid concentration of 3% but adds room for variability. Our procedure uses a final concentration of nitric acid at 2% and a final hydrochloric acid concentration of 1% for a total acid concentration of 3%. This is consistent with the digestion for EPA Method 200.8.

19.3. Method 3020A has been modified to follow EPA 200.8 given the scope of metals in 200.8 are similar to the scope of metals in 6020A.

19.4. Our procedure uses 50 mL initial and final volumes using the Hot Block digestion system rather than glassware. Acid volumes are scaled accordingly.

19.5. The 4 hour digestion time is a modification of the 2 hours indicated in 200.8 due to the difference in the amount of time it takes to reach ~20 mL using the digestion tubes and watch glasses with the hot block.

20. Instrument/Equipment Maintenance

20.1. Please refer to the specific manufacturer's instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A, 1992.

25.5. U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Wisconsin Procedure 3020A/3050B

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-523-Rev.13	1.**.**.- number format was reformatted back to standard SOP numbering.	18-Apr-2016
S-MN-I-523-Rev.14	LLC Update Removed Uncontrolled Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright.	28Sep2017

Attachment I – Wisconsin Procedure for 3020A and 3050B

Pace Analytical®	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2016 Page 1 of 1
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For Wisconsin solid samples only

ICPMS/ ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

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STANDARD OPERATING PROCEDURE

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

Reference Methods: EPA 6010B, 6010C, 6010D, and EPA 200.7

Local SOP Number:	S-MN-I-313-rev.30
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APPROVALS

Laboratory General Manager — Date: 14 Apr 2017

Laboratory Quality Manager — Date: 14 Apr 2017

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the determination of metals by inductively coupled plasma atomic emission spectroscopy (ICP-AES) as delineated in EPA Method 6010B (Dec. 1996), 6010C (Feb. 2007), 6010D (Jul. 2014) or 200.7 (Rev. 4.4).

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods.

2.2. This method describes the determination of elements by ICP-AES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector (CCD).

2.3. All data is collected by simultaneous measurement. Software is used to measure and apply corrections due to background or inter-element interferences using a variety of techniques. Alternate wavelengths are also monitored for confirmation or to use in correction equations.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to the elements listed in Attachment I.

4. Applicable Matrices

4.1. This SOP is applicable to drinking water, ground water, aqueous samples, liquid samples, leachates, industrial wastes, soils, sludges, sediments, and other solid wastes.

5. Limits of Detection and Quantitation

5.1. The reporting limit (RL) / Limit of Quantitation (LOQ) for all analytes is listed in Attachment I. All current method detection limits (MDL) are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

6.1.1. Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.2. Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A high solids nebulizer is used on all instruments. Internal standards are also used to monitor and correct for physical effects.

6.3. Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions, use of an ionization buffer, or by matrix matching of standards and samples.

6.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber. Regular maintenance and awareness of samples with high concentrations minimize these interferences.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Liquid	Polyethylene containers. Collect dissolved metal samples and filter them immediately through a 0.45-micron filter on-site by the sampler before adding preservative. If samples are filtered at the laboratory, use a polyethylene container and preserve after filtration with HNO_3. Collect total metal samples into a nitric acid (HNO_3) preserved bottle.	Preserve immediately with HNO_3 to bring the pH to <2 For samples received with a pH>2, additional nitric acid must be added upon receipt to dissolve the metals that may have adhered to the sample container. Sample receiving personnel add the additional acid, labels the samples with the amount of acid added, the lot number of the acid, date, time and initials of person that added the acid. NOTE: Do not add HNO3 to exceed 1% of the total volume of the sample container (example: 2.5 mL for 250 mL bottle, 10 mL for 1000 mL bottle). The samples must not be analyzed for 24 hours from acid addition per the Method Update Rules. The final pH is checked and recorded prior to sample preparation.	Store total and dissolved metal samples at room temperature.	The maximum sample holding time for metals is 180 days from sample collection.
Solid	Glass or polyethylene container	N/A	Above freezing but below 6°C.	The maximum sample holding time for metals is 180 days from sample collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Simultaneous ICP-AES	CCD Detector, full wavelength region	Agilent 720 or Agilent 5100/5110.
Desktop computer and printer	Optimized per instrument	Various –matched with instrument
Auto-sampler	Optimized per instrument	Cetac 520, Agilent SPS3, or Agilent

		SPS4.
Peristaltic pump tubing	Various – including but not limited to blue-blue, red-red, white-white, black-black, orange-blue, orange-green	Fisher, Agilent, Environmental Express, SCP, Perkin-Elmer, or equivalent
Refrigerated Circulator	One per instrument	PolyScience or equivalent
Argon gas supply	high-purity grade, 99.99%	House Argon
Mechanical pipettes, and metals-free disposable pipet tips	Various	Eppendorf, Fisherband, or equivalent
Glassware / Plastic ware	Class A volumetric flasks or calibrated non-class A plastic ware	Fisher or equivalent
Disposable digestion cups	50 mL or 100 mL	Environmental Express or equivalent
Epic Pro	Data reporting software	See master list for current version
LimsLink	Data transmission software	See master list for current version
Agilent ICP Expert Software	Agilent Control & Data	See master list for current versions
Filtermate Plunge filters	2 um PTFE	Environmental Express, SC0408

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized Water	ASTM Type II	House E-pure DI water (>17.5 MOhm)
Concentrated Hydrochloric acid (HCl)	Trace Metals grade	Fisher or equivalent
Concentrated Nitric Acid (HNO3)	Trace Metals grade	Fisher or equivalent
Calibration Standard Stock Solutions	Custom blend	Inorganic Ventures or equivalent
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards.	Spex Certiprep or equivalent
– Cesium Ionization Buffer for use with Agilent 720	50,000 PPM	High Purity Standards P/N 1B-CS-B5 or equivalent.
Wavelength Cal Solution - Agilent	Various analytes	Agilent P/N 6610030100
Internal Standards	Yttrium	Inorganic Ventures or equivalent

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Initial Calibration (ICAL)	Instruments must be calibrated at a minimum once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data. See Attachment VI for an example run sequence.	A calibration curve must consist of a blank and at least one calibration standard.	If not met, remake standards, recalibrate, and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after the calibration standards have been analyzed, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of an ICV Solution at each wavelength used for analysis. The Initial Calibration Verification (ICV) Stock Solution(s) must be obtained from a different source than the calibration standards.	± 10% for method 6010B,6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C,and 6010D and below 3% for 200.7.	If the ICV fails, take the following action. Remake the standard accordingly if that is the cause. Re-inject the ICV one more time; if it fails stop all analysis. Perform all necessary instrument maintenance and recalibrate the instrument. Only two injections are allowed back to back, and then the system must be recalibrated.

Continuing Calibration Verification (CCV)	To ensure calibration accuracy during each analytical run, a CCV standard must be analyzed after no more than 10 samples and at the end of the run for each wavelength. The ICV solution can be utilized as the CCV.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	If the requirements for continuing calibration are not met, review for preparation error or instrument drift. A CCV may be repeated once, but a second failure requires re-analysis of any samples bracketed by a failing CCV with the following exception: If the samples bracketed are non-detect and the CCV is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/LCSD/MS/MSD) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples.
6010B/200.7 - Contract Required Detection Limit Sample (CRDLA)	The CRDLA must be analyzed at the beginning of each run for every analyte of interest. The CRDLA is analyzed at or below the RL.	± 40% (or specified by the client)	If the CRDLA fails, the system must be stopped. The CRDLA may be repeated once. If it fails again, perform any necessary maintenance and recalibrate accordingly.
6010C/D – Low Level Initial/Continuing Calibration Verification (LLICV/LLCCV)	The LLICV/LLCCV is named CRDLA for purposes of consistency. It is the same solution. The CRDLA must be analyzed following the ICV at a concentration at or below the RL. Additionally, the CRDLA must be analyzed after samples to cap them for method 6010C or client request. This frequency varies by client. In some cases it is every 10 samples and some cases by batch. The method requires that it be run at least once capping samples. The CRDLA need not be bracketed by a CCV/CCB to be considered valid in most cases. In some instances the CRDLA must be bracketed by a valid CCV/CCB depending on the QAPP. It is best to bracket the CRDLA.	For method 6010C, must be within ± 30% For method 6010D, must be within .± 20%	If the CRDLA fails, it may be repeated once. If it fails again, samples bracketed by a failing CRDLA must be re-analyzed with the following exception. If the samples bracketed are non-detect and the CRDLA is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/LCSD/MS/MSD) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples.
Initial Calibration Blank (ICB)	An ICB must be analyzed immediately following an ICV for each element of interest.	All elements of interest must be evaluated to a criteria of < ½ the absolute value of the RL for method 6010D. All elements of	If the ICB fails, re-pour the sample and analyze a second time. If the ICB is still out of control, re-calibrate.

		interest must be evaluated to absolute value of the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client	
Continuing Calibration Blank (CCB)	A CCB must be analyzed, for each element of interest after every CCV.	All elements of interest must be evaluated to a criteria of < than the absolute value of the RL for 200.7, 6010B, and 6010C.. Depending on the data quality objective of individual client's different criteria may apply. For example data may need to be evaluated to ½ the RL or to the MDL. For 6010D, data must be evaluated to an absolute value of ½ the RL	If the absolute value of an analyte of interest is greater than the RL (or 1/2 RL, if specified), the CCB may be repeated once. If it fails again, all samples bracketed by the failed CCB must be re-analyzed with the following exception. If the sample concentration is greater than 10 times the CCB concentration, or the result is non-detect, the result may be reported. If associated projects are evaluated to the method detection limits per data quality objectives, the detections must be evaluated for data impact and the system evaluated for necessary corrective actions.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.	SIC absolute value must be less than RL.	If SIC fails, re-calculate IEC and re-process data. If sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.
Interelement Correction Standard A (ICSA)	A solution containing high concentrations of Al, Ca Fe and Mg is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked interferent elements are ± 20% and ± the RL for target analytes.	If the initial ICSA fails criteria, it may be re-analyzed once. Also, the ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue. If a bracketing ICSA fails, it may be repeated once, and if it fails again, all affected samples must be re-analyzed.
Interelement Correction Standard AB	A solution containing high concentrations of Al, Ca, Fe and Mg and low to mid-range concentrations of target analytes as	The acceptance criteria are ± 20% for all interferent	If the initial ICSAB fails criteria, it may be re-analyzed once. Also, the ICSAB can be re-processed after

(ICSAB)	outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	elements and target elements and +/- the RL for non-spiked analytes.	appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue... If a bracketing ICSAB fails, it may be repeated once, and, if it fails again, all affected samples must be re-analyzed.

12. Procedure

12.1. Instrument Set up and Operation. Set up and calibrate the instrument per the manufacturer's instruction and individual training provided by senior staff. It is outside the scope of this SOP to provide detailed instruction on instrument and/or software operation. The following highlights common items that are critical to success. Method conditions are stored with each instrument run.

12.1.1. Perform maintenance as needed and record in the daily maintenance log.

12.1.1.1. Ensure rinse is above ¼ full.

12.1.1.2. Ensure waste is below ½ full. Back pressure can cause problems.

12.1.1.3. Clean nebulizer once per month or more if drifting / clogging occurs.

12.1.2. ICP Ignition / Warm-up.

12.1.2.1. Check the gas supply.

12.1.2.2. Confirm the water circulator/chiller is on.

12.1.2.3. Leave instrument on and software up when not in use. Turning things off and on are bad for the instruments.

12.1.2.4. A periodic re-boot of the PC is necessary and recommended at least once per month.

12.1.2.5. Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma.

12.1.2.5.1. Decrease flow to where flow of bubble actually stops or barely moves.

12.1.2.5.2. Turn knob 2 full turns.

12.1.2.6. Ignite plasma while tubing is in a rinse solution.

12.1.2.7. Allow plasma to warm up at least 30 minutes and preferably 60-90 minutes.

12.1.3. Use the warm up time to create the sequence and pour samples.

12.1.3.1. Use Horizon Uploader to copy labels into the sequence.

12.1.3.2. Label all sample tubes so that each sample can be uniquely identified on the rack.

12.1.3.2.1. Alternate red, blue, and black Sharpie so that batches can be readily identified.

12.1.3.3. If any samples in a batch need to be filtered because of suspended material, use an Environmental Express Filtermate. The Method Blank and LCS must also be filtered if any samples are. Record the ID of the Filtermates used.

12.1.4. Pour the standards and start the calibration of the instrument.

12.1.5. Set the system to either a) shut down once complete or b) leave plasma and pump on.

12.1.5.1. If option b is used it is critical that someone be present when run completes – be very careful using this option.

12.1.6. Monitor all initial QC checks. One re-analysis of QC checks is allowed. If initial QC fails twice, make instrument modifications and recalibrate. If checks pass criteria, continue with sample analysis.

12.1.6.1. If ICSA fails, analyze SIC 1-4 and re-process IECs based on the data gathered.

12.1.7. During the sample analysis or after the analysis is completed, transfer valid data into LIMS system using LIMS LINK.

12.1.7.1. Export data from instrument to CSV file.

12.1.7.2. Open LIMSLINK

12.1.7.3. Click open instrument, select CSV file from list, data will import

12.1.7.4. Highlight QC + samples, select "Get LIMS Info"

12.1.7.4.1. Run QC will prompt for Q-Batch # plus standard selection

12.1.7.4.2. Sample data will prompt for SD/PDS source sample.

12.1.7.5. Right click on samples to select/de-select elements

12.1.7.5.1. Be sure to make the appropriate selections in LIMSLNK rather than post-editing in EPIC. This provides for a much smoother experience and minimizes chance for error. If edits must be done in EPIC be sure to make edits prior to uploading new data from LIMSLINK, as this, again minimizes error due to confusion.

12.1.7.6. Highlight samples to upload and select "Export Run to Epic Pro".

12.1.7.7. When complete select "Excel bench sheet"

12.1.7.8. Save the Excel Bench sheet to the instrument folder marked "LIMSLINK RAW DATA and to the DATA REVIEW FOLDER (see below) Use convention of run date (e.g. 032917ICP5) Note discrepancies in the notes section of the run log (including dilutions, QC issues, re-runs, etc).

12.1.8. In LIMS system make final adjustments and add any required footnotes. Print validation lists and complete checklist. Turn data in for validation.

12.2. Documentation for Data Review /Daily File

12.2.1. Documentation is a mix of electronic and paper files. Key data must be stored electronically so that data review may be performed from any location. Some documents are stored in the physical daily folder and archived for easy reference.

12.2.2. Label a physical file with the date.

12.2.3. Record the file name, Q-Batch, and all prep batches on the folder for each run that day (example: 032917ICP5 and 032917ICP5B..

12.2.4. Store printed copies batch worklist reports, prep bench sheets, the original checklist, a printed copy of the IEC Form 10-IN generated from Gandolf, and a printed copy of the run log from LIMSLINK file in this folder. If the data reviewer requests additional printed information place it in this folder as well.

12.2.5. On the G: drive created a new folder for the run labeled using the run name. This folder is created under G:\METALS\Instrument Data\10ICP5\DATA REVIEW\032917ICP5. Substitute ICP4 when using ICP4 and use the file name current to the data being used.

12.2.6. In this folder store electronic copies of the LIMSLINK Raw Data File, Validation Reports from LIMS (labeled using the LIMS Batch # first), scanned copies of the completed checklists (labeled using the LIMS Batch # first), the Instrument QC Report, the IEC Form 10-IN, and a copy of the raw data that is the same as that stored on the X: Drive.

12.2.7. For data validated off-site the electronic copy is signed and data validated in LIMS. The original copies of the checklists are signed off by the data review chemist the next time he or she is in the office and archived in the physical folder.

12.2.8. Label the daily file folder with date, Q-Batch, and instrument information.

12.3. Calibration Standards. There may be some exceptions due to client specific requests for non-routine analysis; however, for most samples the following calibration standards apply.

12.3.1. Calibration levels are set at the levels denoted in attachments II (calibration) and III (calibration verification).

12.3.2. CRDLA is prepared at the RL/LOQ listed in attachment I.

12.3.3. ICSA and ICSAB levels are noted in attachments IV and V and follow the definitions given in ILM05.3.

12.3.4. The acid matrix is typically 4% nitric acid and 5% hydrochloric acid.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI water for liquid samples Resin beads for solid samples	Prepared and analyzed with each group of samples digested. Carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering and analysis.	All elements of interest must be evaluated to a criteria of the absolute value being < ½ the RL for method 6010D. All elements of interest must be evaluated to a critera of the absolute value being< the RL for method 6010,6010B,6010C and 200.7 If the method blank does not contain target analytes at a level that interferes with project-specific DQOs, then the method blank would be considered acceptable.	If MB fails, one reanalysis allowed. If it fails again, affected samples should be re-prepared and re-analyzed with following exceptions: If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate B-flag qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	DI water for liquids and resin beads for solids, spiked with analytes of interest at same level as MS/MSD	Prepared and analyzed for every batch of 20 or less samples digested	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	If the percent recovery for the LCS falls outside the control limits of 80-120% for 6010B, 6010C,and 6010Dor 85-115% for 200.7, one reanalysis of the LCS is allowed. . If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. ***EXCEPTION:*** if LCS fails high and samples are ND, the data may be reported with appropriate qualification.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	The spike is added to a well-mixed aliquot of a selected sample before the digestion (i.e., prior to the addition of	One MS/MSD per batch. If batch consists of more than10 samples for 200.7, an additional MS is required. Clients may have	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria, the MSD sample and associated parent sample need to be flagged.

	other reagents).	requirements that create a higher frequency of MS/MSD samples.		If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Post Digestion Spike (PDS)	Spike is added to the native QC sample at the same concentration as the MS but at the instrument.	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	75-125% for 6010B and 80-120% for 6010C.	Data is provided to data package clients for their evaluation
Internal Standard	The same concentration should be used for standards and samples throughout the entire analytical run.	Introduced automatically with every sample.	70-130% of its true concentration	If the recovery is outside the criteria, sample is reanalyzed at a 5X dilution. If it fails at a 5X dilution, higher dilutions are made until result is within specification.
Serial Dilution	A 1:5 dilution of the sample used for the QC. This is performed at the bench.	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: SD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL. The SD test is not applicable to sample concentrations < 10x the RL. 6010D: 1:5 Dilution of MS, or concentrations 25x > LLOQ in parent sample, results within +/- 20%	Data is provided to data package clients for their evaluation.
Laboratory Filter Blank (FB)	A filtered aliquot of reagent water treated and prepared exactly as all samples when lab filtration is requested.	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of the absolute value of the result being < ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of the absolute value of the result being < the RL for method 60106010B,6010C and 200.7 . . If the FBlank does not contain target analytes at a level that interferes with	If FB fails, one reanalysis allowed. If it fails again, affected samples should be re-analyzed with following exceptions: If sample ND, report sample without qualification; If sample result >10x FB detects, report sample as not impacted by the blank contamination; If sample result <10x FB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

			project-specific DQOs, then the FB would be considered acceptable.	

14. Data Analysis and Calculations

14.1. Inter-element Correction Factor (IEC) = Concentration of apparent concentration (observed) in mg/L / Concentration of Interferent in mg/L.

14.2. The percent recovery of the spike is calculated from the following equation:

$$\% \text{ Recovery} = \frac{(SSR-SR) \times 100}{ST}$$

Where:
SSR = Spiked Sample Result, ug/L or mg/kg dry
SR = Sample Result, ug/L or mg/kg dry
ST = Spike Target, ug/L or mg/kg dry

14.3. The relative percent difference between the MS/MSD can be calculated as follows:

$$RPD = \frac{|(S-D)| \times (100)}{(S+D)/2}$$

Where:
RPD = Relative Percent Difference
S = Original Spiked Sample Value, ug/L or mg/kg dry
D = Second Spiked Sample Value, ug/L or mg/kg dry

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 or 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Instrument Detection Limit (IDL) Study**: An IDL study must be conducted quarterly per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement).

18.4. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.5. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

18.6. **Linear Dynamic Range (LDR) study:** The upper limit of the LDR must be established annually for each instrument and each wavelength utilized. The LDR is determined by analyzing progressively higher standard concentrations of the analyte until the observed analyte concentration remains within +/- 10% of the known concentration of the study. Method 6010D requires that a LDR check sample be analyzed daily prior to any samples. Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent.

19. Method Modifications

19.1. There is considerable variability and confusion in the reference methods concerning blank evaluation. In order to provide consistency, blanks are evaluated as the absolute value compared to either the reporting limit (RL), ½ the RL, or to the method detection limit depending on the method or client QAPP. Data is rejected and re-prepared/re-analyzed based on either the RL or ½ the RL only. If data is being evaluated to the MDL, data is not rejected based on the MDL but rather on the RL or ½ the RL. However, data is qualified with a B-Flag if the absolute value is greater than the MDL in the case where data is evaluated to the MDL and passed criteria for the RL or 1/2 RL.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable for this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Method 6010B.

25.5. Test Methods for Evaluating Water and Solid Waste, SW-846, Method 6010C Update IV, Feb. 2007.

25.6. Test Methods for Evaluating Water and Solid Waste, SW-846, Method 6010D Update V, July 2014.

25.7. Method 200.7 Revision 4.4 , Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-atomic Emission Spectrometry.

25.8. US EPA Contract Laboratory Program Statement of Work ILM05.3, March 2004.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I - Target Analyte List and Reporting Limits (PRL)

26.2. Attachment II - ICP Working Calibration Standard

26.3. Attachment III - ICP Calibration Verification Standard

26.4. Attachment IV - ICSA

26.5. Attachment V - ICSAB

26.6. Attachment VI - Sample Run Sequence

26.7. Attachment VII – Wisconsin Procedures for 3020A/3050B

26.8. Attachment VIII – ICP Linear Ranges

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-313-rev.30	Updated to LLC throughout document Removed uncontrolled Updated attachments II/III/IV/V/VII/VIII Updated sections 1/2/3/4/5/12/14/18/19/25 & Tables 7.1/9.1/10.1/11.1/13.1 Removed form F-MN-I-412-rev.01	14Apr2017

ATTACHMENT I – Target Analyte List and Reporting Limits (PRL)

Element	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	200	10
Antimony	20	1.0
Arsenic	20	1.0
Barium	10	0.50
Beryllium	5.0	0.25
Boron	150	7.5
Cadmium	3.0	0.15
Calcium	500	25
Chromium	10	0.50
Cobalt	10	0.50
Copper	10	0.50
Iron	50	2.5
Lead	10	0.5
Magnesium	500	25
Manganese	5.0	0.25
Molybdenum	15	0.75
Nickel	20	1.0
Potassium	2500	125
Selenium	20	1.0
Silver	10	0.50
Sodium	1000	50
Sulfur	500	25
Thallium	20	1.0
Tin	75	3.75
Titanium	25	1.25
Vanadium	15	0.75
Zinc	20	1.0
Hardness	3300	N/A

ATTACHMENT II –ICP Working Calibration Standard

Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)
Ag	100	1.0	50	2
Al	2,000	0.5	50	20
As	200	1.0	50	4
Ba	200	1.0	50	4
Be	200	1.0	50	4
Ca	2000	0.5	50	20
Cd	200	1.0	50	4
Co	200	1.0	50	4
Cr	200	1.0	50	4
Cu	200	1.0	50	4
Fe	2000	0.5	50	20
K	2000	0.5	50	20
Mg	2000	0.5	50	20
Mn	200	1.0	50	4
Na	2000	0.5	50	20
Ni	200	1.0	50	4
Pb	200	1.0	50	4
S	10000	0.1	50	20
Sb	200	1.0	50	4
Se	200	1.0	50	4
Tl	200	1.0	50	4
V	200	1.0	50	4
Zn	200	1.0	50	4
Mo	200	1.0	50	4
B	200	1.0	50	4
Sn	200	1.0	50	4
Ti	200	1.0	50	4
Si	1000	1	50	20
Li	200	1	50	4
P	200	1	50	4
Sr	200	1	50	4

ATTACHMENT III –ICP Calibration Verification Standard

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	50	1.0	50	1
Al	1000	0.5	50	10
As	100	1.0	50	2
Ba	100	1.0	50	2
Be	100	1.0	50	2
Ca	1000	0.5	50	10
Cd	100	1.0	50	2
Co	100	1.0	50	2
Cr	100	1.0	50	2
Cu	100	1.0	50	2
Fe	1000	0.5	50	10
K	1000	0.5	50	10
Mg	1000	0.5	50	10
Mn	100	1.0	50	2
Na	1000	0.5	50	10
Ni	100	1.0	50	2
Pb	100	1.0	50	2
S	10000	0.05	50	10
Sb	100	1.0	50	2
Se	100	1.0	50	2
Tl	100	1.0	50	2
V	100	1.0	50	2
Zn	100	1.0	50	2
Mo	100	1.0	50	2
B	100	1.0	50	2
Sn	100	1.0	50	2
Ti	100	1.0	50	2
Si	500	1	50	10
Li	100	1	50	2
P	100	1	50	2
Sr	100	1	50	2

ATTACHMENT IV – ICSA

Table 1

Source: CLP SOW ILM 5.3 for 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	5	100	250000
Ca	5000	5	100	250000
Fe	2000	5	100	100000
Mg	5000	5	100	250000

Table 2

For 6010D, may be used with 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	10	100	500000
Ca	5000	10	100	500000
Fe	2000	10	100	200000
Mg	5000	10	100	500000

ATTACHMENT V – ICSAB

Source: CLP SOW ILM 5.3 for 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	5.0	100	250000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	5.0	100	250000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	5.0	100	100000
Mg	5000	5.0	100	250000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

For 6010D, substitute values for Al, Ca, Fe, and Mg from Attachment IV, Table 2 above. This may also be used with 200.7, 6010B, and 6010C.

ATTACHMENT VI – Sample Run Sequence

1. **CAL0**
2. **CAL1**
3. **ICV**
4. **ICB**
5. **CRDLA**
6. **ICSA**
7. **ICSAB**
8. **SIC-1 Fe**
9. **SIC-2 Ca**
10. **SIC-3 Al**
11. **SIC-4 Mg**
12. **CCV**
13. **CCB**
14. **SAMPLE 1**
15. **SAMPLE 2**
16. **SAMPLE 3**
17. **SAMPLE 4**
18. **SAMPLE 5**
19. **SAMPLE 6**
20. **SAMPLE 7**
21. **SAMPLE 8**
22. **SAMPLE 9**
23. **SAMPLE 10**
24. **CCV**
25. **CCB**
26. **SAMPLE 11**
27. **SAMPLE 12**
28. **SAMPLE 13**
29. **SAMPLE 14**
30. **SAMPLE 15**
31. **SAMPLE 16**
32. **SAMPLE 17**
33. **SAMPLE 18**
34. **SAMPLE 19**
35. **SAMPLE 20**
36. **CCV**
37. **CCB**
38. **CRDLA**
39. **CCV**
40. **CCB**

ATTACHMENT VII – Procedure for Wisconsin Samples 3020A/3050B

Pace Analytical	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2015 Page 1 of 1
	Document No.: F-MN-I-411-Rev.02	Issuing Authority: Pace Minnesota Quality Office

For Wisconsin solid samples only

ICPMS/ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

Attachment VIII – ICP Linear Ranges

10ICP5		10ICP4	
Wavelength	**LDR (PPM)**	**Wavelength**	**LDR (PPM)**
Ag 328	5	Ag 328	2.5
Al 237	1500	Al 308	500
As 188	50	As 188	50
B 249	50	B 249	50
Ba 455	30	Ba 585	50
Ba 493*	30		
Be 234	20	Be 234	30
Ca 370	3000	Ca 370	3000
Cd 228	30	Cd 228	50
Co 228	200	Co 228	200
Cr 267	50	Cr 267	50
Cu 327	50	Cu 327	50
Fe 261	200	Fe 261	200
Fe 273*	3000	Fe 273	3000
K 766	500	K 766	50
Li 610	10	Li 610	20
Mg 383	1500	Mg 383	1000
Mn 257	30	Mn 257	30
Mn 293*	200	Mn 293	50
Mo 204	50	Mo 204	50
Na 589	500	Na 589	50
Ni 231	50	Ni 231	50
P 213	50	P 213	50
Pb 220	200	Pb 220	200
S 181	300	S 181	300
Sb 206	50	Sb 206	50
Se 196	50	Se 196	50
Si 251	200	Si 251	200
Sn 189	50	Sn 189	50
Sr 421	5	Sr 421	5
Ti 334	50	Ti 334	50
Tl 190	50	Tl 190	50
V 292	50	V 292	50
Zn 206	80	Zn 206	100

***Used for Interference Correction Only**

Document Information

Document Number: ENV-SOP-MIN4-0056 **Revision:** 00

Document Title: Metals Preparation for Solid samples, Wipes and Filters

Department(s): Metals

Previous Document Number: S-MN-I-460-rev.19

Date Information

Effective Date: 31 Jul 2017

Next Review Date: 31 Jul 2019 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

PREPARATION OF SOLID SAMPLES FOR ANALYSIS BY ICP AND ICP-MS

Reference Methods: EPA 3050B

Local SOP Number:	S-MN-I-460-rev.19
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-460-rev.18

APPROVALS

Laboratory General Manager — Date: 31 Jul 2017

Laboratory Quality Manager — Date: 17 Jul 2017

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-460-Rev.19

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the digestion of solid samples to be analyzed by ICP and ICP-MS as described in EPA Method 3050B.

2. Summary of Method

2.1. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are utilized for the determination of metals in solution. The method is applicable to a large number of matrices.

2.2. The samples are digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are filtered (unless ICPMS) and brought to volume.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. This SOP is applicable to solid samples.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Plastic or glass containers. Pre-cleaned containers are purchased from a supplier.	N/A	Above freezing but below 6°C until digested if samples are to be tested for mercury too	Must be analyzed within 6 months of collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Digestion Cups	50 mL	Environmental Express or equivalent
Filtermate Plunge filters	2 um PTFE SC0408	Environmental Express
Hot Block ™	54 Place Hot Block	Environmental Express
Reflux Caps	Caps with a center hole	Environmental Express or equivalent

Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Hydrogen Peroxide	30% ACS Grade	Fisher brand
Hydrogen Peroxide	30%, Optima Grade for tin only	Fisher brand
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are PACE-67Aand Pace-67B. See 10.1.1.	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.

10.1.1. Metals Stock Standards Table

PACE-67B		PACE-67A	
Element	(mg/L)	Element	(µg/L)
Ca	4000	Si	1000
Fe	4000	Sb	200
Mg	4000	Mo	200
K	4000	Sn	200
Na	4000	Ti	200
Se	200		
Al	4000		
Ba	200		
Be	200		
Bi	200		
B	200		
Cd	200		
S	4000		
Cs	200		
Cr	200		
Co	200		
Cu	200		
As	200		
Li	200		
P	200		
Mn	200		
Pb	200		
Ni	200		
Ag	100		
Sr	200		
Tl	200		

V	200		
Zn	200		
U	200		
Pd	40		
Pt	40		

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

11.2. Calibrate the thermometer as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

12. Procedure

12.1. Sample Preparation

12.1.1. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.1g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestion cup. Alternative sample volume may be used based on sample matrix. Weigh out 3 aliquots for the batch QC sample (background, matrix spike (MS), and matrix spike supplicate (MSD) being sure to weigh them as close to the same weight as possible.

12.1.1.1. Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each.

12.1.1.2. Spike the LCS, MS/MSD using 0.25 mL of each PACE-67A and PACE-67B.

12.1.2. Add 10mL of DI water to each sample.

12.1.3. Add 7.5mL of concentrated HNO3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 2°C and reflux for 70 minutes without boiling. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO3 and re-heat. Repeat this process until no fumes are given off during heating. Record on the digestion log to what samples and how much additional acid was added.

Note: record initial Hot Black temperature in the digestion log.

12.1.4. Cool the sample 10 minutes. Add 2.5mL of 30% hydrogen peroxide. Cover with reflux cap and return to the Hot Block for warming which will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Heat until effervescence subsides for a total of 10 minutes. Cool the samples in the plastic cups.

Note: use Optima grade hydrogen peroxide if the analysis of tin (Sn) is required. Tin is used as a stabilizer in the ACS grade of hydrogen peroxide.

12.1.4.1. If effervescence does not subside, continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note in the comments section of prep sheet the additional aliquots.

12.1.4.1.1. NOTE: Do NOT add more than a total of 10mL hydrogen peroxide.

12.1.5. Add 5mL of concentrated HCl, return the sample to the Hot Block and reflux for an additional 15 minutes without boiling.

12.1.6. Remove samples from Hot Block and record final temperature in digestion log. Allow samples to cool. Bring samples up to a final volume of 50 ml with DI water. Invert several times for good mixing. FOR ICP-MS sample prep, cap and label samples for analysis – do not filter if analyzed by ICPMS.

12.1.7. For ICP-AES, samples may be allowed to sit overnight while solid materials settle out or samples may be filtered. If filtered, use FilterMate plunge filters following manufacturers instructions. If

samples are filtered all QC samples including the method blank and laboratory control sample (LCS) must also be filtered.

12.1.7.1. Note: The method modifications that have been utilized have been defined in the above process have been demonstrated effective in MDLs, DOCs, successful PTs, and ongoing precision and accuracy data samples.

12.2. Documentation

12.2.1. Digestion Logbook

12.2.1.1. Record the necessary information in the digestion log book including sample ID, initial and final volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used, including spike solutions and LCS solutions.

12.2.1.2. Also include any additional comments if needed.

12.2.2. Temperature Logbook

12.2.2.1. Record the temperature of each hot block daily in the temperature logbook.

12.2.2.2. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the Hot Blocks such that all wells are evaluated over a period of time.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Preparation Blank	A clean matrix similar to the samples. For solids, 1.0 grams of resin beads. For wipes, use a new Ghost Wipe.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Laboratory Control Sample (LCS)	For solids, weigh 1.0 gram of resin beads. Spike with appropriate spiking solutions.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Weigh out similar amounts of soil as the parent sample; be sure to weigh QC sample and MS/MSD samples as close as possible. Spike with appropriate spike solutions and record in digestion log.	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch.	See appropriate analysis SOP.	See appropriate analysis SOP.
Duplicate (DUP)	In some cases the client may request a duplicate in lieu of an MSD. This is weighed out in similar amount (as close as possible) to the background sample.	As requested.	See appropriate analysis SOP.	See appropriate analysis SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Part of the reason for this modification is better performance for silver and antimony. PT samples are analyzed regularly to validate that the modifications are effective. Per the method, the nitric acid and peroxide amounts are varied based on the sample reaction and this is the case with the Pace method. Overall, the Pace digestion ends up with a higher total acid concentration.

19.2. The final volume for the Pace method is 50 mL, opposed to 100 mL for the reference method.

19.3. Samples are processed using the Hot Block digestion system employing metals free disposable plastic ware rather than glass beakers.

20. Instrument/Equipment Maintenance

20.1. Please refer to the specific manufacturer's instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3. Logs are kept daily for each hot block, monitoring temperature. The temperature probe is varied daily so that each individual hot block sample cell is monitored to ensure consistency across the block.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Wisconsin Procedure 3020A/3050B

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-460-Rev.19	"And Wipes" removed from SOP title throughout. Section 4.1: "and wipes" deleted. Table 9.1: "or equivalent" added to vendor item description of digestion cups and reflux caps. Ghost wipes row deleted. Filtermate plunge filters description updated. Table 10.1: ICP spike updated as metals spike with updated solution identification information provided. ICP MS Spike row deleted. Table 10.1.1, ICP Stock Standards Table, deleted. Table 10.1.2 renumbered, and renamed "Metals Stock Standards Table, with updated element and unit of measurement values. Table 10.2 deleted. Section 10.2.1 deleted. Section 12.1.1: sample portion changed from 1.5 g to 1.1 g. Section 12.1.1.1 deleted Text regarding wipes removed from 12.1.1.1.1, as renumbered. Section 12.1.1.2, previously 12.1.1.1.2, appended with "MS/MSD using 025 mL of each PACE-67A and PACE-67B. Section 12.2.1 deleted. Subsequent sections renumbered. Section 12.2.1.1: "supporting equipment" added following "prep analyst". Table 13.1: instructions regarding Ghost wipe use deleted from LCS row. Attachment I – updated to current revision Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled" to the statement of copyright.	19June2017

Attachment I – Wisconsin Procedure for 3020A and 3050B

Pace Analytical	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2015 Page 1 of 1
	Document No.: F-MN-I-411-Rev.02	Issuing Authority: Pace Minnesota Quality Office

For Wisconsin solid samples only

ICPMS/ ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

Document Information

Document Number: ENV-SOP-MIN4-0062 **Revision:** 00

Document Title: Extractable Base/Neutral and Acid Organic Compounds in Liquid, Solid, and TCLP Matrices by Gas Chromatography/Mass Spectrometry Capillary Column Technique

Department(s): SVOA

Previous Document Number: S-MN-O-436-rev.28

Date Information

Effective Date: 10 Jul 2018

Next Review Date: 10 Jul 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

EXTRACTABLE BASE/NEUTRAL AND ACID ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE FOR WATER, SOIL AND WASTE DILUTION

Reference Methods: SW 846 8270C and 8270D

Local SOP Number:	S-MN-O-436-Rev.28
Effective Date:	Date of Final Signature
Supersedes:	S-MN- O-436-Rev.27

APPROVALS

Laboratory General Manager — Date: 10 Jul 2018

Laboratory Quality Manager — Date: 02 Jul 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-436-Rev.28

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to set forth the procedure used for the determination of a number of organic compounds that are partitioned into an organic solvent and are amendable to gas chromatography/mass spectrometry by EPA SW-846 8270C and 8270D.

2. Summary of Method

2.1. A measured amount of sample is extracted, dried, and concentrated to a specific final volume, and analyzed by GC/MS. Qualitative identification of the analyte of interest in the extract is performed using the retention time and relative abundance of at least two characteristic masses. Quantitation is performed using the internal standard technique with a single characteristic mass in coordination with the average relative response factor from the initial calibration.

2.2. This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

2.3. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Under the alkaline conditions of the extraction step, a-BHC, g-BHC, endosulfan I and II, and endrin are subject to decomposition. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to semivolatile organic compounds.

4. Applicable Matrices

4.1. This SOP is applicable to all types of water, oil, soil, and solid waste matrices.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 330-1700 ug/kg for soils (see Table IIa and IIb for each compound). The reporting limit (LOQ) is 10-160 ug/L for waters with some analytes having an RL raised above 10 ug/L. All current RLs and MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Matrix interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

6.3. An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which contains the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil	4 oz amber glass bottles with Teflon-lined lids	Unpreserved	The samples must be refrigerated above freezing but below 6°C from the time of collection until extraction. The extracts must also be kept refrigerated above freezing but below 6°C until analysis.	Soil samples must be extracted within 14 days from date of collection. The sample extracts must be analyzed within 40 days of sample extraction.
Water	1 liter glass amber	Unpreserved	Above freezing but below 6°C	Must be extracted within 7 days from the date collected.
Leachate. See 7.1.1.	Glass container	Unpreserved. The TCLP extraction fluid is at a pH of 5.	Above freezing but below 6°C	Must be extracted within 7 days from the date leached.

7.1.1. See SOP S-MN-I-312 (or equivalent replacement) for TCLP.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Syringe	10 µL, Hamilton or equivalent	Fisher/Hamilton 80390
Agilent gas chromatograph	Model 5890 (or equivalent)	Agilent
Agilent mass spectrometer	Model 5972 (or equivalent)	Agilent
Agilent Autosampler	Model 7673A (or equivalent)	Agilent
Column	Zebron ZB-Semivolatiles30 m x 0.25 mm x .50 um (ID) bonded-phase silicone coated fused silica capillary column, 0.25 – 0.5 µm film thickness (or equivalent)	#Phenomenex /7HG-G027-17
Chemstation	Data Acquisition Software	See master list for current version

Target	Data Processing Software	See master list for current version
EpicPro (Horizon)	Data Reporting Software	See master list for current version
Gandalph	Data Packaging Software	See master list for current version

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
Methylene Chloride	Optima grade	Fisher Scientific or equivalent
Acetone	Optima grade	Fisher Scientific or equivalent
8270 Mega Mix	500-1000 µg/mL	Restek, 31850
605 Benzidine	2,000 µg/mL	Restek, 31030
Benzoic Acid	2,000 µg/mL	Restek, 2-0140-1
Acid Surrogate Mix	10,000 µg/mL	Restek, 31086
B/N Surrogate Mix	5,000 µg/mL	Restek, 31087
Custom 8270 calibration	2000 µg/mL	Phenova, AL0-130213
605 Benzidines	2000 µg/mL	Phenova, AL0-101501
Benzoic Acid	2000 µg/mL	Phenova, AL0-101246
Internal Standard Mix	4000 µg/mL	Accustd, Z-014J-PAK
DFTPP – Decafluorotripheylphosphine	1000 ug/mL	Accustandard, M-625-TS-20X, or equivalent

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Used/Vendor	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
8270-SS	Acid Surrogate Mix (Restek)	5.0 mL	Acetone	37.5 mL	50.0 mL	1000 µg/mL
	B/N Surrogate Mix(Restek)	7.5 mL				750 µg/mL
8270-SPK	Custom 8270 calibration (Restek)	10mL	Acetone	80 mL	100 mL	100 µg/mL
	605 Benzidines(Restek)	5mL				
	Benzoic Acid(Restek)	5mL				
Initial Calibration (ICAL) Stock Standard	8270 Mega Mix (Restek)	0.8 mL	Methylene Chloride	2.6 mL	5 mL	80-160 µg/mL
	605 Benzidine (Restek)	0.4mL				160 µg/mL
	Benzoic Acid (Restek)	0.4 mL				160 µg/mL
	8270-SS	0.8 mL				120/160 µg/mL
The initial calibration verification (ICV) (also called 8270 external check)	Custom 8270 calibration (Phenova)	0.040 µg/mL	Methylene Chloride	0.800mL	1.0 mL	80 µg/mL
	605 Benzidines(Phenova)	0.040 µg/mL				80 µg/mL
	Benzoic Acid(Phenova)	0.040 µg/mL				80µg/mL
	8270-SS	0.080 µg/mL				60-80 µg/mL
	Internal Standard Mix	0.010 µg/mL				40 µg/mL
Continuing Calibration Verification (CCV) Standard	8270-6	0.5	Methylene Chloride	0.5mL	1.0mL	40-80 ug/mL
Tune Standard	DFTPP – Decafluorotripheylphosphine	0.5mL	Methylene Chloride	9.5mL	10 mL	50ug/mL

10.3. The initial calibration working standards should be prepared as follows using the stock standard:

Solution Name	Conc. of Parent Sol'n (µg/mL)	Aliquot Volume (mL)	Internal Std (µL)	Diluent Vol. (mL)**	Final Conc. (µg/mL)
8270-8	80-160	1.0	10	To 1.0	80-160
8270-7	80-160	0.750	10	To 1.0	60-120
8270-6	80-160	0.500	10	To 1.0	40-80
8270-5	80-160	0.250	10	To 1.0	20-40
8270-4	80-160	0.125	10	To 1.0	10-20
8270-3	80-160	0.0625	10	To 1.0	5-10
8270-2	80-160	0.03125	10	To 1.0	2.5-5
8270-1	80-160	0.00625	10	To 1.0	0.5-1

** Diluent is Methylene Chloride.

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Initial Calibration. See 11.2.	Prior to the analysis of samples and after tuning criteria have been met (See table 1). The system should be set up to analyze the eight calibration levels from section 10.3.	The %RSD should be less than or equal to 15% for each target analyte for 8270C and 20% for 8270D, except the Calibration Check Compounds (CCC) for 8270C only (see Table V) which MUST be less than 30%. See 11.2.4 note. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. See 11.2.5 for further 8270C criteria. The reporting limit standard must be evaluated following the initial calibration by requanting the standard against the passing ICAL. Per 8270D, the calibration standard equivalent to the reporting limit is to be within ±30% for linear regression curves, for MN work MPCA requires that the reporting limit verification be within 40% for all compounds for all curve fits.	When target analytes are >15% for 8270C or >20% for 8270D RSD, performing a linear regression (0.990 or better for 8270C and 8270D) or a weighted least squares regression (0.99 or better) must be employed to achieve linearity. It should be noted that clients may specify other criteria. The initial calibration is valid only after the requirements listed in this table and in section 11.2.5 have been met or justification is given that would support valid generation of data. Note: Sample analysis can only begin after these criteria are met.
Continuing GC/MS Calibration Verification Or External Check	If the External Check is not the CCV it must, at a minimum, be analyzed after the initial calibration for verification	70-130% for 8270C and 70-130% for 8270D unless client specific	See 11.3

11.2. Initial Calibration

11.2.1. Inject an aliquot (1-3uL) of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table IV).

11.2.2. Calculate response factors (RFs) for each compound in each calibration level using equation 1 in 14.1.

11.2.3. Calculate the percent relative standard deviation for each compound using equation 2 in 14.2.

11.2.4. Note: For GC/MS calibration, Method 8270C requires 15% RSD as evidence of sufficient linearity to employ an average response factor. For the following compounds, some project QAPPs may allow 25% RSD:

Benzyl alcohol	4-Chloroaniline
Hexachlorocyclopentadiene	3,3-Dichlorobenzidine

11.2.5. For 8270C: A system performance check compounds must be evaluated to ensure that minimum average RFs are met before the calibration curve is used.

11.2.5.1. The System Performance Check Compounds (SPCCs) (See Table VI) have a minimum acceptable average RF of 0.050.

11.2.5.2. These SPCCs typically tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

11.3. Continuing GC/MS Calibration Verification or External Check

11.3.1. The CCV is valid only after all the criteria has been met:

11.3.1.1. Compare the response factor data from the standards every 12 hours with the average response factor from the initial calibration and calculate the percent difference using equation 4 in 14.3.

11.3.1.1.1. For 8270C: The %D MUST be less than or equal to 20% for each Calibration Check Compounds (CCC) (see standard traceability SOP ALL-Q-025, or equivalent replacement).

11.3.1.1.2. For 8720C: If the CCC compounds are not on the list being evaluated, all analytes of interest must have a %D less than 20%.

11.3.1.1.3. For 8270C: If the CCC compounds are on the list being evaluated, all other target analytes must be evaluated utilizing a criterion of 40% with technical justification and footnoting of sample data to all outliers.

11.3.1.1.4. To ensure internal standard recoveries from samples that run after the Ical under the same folder go off the Ical and not the ICV, process the ICV as a sample. To see if the ICV meets passing requirements pull the ICV file into a batch.b along with the method and requant the ICV as a continuing calibration.

11.3.2. The internal standard responses and retention times in the CCV must be evaluated immediately after or during data acquisition.

11.3.2.1.1. If the retention time for any internal standard changes by more than 30 seconds from the midpoint calibration standard of the initial calibration, the analytical system must be inspected for malfunctions and corrections must be made.

11.3.2.1.2. If the EICP area for any of the internal standards changes by a factor of two, (-50% to +100%) from the midpoint calibration standard of the initial calibration, the MS must be inspected for malfunctions and corrections must be made.

11.3.2.1.3. Internal standard recoveries out low (high bias) – if compounds associated with the internal standard(s) that are outside the control limits are non-detect, the sample can be reported without re-analysis, however, if the outlier is not indicative of a system drift (i.e. If only one sample has internal standard drift, which is dissimilar from other samples around the injection time), re-analysis should be performed to rule out matrix effects.

11.3.2.2. Internal standard recoveries out high (low bias) – re-analysis should be performed assuming there is sufficient sample volume remaining. Appropriate footnoting practices are also observed.

11.3.2.3. For 8270C: The CCV is valid only after both the %D (20%) for CCC compounds, and the minimum RF (0.05) for SPCC have been met or justification is given that would

support valid generation of data. Only after both these criteria are met may sample analysis begin.

11.3.2.4. For 8270D, the CCV is valid after the 20 %D and minimum response criteria in Attachment VII has passed criteria. Due to the number of analytes being analyzed, some compounds may fail to meet the 20% criteria. Any detections reported in the samples need to be within the 20% D criteria on the associated CCV. To report non detect compounds in samples up to 20% of the compounds in the CCV, the value may be outside 20% D criteria but it must be ≤ 40% D. If greater than 20% of the analytes fail the 20% criteria, corrective action must take place prior to sample analysis. For Arizona samples the reporting limit must be ± 20% of the true value.

12. Procedure

12.1. Sample Preparation

12.1.1. EPA method 3550, Sonication Extraction Technique.

12.1.1.1. Method -Attachment I.

12.1.1.2. Extraction bench sheet - Attachment II.

12.1.2. EPA method 3580A, Waste Dilution Technique.

12.1.2.1. Method - Attachment III.

12.1.2.2. Extraction bench sheet - Attachment IV.

12.1.3. EPA method 3520 Continuous Liquid Liquid Extraction

12.1.3.1. See Pace SOP's S-MN-O-496 and S-MN-O-539

12.1.4. EPA method 3510 Separatory Funnel Extraction

12.1.4.1. See Pace SOP S-MN-O-566

12.2. GC/MS Operating Conditions

12.2.1. The recommended GC/MS operating conditions:

Mass Range:	35-500 amu
Scan Time:	1 sec/scan
Initial Temperature:	45° C
Temperature Program:	45-320°C at 20° C/min
Final Temperature:	320°C
Injector Temperature:	275° C
Transfer Line Temperature:	300° C
Source Temperature:	210 to 250° C
Injector:	Splitless
Sample Volume:	1 µL
Carrier Gas:	Helium at 30 mL/min

NOTE: These values may change to optimize the efficiency of the GC/MS system.

12.3. GC/MS Hardware Tuning

12.3.1. Each GC/MS system must be hardware-tuned to meet the criteria in Table I for a 50 ng injection of decafluorotriphenylphosphine (DFTPP).

12.3.1.1. DDT, Pentachlorophenol, and Benzidine should also be evaluated during the tuning process.

12.3.1.2. Whenever the laboratory takes corrective action which may change the tuning criteria for DFTPP (e.g., ion source cleaning or repair, etc.) the tune must be verified irrespective of the 12-hour tuning requirements.

12.3.1.3. Analyses should not begin until all these criteria are met.

12.3.2. The twelve-hour time period for GC/MS system tuning and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of compliant tune. The time period ends after twelve hours have elapsed according to the system clock.

12.3.2.1. The analysis of DFTPP may be performed by:

12.3.2.1.1. Injection of 50 ng of DFTPP

12.3.2.1.2. By adding 50 ng to continuing calibration standard.

12.4. GC/MS Sample Analytical Procedure

12.4.1. Sample Analysis

12.4.1.1. Extracts may be screened on a GC/FID using the same type of capillary column. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

12.4.1.2. The one mL extract obtained from sample preparation laboratory should be fortified with 10 µL of internal standard solution just prior to analysis such that 40 ng of each internal standard is injected on the column.

12.4.1.3. Analyze each extract by GC/MS by injecting 1 µL onto the column.

12.4.1.4. Store the extracts above freezing but below 6°C protected from light in crimp-top vials equipped with unpierced Teflon lined septa.

12.4.2. Qualitative Analysis - Target Analytes

12.4.2.1. Two criteria must be satisfied to verify the identifications of compounds in the sample:

12.4.2.1.1. Elution of the sample component at the same GC relative retention time as the standard component. The sample component RRT must compare within ± 0.06 RRT units of the RRT of the preceding CCV.

12.4.2.1.2. Correspondence of the sample component and standard component mass spectra.

12.4.2.2. For comparison of standard and sample component mass spectra, mass spectra obtained on each PACE GC/MS system are utilized from the preceding CCV.

12.4.2.3. The requirements for qualitative verification by comparison of mass spectra are as follows:

12.4.2.3.1. All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.4.2.3.2. The relative intensities of must agree within plus or minus 30% between the standard and sample spectra.

12.4.2.3.3. Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

12.4.2.4. Refer to Attachment V for large detections quanting to zero.

12.4.2.5. Refer to Attachment VI for j flag hits vs noise.

12.4.3. Quantitative Analysis - Target Analytes

12.4.3.1. The internal standard used shall be the one nearest the retention time to that of a given analyte. See Table III for guidelines.

12.4.3.2. The EICP area of characteristic ions of analytes listed in Tables IV and VII are used.

12.4.3.2.1. Secondary ions may be used if interferences are present.

12.4.3.2.2. The area of a secondary ion cannot be substituted for the area of a primary ion unless an average response factor is calculated using the secondary ion.

12.4.3.3. The average response factor from the initial calibration is used to calculate the concentration in the sample. All soil samples are corrected for moisture by the LIMs unless there are state or contractual differences. See equation 5 in 14.4.

12.4.4. Qualitative Analysis - Non-Target Analytes (TICs)

12.4.4.1. A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST05 Mass Spectral Library should be used.

12.4.4.2. Up to 10 substances (Contract Specific) of greatest apparent concentration not listed in Table II, Table IIA, Table IIB (depending on the client request) for the combined base/neutral/ acid fraction shall be tentatively identified via a forward search of the NIST05 mass spectral library. (Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion).

12.4.4.3. If in the opinion of the analyst, no valid tentative identification can be made, the compound should be reported as unknown.

12.4.4.4. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e. unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

12.4.5. Quantitative Analysis - Non -Target Analytes (TICs)

12.4.5.1. For quantitation, the nearest internal standard free of interferences shall be used.

12.4.5.2. When calculating concentration for non-calibrated components, total area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard.

12.4.5.3. An average response factor of 1.0 is to be assumed.

12.4.5.4. The value from this quantitation shall be qualified as estimated and the nearest resolved internal standard used to quantitate shall be identified.

12.4.5.5. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Leachate = Solution used to leach samples. A method blank is leached with each set and is continued through extraction process. Soil = Sand (baked, and solvent washed) Water = Deionized water The initial volume/weight used for the method blank must be approximately equal to the sample aliquots being processed.	Method blank analysis must be performed at the following frequency: Once each batch. With every twenty (20) samples of similar concentration and/or sample matrix. Whenever samples are extracted by the same procedure.	Less than the reporting limit of any single target analyte, or to the MDL for data quality objectives.	If a laboratory reagent blank exceeds criteria, the laboratory must consider the analytical system out of control. The source of the contamination investigated and appropriate corrective measures must be taken and documented. All sample processed with an unacceptable method blank must be reprepared (if additional raw sample is available) and reanalyzed; unless they are non-detect for analytes present in the method blank or greater than 10 times the concentration found in the associated method blank.

				The laboratory will report ALL sample concentration data as UNCORRECTED for blanks. Exceptions: For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	A control matrix injected with target analytes. The control matrices include: Leachate = Solution used to leach samples Water = Deionized water Soil = Sand (baked, and solvent washed)	Each analytical batch or once per 20 samples, whichever is more frequent. A LCSD is prepared ONLY when an MS/MSD is not available to analyze.	Limits are established by the laboratory and are updated annually by the QA office. The most current limits can be found in LIMS. If an LCSD is performed <20% relative percent difference (RPD). NELAC requirements for how many analytes can be out per analyte list can be found in the Quality Assurance Manual. Certain clients may have tighter criteria per QAPP or Tech. Specs.	If a laboratory control sample exceeds criteria all samples in the batch with no detections can be reported. If a laboratory control sample is below the required criteria all samples must be reprepared (if additional raw sample is available) and reanalyzed. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. If the LCSD is within criteria, but the RPD fails treat like a biased high LCS recovery.
Surrogates			Limits are established by the laboratory and are updated annually by the QA office. The most current limits can be found in LIMS. It is our general practice to allow one surrogate to be outside criteria. Some clients may have tighter criteria per QAPP or Tech specs.	Surrogates out due to matrix can be reported as such. Samples with surrogates exceeding criteriacan be reported if they are non-detect, samples with hits will have to be reprepared (if additional raw sample is available) and reanalyzed. Samples with surrogates below the required criteria have to be reprepared (if additional raw sample is available) and reanalyzed.
Matrix Spike (MS)/ Matrix Spike Duplicate (MSD)	Prepared like the laboratory control sample with a designated sample being the control matrix.	Performed with every twenty (20) samples of similar concentration and/or similar sample matrix if sufficient sample	The recoveries and RPD should fall between the limits updated annually by the QA office. The most current limits can be found in LIMS.	If a recovery fails criteria, it is the responsibility of the analyst and supervisor to determine if the outliers are related to the matrix of the unspiked sample. For Minnesota Admin Contract

		is provided by the client. (Note: If sample is not available, an LCS and an LCS duplicate should be analyzed.) Analyzed at the same dilution as the original unspiked sample.		clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying properly.

14. Data Analysis and Calculations

14.1. Calculate response factors (RFs) for each compound in each calibration level using equation 1:

Equation 1: $$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

14.2. Calculate the percent relative standard deviation using equation 2:

Equation 2: $$\%RSD = \frac{SD}{\overline{RF}} x100$$

Where:
$\overline{RF}$ = Mean of the Response Factors mentioned above.
SD = Standard Deviation of initial response (Equation 3).

Equation 3: $$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_1 - \overline{RF}\right)^2}{n-1}}$$

Where:
RF1 = Each individual response factor
$\overline{RF}$ = Mean of the Response Factors mentioned above.
n = Number of response factors

14.3. Calculate the percent difference using equation 4:

Equation 4: $$\%Difference = \frac{(RF_i - RF_c)}{RF_i} x100$$

Where:
RFi = Average response factor from initial calibration
RFc = Response factor from current verification check standard

14.4. Calculate the concentration in the sample using equation 5:

Equation 5: $$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF})(W_o)(V_i)}$$

Where:
A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
W_o = Volume of sample extracted in liters (or weight in g)
V_i = Volume of extract injected (mL)
V_t = Final volume of total extract
$\overline{RF}$ = Average response factor from initial calibration

14.5. The recoveries and RPDs are calculated as follows and are used to verify that the precision and bias of the analytical process are within control limits.

Equation 6:

$$\%\,\text{Recovery} = \frac{SSR - SR}{SA} x100$$

Where:
SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

Equation 7:

$$RPD = \frac{|A - B|}{(A + B)/2} x100$$

Where:
RPD = Relative Percent Difference
A = First Sample Value
B = Second Sample Value (duplicate)

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See tables in section 11 & 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4. Periodic **performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.2. Basic Maintenance:

20.2.1. Injection port maintenance

20.2.2. Changing of Septa – daily or as needed

20.2.3. Evaluation/cleaning/replacement of liners – as needed. Daily evaluation.

20.2.4. Evaluation/changing of Gold seal – evaluate daily when liners are being cleaned/replaced. Mass spec tunes will be able to give you a good indication of the condition of the gold seal. Certain compounds are more susceptible to degradation in the injection port.

20.2.5. Clipping of column – as needed. Clipping the column will often decrease tailing, revive some sensitive compounds and help prolong column life.

20.2.6. Split line/Weldment cleaning

20.2.6.1. Frequency– monthly or as needed. Samples are direct injected so it will depend on the samples you are analyzing. If samples are high in petroleum and/or oily matrixes it may require you to clean your split lines and weldment more often. Typical signs of split lines requiring cleaning is poor back end (back half of your chromatogram) response and breaking down of compounds (DDT, endrin, etc.)

21. Troubleshooting

21.1. The tune should help serve as a troubleshooting guide for column degradation and standard issues. For example: if the instrument has trouble passing calibrations consistently and PCP is present and DDT is not breaking down excessively then the result of the calibration issues is more likely due to standard degradation and not the system itself. Conversely if the system has trouble consistently passing calibrations or recoveries are inconsistent from run to run and PCP is not present and/or DDT is breaking down the result is more than likely due to a system issue. This would cue the analyst to perform different maintenance including injection port maintenance, clipping column, cleaning split lines, cleaning weldments, or replacing columns.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data

Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 8270C.

25.5. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Method 8270D

25.6. USEPA Contract Laboratory Program Statement of work for Organics Analysis, OLMO3.0, Exhibit D Semivolatiles.

25.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, March 2003, Revision 3, Method 8000C.

25.8. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition and Update IV, Method 3550C, 2007.

25.9. Test Methods for Evaluating Solid Waste, SW-846, Final Update III, Method 3520C.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table I: DFTPP Key Ions and Ion Abundance Criteria

26.2. Table IIa: Analytes and Quantitation Limits – Standard 8270 Reporting Limit

26.3. Table IIb: Analytes and Quantitation Limits – Additional Extractable 8270 Analytes

26.4. Table IIc: Analytes and Quantitation Limits –Extractable 8270 TCLP Analytes

26.5. Table III: Internal Standard Method of Quantitation

26.6. Table IV: Characteristic Ions for Target Compounds

26.7. Table V: Calibration Check Compounds (CCC)

26.8. Table VI: System Performance Check Compounds (SPCC)

26.9. Table VII: Characteristic Ions for Internal Standards

26.10. Table VIII: Characteristic Ions for Surrogates
26.11. Table IX: Surrogate Compounds
26.12. Attachment I: Sonication Extraction Technique – Benchsheet (example)
26.13. Attachment II: Waste Dilution Technique – Method
26.14. Attachment III: Instrument Run Log (example)
26.15. Attachment IV: Method 8270D Minimum Response Factor Criteria
26.16. Attachment V: Large Hits Quanting to Zero
26.17. Attachment VI: J Flag Hits vs Noise

27. Revisions

Document Number	Reason for Change	Date
S-MN-O-436-Rev.27	Replaced reference to training SOP with new local number in 18.3. Table 10.1 – removed 1-methylnapthalene, carbazole, mix #7 benzidine, acid comp mix, B/N comp mix, comp mix #2; added custom 8270 calibration, 605 benzidines, and benzoic acid. Table 10.2 – Added 8270-SPK; edited standards used, amounts, and final concentrations for the ICV; edited the standard used for the CCV and Tune Standard rows. Added Section 11.3.1.1.4.	12Jan2018
S-MN-O-436-Rev.28	Table 10.2 – updated 8270-SPK vendors, standard amount, and solvent volume Added "For WI samples…" exception to Table 13.1, MB row, Corrective Action column.	29Jun2018

TABLE I: DFTPP Key Ions and Ion Abundance Criteria for 8270C

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	Mass 69 relative abundance
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	Greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

Tailing analysis

Pentachlorophenal less than or equal to 5.0

Benzidine less than or equal to 3.0

DDT Degradation % Breakdown analysis Summary

4,4-DDE less than or equal to 20.0

4,4-DDD less than or equal to 20.0

4,4-DDD + DDE less than or equal to 20.0

TABLE I Continued: DFTPP Key Ions and Ion Abundance Criteria for 8270D

Mass	Ion Abundance Criteria
51	10.0 - 80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	Mass 69 relative abundance
70	less than 2.0 percent of mass 69
127	10.0 - 80.0 percent of mass 198
197	less than 2.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 60.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than 24% of mass 443
442	Greater than 40.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

Note: Tighter criteria may be used such as the criteria listed in 8270C for the above abundances

Tailing analysis

Pentachlorophenol and Benzidine less than or equal to 2.0

DDT Degradation % Breakdown analysis Summary

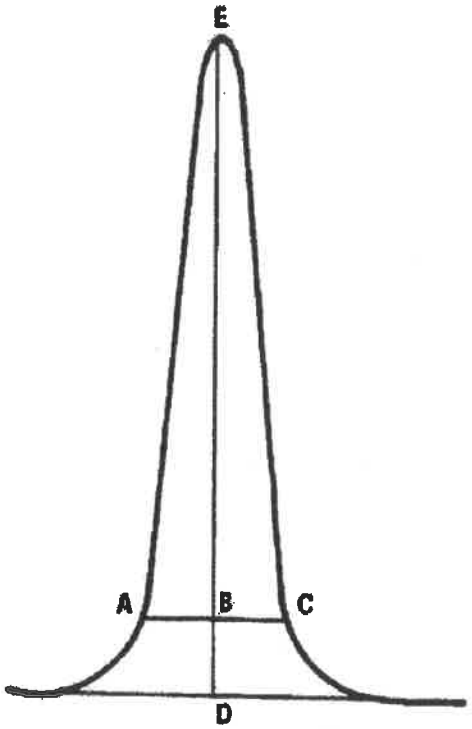
4,4-DDE less than or equal to 20.0

4,4-DDD less than or equal to 20.0

4,4-DDD + DDE less than or equal to 20.0

TABLE I Continued: DFTPP Key Ions and Ion Abundance Criteria for 8270D

FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

TABLE IIa: Analytes and Quantitation Limits-Standard 8270C/D reporting list

Analyte	CAS #	PQL (ug/kg)
1,2,4-Trichlorobenzene	120-82-1	330
1,2-Dichlorobenzene	95-50-1	330
1,2-Diphenylhydrazine	122-66-7	330
1,3-Dichlorobenzene	541-73-1	330
1,4-Dichlorobenzene	106-46-7	330
1-Methylnaphthalene	90-12-0	330
2,4,5-Trichlorophenol	95-95-4	330
2,4,6-Trichlorophenol	88-06-2	330
2,4-Dichlorophenol	120-83-2	330
2,4-Dimethylphenol	105-67-9	330
2.4-Dinitrophenol	51-28-5	330
2,4-Dinitrotoluene	121-14-2	330
2.6-Dinitrotoluene	606-20-2	330
2-Chloronaphthalene	91-58-7	330
2-Chlorophenol	95-57-8	330
2-Methylnaphthalene	91-57-6	330
2-Methylphenol(o-Cresol)	95-48-7	330
2-Nitroaniline	88-74-4	330
2-Nitrophenol	88-75-5	330
3&4-Methylphenol	108-39-4 106-44-5	660
3,3'-Dichlorobenzidine	91-94-1	660
3-Nitroaniline	99-09-2	330
4,6-Dinitro-2-methylphenol	534-52-1	1700
4-Bromophenylphenyl ether	101-55-3	330
4-Chloro-3-methylphenol	59-50-7	330
4-Chloroaniline	106-47-8	330
4-Chlorophenylphenyl ether	7005-72-3	330
4-Nitroaniline	100-01-6	330
4-Nitrophenol	100-02-7	330
Acenaphthene	83-32-9	330
Acenaphthylene	208-96-8	330
Anthracene	120-12-7	330
Benzo(a)anthracene	56-55-3	330
Benzo(a)pyrene	50-32-8	330
Benzo(b)fluoranthene	205-99-2	330
Benzo(g,h,i)perylene	191-24-2	330

TABLE IIa: Analytes and Quantitation Limits-Standard 8270C/D reporting list (Continued)

Analyte	CAS #	PQL (ug/kg)
Benzo(k)fluoranthene	207-08-9	330
Butylbenzylphthalate	85-68-7	330
Benzoic acid	65-85-0	1700
Benzyl alcohol	100-51-6	330
Benzidine		1600
Carbazole	86-74-8	330
Chrysene	218-01-9	330
Di-n-butylphthalate	84-74-2	330
Di-n-octylphthalate	117-84-0	330
Dibenz(a,h)anthracene	53-70-3	330
Dibenzofuran	132-64-9	330
Diethylphthalate	84-66-2	330
Dimethylphthalate	131-11-3	300
Fluoranthene	206-44-0	330
Fluorene	86-73-7	330
Hexachloro-1,3-butadiene	87-68-3	330
Hexachlorobenzene	118-74-1	330
Hexachloroethane	67-72-1	330
Hexachlorocyclopentadiene	118-74-1	330
Indeno(1,2,3-cd)pyrene	193-39-5	330
Isophorone	78-59-1	330
N-Nitroso-di-n-propylamine	621-64-7	330
N-Nitrosodimethylamine	62-75-9	330
N-Nitrosodiphenylamine	86-30-6	330
Naphthalene	91-20-3	330
Nitrobenzene	98-95-3	330
Pentachlorophenol	87-86-5	670
Phenanthrene	85-01-8	330
Phenol	108-95-2	330
Pyrene	129-00-0	330
Pyridine	110-86-1	330
bis(2-Chloroethoxy)methane	111-91-1	330
bis(2-Chloroethyl) ether	111-44-4	330
bis(2-Chloroisopropyl)ether	108-60-1	330
bis(2-Ethylhexyl)phthalate	117-81-7	330

TABLE IIb: Analytes & Quantitation Limits -Extractable 8270-TCLP Analytes

Compound	CAS Number	TCLP µg/L
Pyridine	110-86-1	100
1,4-Dichlorobenzene	106-46-7	100
2-Methylphenol	95-48-7	100
3&4-Methylphenol	109-39-4 & 106-44-5	100
Hexachloroethane	67-72-1	100
Nitrobenzene	98-95-3	100
2,4-Dinitrotoluene	121-14-1	100
2,4,6-Trichlorophenol	88-06-2	100
2,4,5-Trichlorophenol	95-95-4	100
Hexachlorobenzene	118-74-1	100
Pentachlorophenol	87-86-5	200
Hexachloro-1,3-butadiene	87-68-3	100

TABLE III: Internal Standard Method of Quantitation (Standard 8270 list)**

Pace Analytical	Document Name: 8270 Characteristic Ions and IS Associations	Document Revised: 14Apr2016 Page 1 of 4
	Document No.: F-MN-O-246-rev.03	Issuing Authority: Pace Minnesota Quality Office

Analyte	Primary Ion	Secondary Ion (s)	Internal Standard used for Quantitation
N-nitrosodimethylamine	74	42, 44	1
Pyridine	79	52	1
2-Fluorophenol (S)	*112*	*64*	*1*
Phenol-d6 (S)	*99*	*71, 42*	*1*
Phenol	94	66, 65	1
bis(2-Chloroethyl) ether	63	93, 95,	1
2-Chlorophenol	128	64, 130	1
1,3-Dichlorobenzene	146	148, 111	1
1-4-Dichlorobenzene-d4 (IS #1)	152	150, 115	
1,4-Dichlorobenzene	146	148, 111	1
Benzyl Alcohol	79	108, 77	1
1,2-Dichlorobenzene	146	148, 111	1
2-Methylphenol	107	108, 77	1
bis-(2-Chloroisopropyl) ether	45	77, 121	1
N-Nitroso-di-n-propylamine	70	42, 101, 130	1
3&4-Methylphenol	107	108, 77	1
Hexachloroethane	117	201, 199	1
Nitrobenzene-d5 (S)	*82*	*54*	*2*
Nitrobenzene	77	123, 65	2
Isophorone	82	138, 95	2
2-Nitrophenol	139	65, 109	2
2,4-Dimethylphenol	107	122, 121	2
bis(2-Chloroethoxy)methane	93	95, 123	2

TABLE III: Internal Standard Method of Quantitation (Standard 8270 list)** **(Continued)**

Pace Analytical™	Document Name: 8270 Characteristic Ions and IS Associations	Document Revised: 14Apr2016 Page 2 of 4
	Document No.: F-MN-O-246-rev.03	Issuing Authority: Pace Minnesota Quality Office

Benzoic Acid	105	102, 77	2
2,4-Dichlorophenol	162	164, 98	2
1,2,4-Trichlorobenzene	180	182, 145	2
Naphthalene-d8 (IS #2)	136	68	
Naphthalene	128	129, 127	2
4-Chloroaniline	127	129, 65	2
Hexachlorobutadiene	225	223, 227	2
4-Chloro-3-methylphenol	107	142, 144	2
2-Methylnaphthalene	142	141	2
1-Methylnaphthalene	142	141	2
Hexachlorocyclopentadiene	237	235, 272	3
2,4,6-Trichlorophenol	196	198, 200	3
2,4,5-Trichlorophenol	196	198, 97	3
2-Fluorobiphenyl (S)	172		3
2-Chloronaphthalene	162	127, 164	3
2-Nitroaniline	65	138, 92	3
Dimethylphthalate	163	194, 164	3
2,6-Dinitrotoluene	165	63, 89	3
Acenaphthene-d10 (IS #3)	164	162, 160	
Acenaphthylene	152	151, 153	3
3-Nitroaniline	138	92, 108	3
Acenaphthene	154	152, 153	3
2,4-Dinitrophenol	184	63, 154	3
4-Nitrophenol	65	109, 139	3

TABLE III: Internal Standard Method of Quantitation (Standard 8270 list)** **(Continued)**

Pace Analytical™	Document Name: 8270 Characteristic Ions and IS Associations	Document Revised: 14Apr2016 Page 3 of 4
	Document No.: F-MN-O-246-rev.03	Issuing Authority: Pace Minnesota Quality Office

Dibenzofuran	168	139	3
2,4-Dinitrotoluene	165	89, 63	3
Diethylphthalate	149	177, 150	3
4-Chlorophenyl phenyl ether	204	206, 141	3
Fluorene	166	165, 167	3
4-Nitroaniline	138	65, 108	3
4,6-Dinitro-2-methylphenol	198	51, 105	4
N-nitrosodiphenylamine	169	168, 167	4
1,2 Diphenylhydrazine	77		3
2,4,6-Tribromophenol (S)	*330*	*332, 141*	*3*
4-Bromophenyl phenyl ether	248	250, 141	4
Hexachlorobenzene	284	142, 249	4
Pentachlorophenol	266	264, 268	4
Phenanthrene-d10 (IS #4)	188	94, 80	
Phenanthrene	178	179, 176	4
Anthracene	178	176, 179	4
Carbazole	167	166	4
Di-n-butylphthalate	149	150, 104	4
Fluoranthene	202	101, 203	4
Benzidine	184	92	5
Pyrene	202	200, 203	5
Terphenyl-d14 (S)	*244*	*122, 212*	*5*
Butyl benzyl phthalate	149	91, 206	5
bis-(2-ethylhexyl)phthalate	149	167, 279	5

TABLE III: Internal Standard Method of Quantitation (Standard 8270 list)** **(Continued)**

Pace Analytical	Document Name: 8270 Characteristic Ions and IS Associations	Document Revised: 14Apr2015 Page 4 of 4
	Document No.: F-MN-O-246-rev.03	Issuing Authority: Pace Minnesota Quality Office

3,3'-Dichlorobenzidine	252	254, 126	5
Benzo(a)anthracene	228	229, 226	5
Chrysene-d12 (IS #5)	240	120, 236	
Chrysene	228	226, 229	5
Di-n-octyl phthalate	149	167, 43	5
Benzo(b)fluoranthene	252	253, 125	6
Benzo(k)fluoranthene	252	253, 125	6
Benzo(a)pyrene	252	253, 125	6
Perylene-d12 (IS #6)	264	260, 265	
Indeno(1,2,3-cd)pyrene	276	138, 277	6
Dibenz(a,h)anthracene	278	279, 139	6
Benzo(g,h,i)perylene	276	138, 277	6

TABLE IV: Characteristic Ions For Target Compounds – (Standard 8270 List)**

Compound	Primary Ion	Secondary Ions
N-nitrosodimethylamine	74	42, 44
Pyridine	79	52
Phenol	94	66, 65
bis(2-Chloroethyl) ether	63	93, 95,
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl Alcohol	79	108, 77
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	107	108, 77
bis-(2-Chloroisopropyl) ether	45	77, 121
N-Nitroso-di-n-propylamine	70	42, 101, 130
3&4-Methylphenol	107	108, 77
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	138, 95
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	122, 121
bis(2-Chloroethoxy)methane	93	95, 123
Benzoic Acid	105	102, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129, 65
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	142, 144
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 97
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	138, 92
Dimethylphthalate	163	194, 164
2,6-Dinitrotoluene	165	63, 89
Acenaphthylene	152	151, 153
3-Nitroaniline	138	92, 108
Acenaphthene	154	152, 153

TABLE IV - Characteristic Ions For Target Compounds – (Standard 8270 List)** (Continued)

Compound	Primary Ion	Secondary Ions
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	65	109, 139
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	89, 63
Diethylphthalate	149	177, 150
4-Chlorophenyl phenyl ether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	65, 108
4,6-Dinitro-2-methylphenol	198	51, 105
N-nitrosodiphenylamine	169	168, 167
1,2 Diphenylhydrazine	77	
4-Bromophenyl phenyl ether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Carbazole	167	166
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	92
Pyrene	202	200, 203
Butyl benzyl phthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis-(2-ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octyl phthalate	149	167, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 277
Dibenz(a,h)anthracene	278	279, 139
Benzo(g,h,i)perylene	276	138, 277

** Please reference SW-846 method 8270C for quantitation ions for the additional analytes.

TABLE V: Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE VI: System Performance Check Compounds (SPCC)

Base/Neutral Fraction	Acid Fraction
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

TABLE VII: Characteristic Ions for Internal Standards

I.S. Compound	Primary Ion	Secondary Ion(s)
1,4-Dichlorobenzene-d_4	152	150,115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE VIII: Characteristic Ions for Surrogates

Surrogate Compound	Primary Ion	Secondary Ion(s)
Phenol-d_6	99	71, 42
Fluorophenol	112	64
Nitrobenzene-d_5	82	128, 54
Fluorobiphenyl	172	171
Tribromophenol	330	332, 141
Terphenyl-d_{14}	244	122, 212

TABLE IX: Surrogate Compounds*

Compound	Fraction	Concentration, µg/L
Nitrobenzene-d_5	BN	75
2-Fluorobiphenyl	BN	75
Terphenyl-d_{14}	BN	75
Phenol-d_6	Acid	100
2-Fluorophenol	Acid	100
2,4,6-Tribromophenol	Acid	100

*at the time of injection

ATTACHMENT I: Sonication Extraction Bench Sheet
(example)

Pace Analytical	Document Name: **Extraction Sheet – 8270C/D Water Liquid-Liquid**	Document Revised: 04 Mar 2014 Page 1 of 1
	Document No.: **F-MN-O-009 Rev.07**	Issuing Authority: Pace Minnesota Quality Office

SS:		**Amt:** 100 µL	**Analyst:**	**Ext. Date/By:**	
MS:		**Amt:** 500 µL	**Analyst:**	**Batch:**	
BNA SS 750/1500 µg/mL		**BNA MS** 100 µg/mL		**Syringe ID:**	

	Sample ID	I.V. 1000mL	pH	Spike Ver.	pH <2	pH >11	F.V. 1.0mL Lot #	Comments
1	MB-	1000						
2	LCS-	1000						
3	LCSD-	1000						
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample ID Verified by: _____ $MeCl_2$ (Ext):_____ Date Conc. /By:_____
Liquid/Liquid Started:_____ 1:1 H_2SO_4 Lot:_____ $MeCl_2$ (F.V.):_____
Liquid/Liquid Finished:_____ 10N NaOH Lot:_____ Bath Temp (90°) Read:_____
Basic Extraction Started:_____ Na_2SO_4 Lot:_____ Bath Temp (90°) Corrected: _____
Basic Extraction Finished:_____ Glass Wool Lot: _____ Thermometer ID: _____
Pipettor ID: _____

Comments:_____

8270C/D – Water (Liq-Liq)

Posted by/Date:	**Validated by/Date:**

ATTACHMENT II: Waste Dilution Technique – Method

Analysis Method: SW8270C Waste Dilution
Extraction Method: SW3580A

Holding Time:	Samples should be extracted within 14 days from sample collection. All samples must be logged under the soil acode with a comment that the sample is a waste dilution.
QC Requirements:	A method blank (MB) must be performed each day or every 20 samples, whichever is more frequent. If sufficient sample is not provided to perform an MS/MSD, a LCS/LCSD is performed.
Extraction Solvent:	Methylene Chloride
Extraction:	For samples that are liquid or sludge like form but miscible in methylene chloride transfer a ample amount of sample to a 7 mL vial and add a small amount of sodium sulfate. Cap and shake the vial to remove any water. Weigh out 0.1 g into a 2 mL autosampler vial and add 0.1 mL of surrogate. Add 0.5 mL of MS to the appropriate QC. Finalize each sample at 1.0 mL with methylene chloride. Record all data using a 8270 soil prep sheet. Vortex each sample for approximately 1 minute each.
Final Volume:	1.0 mL
Final Solvent:	Methylene Chloride

ATTACHMENT III: Instrument and Maintenance Run Log (example)

Pace Analytical™

Instrument Run Log

1

Instrument: 10MSS9 Method: 8270 Misc. Prep. Info: Surrogate Lot: see extract sheet
Column: DB-5MS 0.25mm Helium Tune Standard: 9000-173 ISTD Lot: 8934-206 Cal. Standard: 9000-180

Path/File	Lab ID	Matrix/Batch	Type	DF	pH	Method	Date & Time	Oper.	Comments
12041101.D	MEGL2 RINSE	/	Sample	1		SW846TUN	12/04/11 11:01	JRH	
12041102.D	TUNE	/	Tune	1		SW846TUN	12/04/11 11:17	JRH	pass
12041103.D	CCAL 6	/	CCal	1		112111-8270MSS9	12/04/11 11:34	JRH	pass
12041104.D	1107809	L7530	Blank	1		112111-8270MSS9	12/04/11 12:05	JLR	pass
12041105.D	1107810	L7530	LCS	1		112111-8270MSS9	12/04/11 12:33	JLR	pass
12041106.D	10176810001	L7530	Sample	1		112111-8270MSS9	12/04/11 13:00	JLR	
12041107.D	1107811	L7530	MS	1		112111-8270MSS9	12/04/11 13:28	JLR	
12041108.D	1107812	L7530	MSD	1		112111-8270MSS9	12/04/11 13:55	JLR	
12041109.D	1107764	S7531	Blank	1		112111-8270MSS9	12/04/11 14:23	JLR	pass
12041110.D	1107765	S7531	LCS	1		112111-8270MSS9	12/04/11 14:51	JLR	pass
12041111.D	10176814002	S7531	Sample	1		112111-8270MSS9	12/04/11 15:18	JLR	
12041112.D	1107766	S7531	MS	1		112111-8270MSS9	12/04/11 15:46	JLR	
12041113.D	1107767	S7531	MSD	1		112111-8270MSS9	12/04/11 16:13	JLR	
12041114.D	10176814004	S7531	Sample	1		112111-8270MSS9	12/04/11 16:41	JLR	
12041115.D	10176814006	S7531	Sample	1		112111-8270MSS9	12/04/11 17:08	JLR	
12041116.D	10176814008	S7531	Sample	1		112111-8270MSS9	12/04/11 17:36	JLR	
12041117.D	1107278	L7532	Blank	1		112111-8270MSS9	12/04/11 18:03	JLR	pass
12041118.D	1107279	L7532	LCS	1		112111-8270MSS9	12/04/11 18:31	JLR	pass
12041119.D	1107280	L7532	LCSD	1		112111-8270MSS9	12/04/11 18:59	JLR	pass
12041120.D	10176936001	L7532	Sample	1		112111-8270MSS9	12/04/11 19:26	JLR	
12041121.D	10177005002	L7532	Sample	1		112111-8270MSS9	12/04/11 19:53	JLR	
12041122.D	1106649	L7533	Blank	1		112111-8270MSS9	12/04/11 20:21	JLR	pass
12041123.D	1106650	L7533	LCS	1		112111-8270MSS9	12/04/11 20:48	JLR	pass
12041124.D	1106651	L7533	LCSD	1		112111-8270MSS9	12/04/11 21:16	JLR	pass
12041125.D	10176555001	L7533	Sample	1		112111-8270MSS9	12/04/11 21:43	JLR	
12041126.D	10176555002	L7533	Sample	1		112111-8270MSS9	12/04/11 22:11	JLR	
12041127.D	10176757001	L7533	Sample	1		112111-8270MSS9	12/04/11 22:38	JLR	

Check Maintenance Items Performed:

- Changed septum
- x Cleaned liner
- x Replaced/Cleaned gold seal

x Clipped column
Changed trap - Lot #
Cleaned MS Source

Changed column - Lot #
Other minor parts replaced
No maintenance performed today

Additional Comments:

File Path 1: U:\10MSS9\120411.B\
Matrix Codes: [G]as, [L]iquid, [S]olid, [N]one

Run order verified: JLR

Report Date: 12/05/2011 09:17
Reviewed By/Date:

ATTACHMENT IV: Method 8270D Minimum Response Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

ATTACHMENT IV: Method 8270D Minimum Response Criteria (Continued)

Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800

ATTACHMENT IV: Method 8270D Minimum Response Criteria **(Continued)**

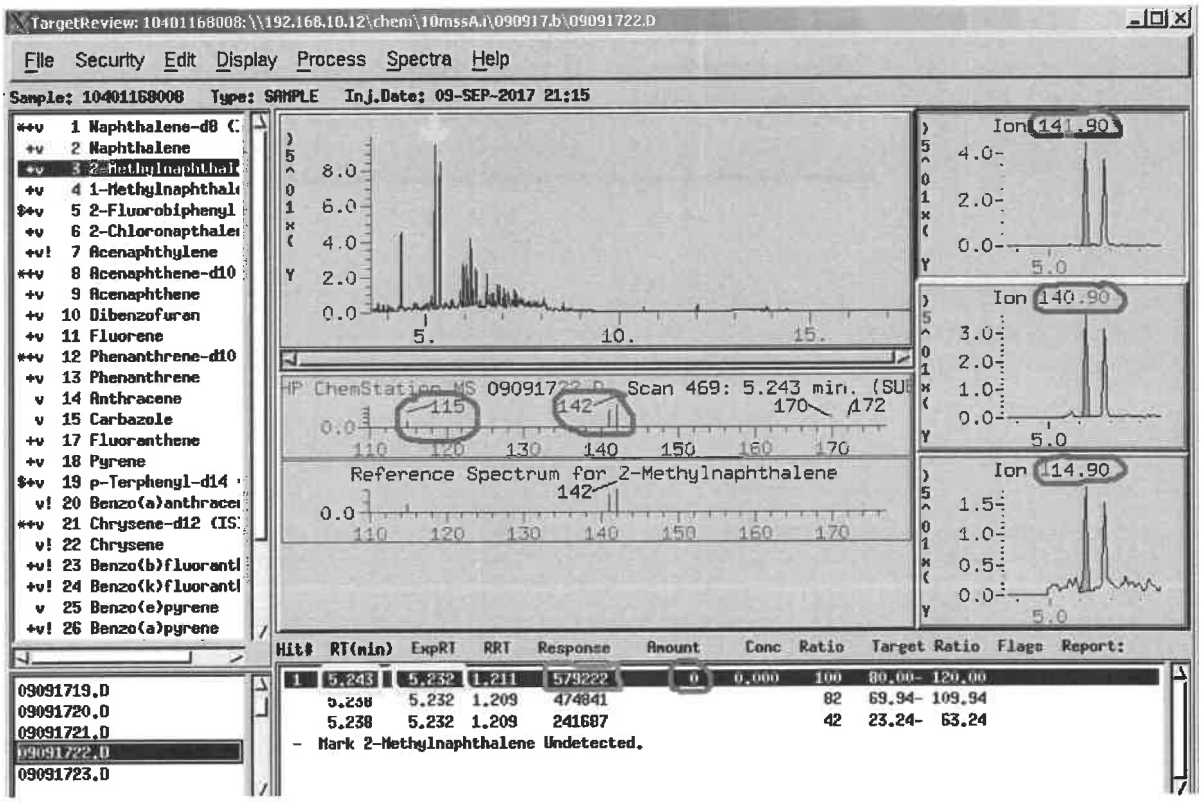
Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Attachment V: Large Hits Quanting to Zero

Pace Analytical®	Document Name: **Large Hits Quanting to Zero**	Document Revised: 18Dec2017 Page 1 of 2
	Document No.: **F-MN-O-338-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

Large Hits Quanting to Zero

How to determine if Amount is really zero or if it is so far above calibration range its quanting to zero



If response is a high number, usually >1000 (579222)

If the peak is in the right RT, <0.06 from ExpRT (5.243RT, 5.232ExpRT)

If all ions are present (141.90, 140.9 and 114.90). They do not need to necessarily match the reference spectrum but they need to present (other matrix in the sample may be skewing the ion ratio height and they may appear to be smaller than reference spectrum).

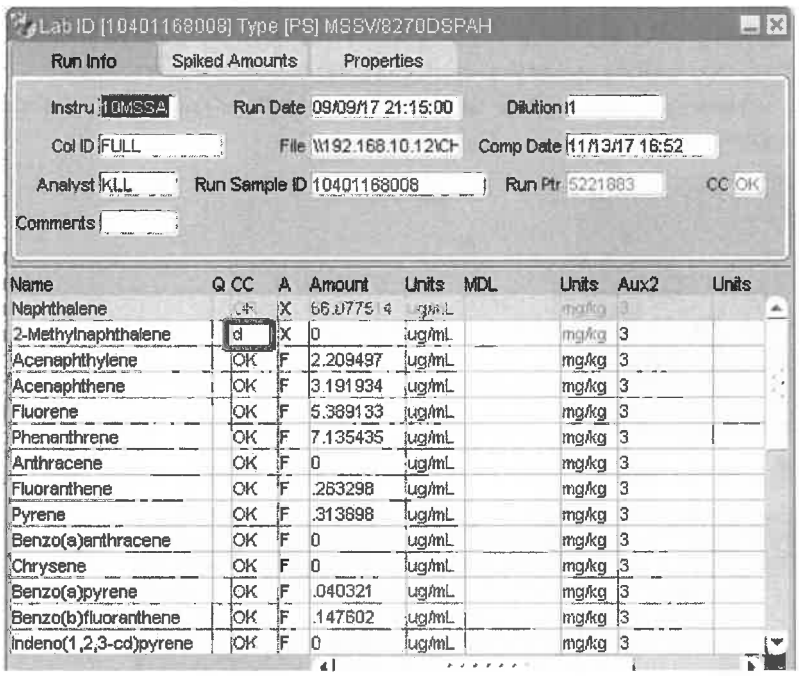
Amount is (0) but all other criteria is met so peak should be left in and a dilution should be ran to capture peak area. Dilution should be started at 50x to determine what the peak area is. A higher or lower dilution may be required depending on calibration curve range.

The Blue arrow is pointing to the total ion on the chrome which shows the peak is very large compared to the rest of the peaks on the chrome which should trigger you to look at the response not the amount.

Attachment V: Large Hits Quanting to Zero (Continued)

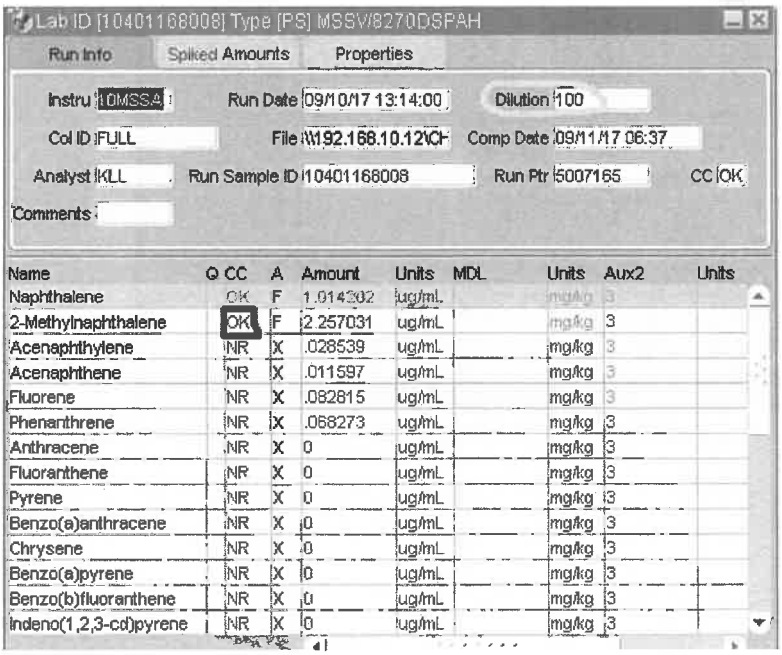
Pace Analytical	Document Name: **Large Hits Quanting to Zero**	Document Revised: 18Dec2017 Page 2 of 2
	Document No.: **F-MN-O-338-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

When uploading the data, horizon will not recognize that a dilution is needed. In the CC (condition code) column you will have to change "OK" to d (don't pick this result to report).



Name	Q	CC	A	Amount	Units	MDL	Units	Aux2	Units
Naphthalene		OK	X	66.0775	ug/mL		mg/kg	3	
2-Methylnaphthalene		d	X	0	ug/mL		mg/kg	3	
Acenaphthylene		OK	F	2.209497	ug/mL		mg/kg	3	
Acenaphthene		OK	F	3.191934	ug/mL		mg/kg	3	
Fluorene		OK	F	5.389133	ug/mL		mg/kg	3	
Phenanthrene		OK	F	7.135435	ug/mL		mg/kg	3	
Anthracene		OK	F	0	ug/mL		mg/kg	3	
Fluoranthene		OK	F	.263298	ug/mL		mg/kg	3	
Pyrene		OK	F	.313898	ug/mL		mg/kg	3	
Benzo(a)anthracene		OK	F	0	ug/mL		mg/kg	3	
Chrysene		OK	F	0	ug/mL		mg/kg	3	
Benzo(a)pyrene		OK	F	.040321	ug/mL		mg/kg	3	
Benzo(b)fluoranthene		OK	F	.147602	ug/mL		mg/kg	3	
Indeno(1,2,3-cd)pyrene		OK	F	0	ug/mL		mg/kg	3	

When uploading the dilution in horizon you will have to change the NR to OK



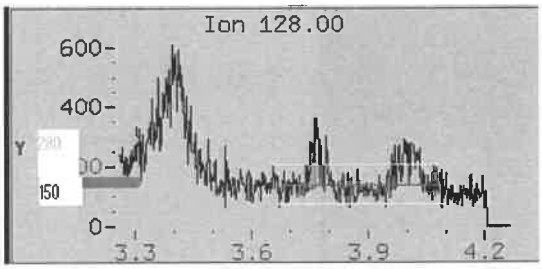
Name	Q	CC	A	Amount	Units	MDL	Units	Aux2	Units
Naphthalene		OK	F	1.014302	ug/mL		mg/kg	3	
2-Methylnaphthalene		OK	F	2.257031	ug/mL		mg/kg	3	
Acenaphthylene		NR	X	.028539	ug/mL		mg/kg	3	
Acenaphthene		NR	X	.011597	ug/mL		mg/kg	3	
Fluorene		NR	X	.082815	ug/mL		mg/kg	3	
Phenanthrene		NR	X	.068273	ug/mL		mg/kg	3	
Anthracene		NR	X	0	ug/mL		mg/kg	3	
Fluoranthene		NR	X	0	ug/mL		mg/kg	3	
Pyrene		NR	X	0	ug/mL		mg/kg	3	
Benzo(a)anthracene		NR	X	0	ug/mL		mg/kg	3	
Chrysene		NR	X	0	ug/mL		mg/kg	3	
Benzo(a)pyrene		NR	X	0	ug/mL		mg/kg	3	
Benzo(b)fluoranthene		NR	X	0	ug/mL		mg/kg	3	
Indeno(1,2,3-cd)pyrene		NR	X	0	ug/mL		mg/kg	3	

Attachment VI: J Flag Hits vs Noise

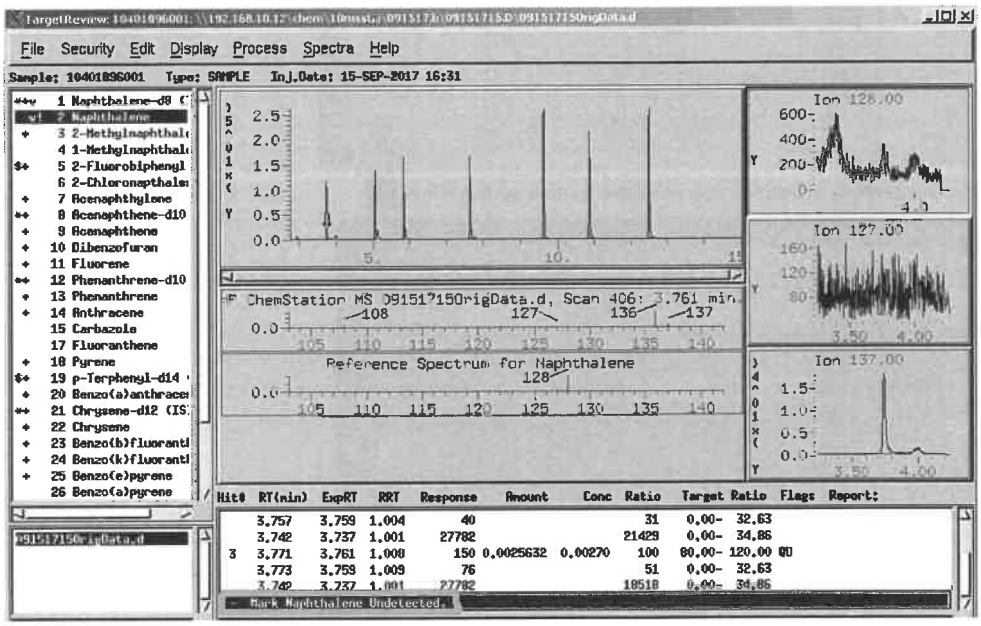
Pace Analytical®	Document Name: **J Flag Hits vs Noise**	Document Revised: 19Dec2017 Page 1 of 2
	Document No.: **F-MN-O-339-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

J Flag Hits vs Noise

Identify signal to noise. To do this you'll have to visualize where the minimum and maximum noise lines would be and then take the average of that noise. That would be the base of your signal. The apex of the peak should be 3 times greater than the base of the signal. In this example the base of the signal is 150, 3 times greater would be 450. The apex of the peak is around 280 which does not meet the criteria.



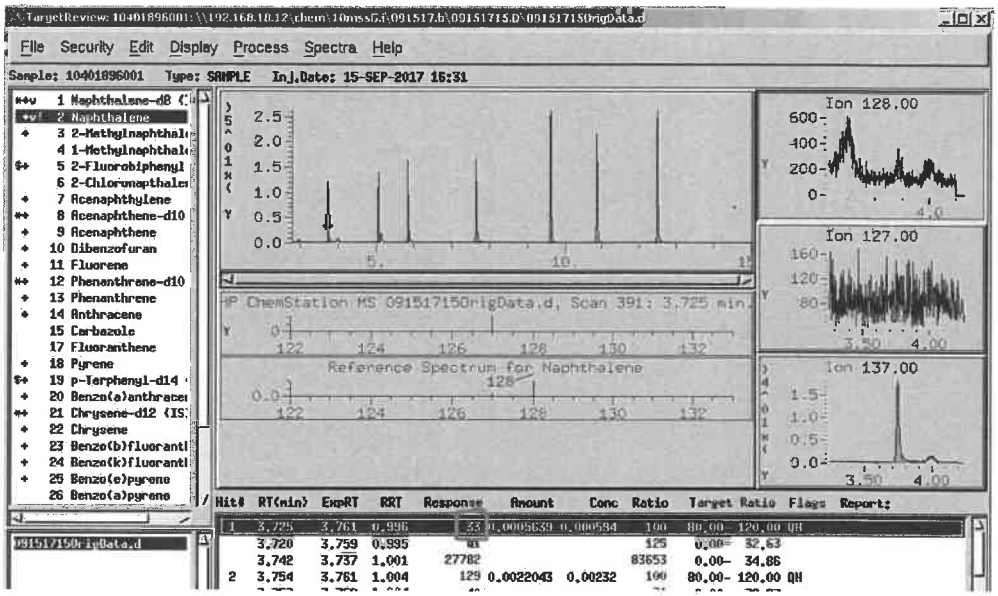
This detection should be undetected or ND(see below).



Attachment VI: J Flag Hits vs Noise (Continued)

Pace Analytical®	Document Name: **J Flag Hits vs Noise**	Document Revised: 19Dec2017 Page 2 of 2
	Document No.: **F-MN-O-339-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

Response should be ≥100 (33)-this peak should be un-detected



Document Information

Document Number: ENV-SOP-MIN4-0063 **Revision:** 00

Document Title: 8270-C/D Extractable Base/Neutral and Acid Organic Compounds in Water and Liquid Matrices by GC/MS Capillary Column Technique w/Selective Ion Monitoring

Department(s): SVOA

Previous Document Number: S-MN-O-507-rev.30

Date Information

Effective Date: 10 Jul 2018

Next Review Date: 10 Jul 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

Extractable Base/Neutral and Acid Organic Compounds (PAHs, cPAHs Pentachlorophenol) in Solid and Liquid Matrices by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique with Selected Ion Monitoring

Reference Methods: EPA 8270C/D, 625 SIM

Local SOP Number:	S-MN-O-507-Rev.30
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-507-Rev.29

APPROVALS

Laboratory General Manager — Date: 10 Jul 2018

Laboratory Quality Manager — Date: 02JUL2018

PERIODIC REVIEW

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Signature	Title	Date

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S-MN-O-507-Rev. 30

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1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to set forth the procedure used for the determination of pentachlorophenol and polyaromatic hydrocarbons in solids and liquids that are partitioned into an organic solvent and are amenable to gas chromatography/mass spectrometry by Method modified 8270C SIM/8270D SIM.

2. Summary of Method

2.1. A measured amount of sample, approximately 1000 mL for liquids and 30 g for solids, is extracted with methylene chloride using an appropriate extraction technique. The methylene chloride extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Qualitative identification of the analyte of interest in the extract is performed using the retention time and relative abundance of at least one characteristic mass. Quantitation is performed using the internal standard technique with a single characteristic mass in coordination with the calibration curve.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid and liquid matrices. Analytes and reporting limits are shown in Table I.

4. Applicable Matrices

4.1. This SOP is applicable to all types of solid and liquid matrices.

5. Limits of Detection and Quantitation

5.1. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Matrix interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

6.3. An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound containing the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Liquid	1L Amber glass bottles with Teflon-lined lid	Keep refrigerated until analysis	<6°C but above freezing	Must be extracted within 7 days from date of collection. Must be analyzed within 40 days of sample extraction.
Solid	4 oz jar with Teflon-lined lid	Keep refrigerated until analysis	<6°C but above freezing	Must be extracted within 14 days from date of collection. Must be analyzed within 40 days of sample extraction.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Agilent gas chromatograph/mass spectrometer	model 5973/ 6890	Agilent
Hewlett-Packard Autosampler	model 7683A (or equivalent)	Agilent
GC Column	DB-5MS, 30 m x 0.25 mm ID bonded-phase silicone coated fused silica capillary column, 0.5 um film thickness (or equivalent).	Phenomenex /7HG-G027-17
Data Processing Software	See master list for most current version	Target
Data Reporting Software	See master list for most current version	Horizon
Data Packaging Software	See master list for most current version	Gandalph
Acquisition Software	See master list for most current version	Agilent Chemstation

9.2. Recommended GC/MS operating conditions:

Dwell Time per ion:	25 to 100 µS
Temperature Program:	75°C, hold for 1 minute 20°C/min to 320 hold for 2.25 minutes
Injection Temperature:	275°C
Transfer Line Temperature:	300°C
Sample Volume:	1 µL
Carrier Gas:	Helium

9.3 Recommended GC/MS operating conditions for MDH CPAH Ext list
Column- 20m x 180um x 0.14um (PAH column)

Mode:	Splitless
Injection Temperature:	300°C
Pressure:	22.291 psi
Total Flow:	104 mL/min
Septum Purge Flow:	3mL/min

Purge Flow to Split vent:	100mL/min at 0.5 min
Transfer Line Temperature:	300˚C
Oven:	70 ˚C
Initial Pressure:	22.291 psi
Flow:	1 mL/min
Average Velocity:	45.066 cm/sec
Holdup Time:	0.73965 min
Oven Ramp	70 ˚C Hold time 1 min 20 ˚C/min, 240 ˚C hold time 2 minutes 20 ˚C/min, 320 ˚C hold time 5 minutes
Run time is around 55 minutes	
Source Temp:	280 ˚C
MS Quad	150 ˚C

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
Organic-free Water (OFW)	De-ionized water	Verify that background levels of volatile compounds are acceptable by analysis
B/N Surrogate	5000 µg/mL	Restek /31086
2,4,6-Tribromophenol	2000 µg/mL	Supelco /4-8084
Custom Full SIM PAH Mix	250 µg/mL	Restek /555193
Custom Semi-volatile Mix, 22-29	100 µg/mL	02SI /113895-03
1,6-dinitropyrene	100 µg/mL	Accustandard /R-032S
1,8-dinitropyrene	100 µg/mL	Accustandard /R-099S
1,6-dinitropyrene	100 µg/mL	Phenova/AL0-130217
1,8-dinitropyrene	100 µg/mL	Phenova/AL0-130216
Pentachlorophenol	5000 µg/mL	Accustandard /AS-E0062
DFTPP	1000 µg/mL	Accustandard/M-625-TS-20X
Internal Standard Mix	4000 µg/mL	Accustandard /Z-014J-PAK
3-Methylchloroanthrene	100 µg/mL	Accustandard/APP-9-128
Custom PAH Mix, 18-52	100 µg/mL	O2Si/ 113892-03-10PAK
Custom PAH Mix	100 ug/mL	Phenova/AL0-130215
4-Nitropyrene	100 µg/mL	Accustandard/R-119S
Custom PAH Standard	100 µg/mL	Restek/566477
CPAH Additions Mix (CPAHADD)	100 ug/mL	Phenova/AL0-130316

10.2. All surrogate and matrix spike solutions are verified prior to use per the S-MN–Q–275, Standard and Reagent Traceability (or equivalent replacement).

10.3. All calibrations are verified using a second source standard. The second source is evaluated as a CCV; except in the case of CPAH the second source is evaluated as a LCS (due to the different concentration of analytes) and the response must be within the acceptance criteria as outlined in S-MN-Q-275 (or equivalent replacement). Some standards are only available through a single manufacturer (example 4-Nitropyrene).

10.4. Table 10.4 - Working Standard Dilutions and Concentrations

Working Standard for PAH SIMs

Standard	Standard(s) used	Vendor	Standard Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
SIM INT SS	B/N surrogate	**Restek**	**3.0 mL**	Acetone	7 mL	10 mL	1500 µg/mL
PAH-SS	SIM INT SS	**Working std.**	**1.0 mL**	Acetone	499 mL	500 mL	3 µg/mL
SIM-SPK	Custom Full SIM PAH Mix conc 250 ug/mL Perylene	**Restek** **Supelco**	**0.8 mL** **0.10 mL**	Acetone	199.1 mL	200 mL	1.0 ug/mL 1.0 ug/mL
CPAH-SPK	Custom Semi-Vol Mix 22-29	**O2Si**	**1.5 mL**	Acetone	42.5 mL	50 mL	3 µg/mL

	Custom PAH Mix 1,6-dinitropyrene 1,8-dinitropyrene CPAH Additions Mix	**Phenova** **Phenova** **Phenova** **Phenova**	**1.5 mL** **1.5 mL** **1.5 mL** **1.5 mL**				3 µg/mL 3 µg/mL 3 µg/mL 3 µg/mL
PCP-SS	2,4,6-tribromophenol	**Supelco**	**0.05 mL**	Acetone	199.95 mL	200 mL	1 µg/mL
PCP-SPK	Pentachlorophenol	**Accustandard**	**0.02 mL**	Acetone	99.98 mL	100 mL	1.0 ug/mL
PAH-IS	Internal Standard	**Accustandard**	**0.625 mL**	$MeCl_2$	9.375 mL	10 mL	250 ug/mL
8270-Tune	DFTPP-Decafluorotriphenylphosphine	**Accustandard**	**0.5mL**	$MeCl_2$	9.5	10 mL	50ug/mL

SIM ICAL Stock (SIM STOCK)

Compound	Vendor	Concentration	Amount	Final Volume *	Final Concentration
Custom Semi-volatile Mix, 22-29	O_2Si	100 µg/mL	**0.500 mL**	5 mL	10 µg/mL
Perlyene	Supelco	2000 ug/mL	**0.025 mL**	5 mL	10 ug/mL
SIM INT SS	Working Std.	1500 µg/mL	**0.040 mL**	5 mL	12 µg/mL

*Made in Methylene Chloride

SIM ICAL

Level	Parent Conc.	Amount	Final Vol *	Final Conc PAH	Final Conc. Surrogate
1	10/12 µg/mL	**0.002 mL**	1 mL	0.02 µg/mL	0.024µg/mL
2 water RL	10/12 µg/mL	**0.004 mL**	1 mL	0.04 µg/mL	0.048 µg/mL
3	10/12 µg/mL	**0.010 mL**	1 mL	0.1 µg/mL	0.12 µg/mL
4 soil RL	10/12 µg/mL	**0.030 mL**	1 mL	0.3 µg/mL	0.36 µg/mL
5	10/12 µg/mL	**0.100 mL**	1 mL	1.0 µg/mL	1.2 µg/mL
6 CCV	10/12 µg/mL	**0.300 mL**	1 mL	3.0 µg/mL	3.6 µg/mL
7	10/12 µg/mL	**0.500 mL**	1 mL	5.0 µg/mL	6 µg/mL
8	10/12 µg/mL	**1.0 mL**	1 mL	10.0 µg/mL	12µg/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS to every level

SIM Intermediate ICV

Compound	Concentration	Amount	Final Volume	Final Concentration
Custom Full PAH Mix (Restek)	250 ug/mL	**0.200 mL**	1.0 mL	50 ug/mL
SIM INT SS	1500 ug/mL	**0.04 mL**	1.0 mL	60 ug/mL

*Made in Methylene Chloride

SIM ICV

Compound	Concentration	Amount	Final Volume	Final Concentration
SIM Intermediate ICV	50/60 ug/mL	**0.060 mL**	1.0 mL	3/3.6 ug/mL
Perylene (Accust)	50 ug/mL	**0.060 mL**	1.0 mL	3 ug/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS to SIM ICV

CPAH ICAL Stock (CPAH-INT)

Compound	Vendor	Concentration	Amount	Final Volume*	Final Concentration
Custom Semi-volatile Mix, 22-29	O_2Si	100 µg/mL	0.500 mL	5 mL	10 µg/mL
Custom PAH Mix	Phenova	100µg/mL	0.500 mL	5 mL	10 µg/mL
CPAH Additions Mix	Phenova	100µg/mL	0.500 mL	5 mL	10 µg/mL
SIM INT SS	Working std	1500 µg/mL	0.04 mL	5 mL	12 µg/mL
1,6-dinitropyrene	Phenova	100 µg/mL	1.5 mL	5 mL	30 µg/mL
1,8-dinitropyrene	Phenova	100 µg/mL	1.5 mL	5 mL	30 µg/mL

*Made in Methylene Chloride

CPAH ICAL (use CPAH-INT as parent)

Level	Parent Concentration	Amount	Final Volume*	Final Conc PAH	Final Conc. Surrogate
1	10/12/30 µg/mL	**0.002 mL**	1 mL	0.02 µg/mL /0.06 µg/mL	0.024µg/mL
2 water RL	10/12/30 µg/mL	**0.004 mL**	1 mL	0.04µg/m/0.12 µg/mL	0.048 µg/mL
3	10/12/30 µg/mL	**0.010 mL**	1 mL	0.1 µg/mL/0.3 µg/mL	0.12 µg/mL
4 soil RL	10/12/30 µg/mL	**0.030 mL**	1 mL	0.3 µg/mL/0.9 µg/mL	0.36 µg/mL
5	10/12/30 µg/mL	**0.100 mL**	1 mL	1.0 µg/mL/3 µg/mL	1.2 µg/mL
6 CCV	10/12/30 µg/mL	**0.300 mL**	1 mL	3.0 µg/mL/9 µg/mL	3.6 µg/mL
7	10/12/30 µg/mL	**0.500 mL**	1 mL	5.0 µg/mL/15µg/mL	6 µg/mL
8	10/12/30 µg/mL	**1.000 mL**	1 mL	10.0 µg/mL/30 µg/mL	12 µg/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS to every level

CPAH ICV

Compound	Vendor	Concentration	Amount	Final Volume*	Final Concentration
Custom Full SIM PAH Mix	Restek	250 ug/mL	**12 uL**	1.0 mL	3 ug/mL
Custom PAH MIX	Restek	100 ug/mL	**30 uL**	1.0 mL	3 ug/mL
3-methylchloranthene	Accustandard	100 ug/mL	**30 uL**	1.0 mL	3 ug/mL
4-nitropyrene	Accustandard	100ug/mL	**30 uL**	1.0 mL	3 ug/mL
1,6-dinitropyrene	Accustandard	100 ug/mL	**90 uL**	1.0 mL	9 ug/mL
1,8-dinitropyrene	Accustandard	100 ug/mL	**90 uL**	1.0 mL	9 ug/mL
SIM INT SS	Working Std.	1500 ug/mL	**2.4 uL**	1.0 mL	3.6 ug/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS added to CPAH ICV

PCP ICAL Stock Intermediate (PCP INT2)

Compound	Vendor	Concentration	Amount	Final Volume *	Final Concentration
Pentachlorophenol	Accustandard	5000 µg/mL	**0.100 mL**	1 mL	500 µg/mL
2,4,6- Tribomophenol	Supelco	2000 µg/mL	**0.250 mL**	1 mL	500 µg/mL

*Made in Methylene Chloride

PCP ICAL Stock (PCP INT)

Compound	Concentration	Amount	Final Volume*	Final Concentration
PCP ICAL stock intermediate	500 ug/mL	**0.100 mL**	5.0	10 ug/mL

*Made in Methylene Chloride

Initial Calibration Example for PCP

Level	Parent Conc.	Amount	Final Vol *	Final Conc.
1	10 µg/mL	**0.010 mL**	1 mL	0.10 µg/mL
2 RL	10 µg/mL	**0.025 mL**	1 mL	0.25 µg/mL
3	10 µg/mL	**0.050 mL**	1 mL	0.50 µg/mL
4	10 µg/mL	**0.100 mL**	1 mL	1 µg/mL
5 CCV	10 µg/mL	**0.300 mL**	1 mL	3 µg/mL
6	10 µg/mL	**0.500 mL**	1 mL	5 µg/mL
7	10 µg/mL	**0.700 mL**	1 mL	7 µg /mL
8	10 µg/mL	**0.900 mL**	1 mL	9 µg/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS to every level

PCP ICV Intermediate (PCP ICV INT)

Compound	Vendor	Concentration	Amount	Final Volume *	Final Concentration
Pentachlorophenol	Restek	1000 ug/mL	**0.100 mL**	1.0 mL	100 ug/mL
8270-SS	Working Std.	1000 ug/mL	**0.100 mL**	1.0 mL	100 ug/mL

*Made in Methylene Chloride

PCP ICV

Compound	Concentration	Amount	Final Volume *	Final Concentration
PCP ICV Intermediate	100 ug/mL	**0.030 mL**	1.0 mL	3 ug/mL

*Made in Methylene Chloride

**add 10uL of PAH-IS to PCP ICV

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Calibration Curve Fit	Average Linear Regression Non-linear Regression	SIM C RSD ≤ 15% SIM D RSD ≤ 20 % r ≥ 0.99 COD ≥ 0.99; if a quadratic curve is used with a minimum of 6 levels.	If not met, try non-linear regression fit. If still not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ±10% from CCV criteria	If the requirements for initial calibration are not met, review standard preparation. Evaluate the instrument for any errors.
Continuing Calibration Verification (CCV)	A calibration standard containing each compound of interest must be analyzed every 12 hours (at the beginning of each tune/shift prior to sample analysis).	The percent difference for each compound must be ≤20% for all compounds in list 1 and 3 (see Attachment III). All compounds in the list 2 must be ≤ 40%. It is suggested that all compounds must meet a minimum average RF of 0.05 but because SIM is spiked at lower concentration of internal standard than 8270 this cannot always be met. (see Attachment III).	If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.

11.2. DFTPP

11.2.1. Section 7.1 and Table 3 of SW846 method 8270C allows for the use of alternate tuning criteria such as CLP or 525 criteria. The CLP Statement of Work (SOW) states that a tune is not required (applicable) for PAH or PCP work only. When analyzing the tune for PCP the PCP tailing must be ≤ 2.0 for 8270D and ≤5 for 8270C. The tune standard (DFTPP) will be analyzed for diagnostic purposes and the ratios used will be those listed in the 525.2 criteria even though it is not necessary to pass ratios, tailing or breakdown. The 12 hour window to analyze samples starts from the injection time of the tune standard. The diagnostic criteria are listed in Table3. The criteria in 525.2 also correspond to the 8270D ion abundances.

11.2.2. When evaluating peaks, SIM is closer to GC than full scan mass spectrometry. The ions are set in the method and will not show interferences from ions that are not monitored. Because of this, the retention time is stressed more than spectral matches for peak identification. The purpose of a tune in full scan mode is to ensure that the mass spectrometer is in a correct ratio of ions to optimize method performance throughout the entire analytical run. Since SIM is more like a GC

run and the ions are set in separate windows across the analytical run, the tune is considered not applicable.

11.2.3. Diagnostics

11.2.3.1. The tune can still serve as a troubleshooting guide for the analyst to assess instrumentation issues such as column degradation.

11.2.3.1.1. If the instrument has trouble passing calibrations consistently, PCP is present, and DDT is not breaking down excessively, then the result of any calibration issues is more likely due to standard degradation and not the system itself.

11.2.3.1.2. Conversely, if the system has trouble consistently passing calibrations or recoveries are inconsistent from run to run, PCP is not present, and/or DDT is breaking down, the result is more than likely due to a system issue. This could indicate that instrument maintenance should be performed; such as injection port maintenance, clipping/replacing the column, cleaning split lines, cleaning weldments or replacing columns.

11.3. INITIAL CALIBRATION VERFICATION (ICV)

11.3.1. To ensure internal standard recoveries from samples that run after the Ical under the same folder go off the Ical and not the ICV, process the ICV as a sample. To see if the ICV meets passing requirements pull the ICV file into a batch.b along with the method and requant the ICV as a continuing calibration.

11.4 CONTINUING CALIBRATION VERIFICATION (CCV)

11.4.1 The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition.

11.4.1.1 If the retention time for any internal standard changes by more than 30 seconds from the last check calibration, the analytical system must be inspected for malfunctions and corrections must be made.

11.4.1.2 If the EICP area for any of the internal standards changes by a factor of two, (-50% to +100%) from the last daily calibration standard check, the system must be inspected for malfunctions and corrections must be made.

12 Procedure

12.1 SAMPLE PREPARATION

12.1.1 Samples are extracted and prepared in accordance with separate extraction procedures (see SOPs MN-O-506 and MN-O-540 (or equivalent replacements)).

12.2 GC/MS ANALYSIS

12.2.1 Initial calibration standards are prepared in methylene chloride at a minimum of 5 calibration levels.

12.2.2 Samples can be analyzed upon successful completion of the initial calibration activities. When twelve (12) hours have elapsed since the initial QC was completed, it is necessary to conduct a calibration check analysis. Any major system maintenance, such as a source cleaning or installation of a new column, requires recalibration of the instrument. Minor or routine maintenance as defined on each instrument specific run log page should necessitate only the calibration verification.

12.2.3 The extract obtained from sample preparation should be spiked at 2.5 µg/mL with internal standard solution.

12.2.4 Analyze utilizing a GC/MS system by injecting the extract onto the column.

12.2.5 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 2.5 µg/mL of each internal standard in the extract volume. The diluted extract must be reanalyzed.

12.2.6 Samples following over-range samples are to be monitored for carryover.

12.2.7 Perform all qualitative and quantitative measurements as described in "Data Interpretation".

12.2.8 Internal Standards Evaluation - Internal standard responses and retention times in all samples must be evaluated.

12.2.8.1 If the retention time for any internal standard changes by more than 30 seconds, then the analytical system must be inspected for malfunctions and corrections made as required.

12.2.8.2 If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to 100%) from the latest daily calibration standard, the MS system must be inspected for malfunction and corrections made as appropriate.

12.2.8.3 Cutting off 1 foot of the column or cleaning the injector sleeve often improves high end sensitivity for the late eluting compounds; repositioning or repacking the front end of the column often improves front end column performance. Poor injection technique can also lead to variable IS ratios. After modification, reanalysis of samples analyzed while the system was malfunctioning is necessary.

12.2.9 Each analytical run must also be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. The initial method calibration requires that the system should not be saturated for high response compounds. If any compound in any sample exceeds the analytical range, that sample must be diluted, the internal standard concentration readjusted, and the sample re-injected, as described in specific methods.

12.2.10 Samples following high standards or over-range samples are to be monitored for carryover.

12.3 DATA INTERPRETATION

12.3.1 Qualitative Analysis – Target Analytes

12.3.2 The target compounds shall be identified by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: 1) elution of the sample component at the same GC relative retention time as the standard component, and 2) correspondence of the sample component and standard component mass spectra.

12.3.3 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For comparison purposes, the RRT should be evaluated against the standard analyzed prior to the sample. See Table 1 for internal standard assignment.

12.3.4 For comparison of standard and sample component mass spectra, mass spectra obtained on the GC/MS system used in the analysis are required. The ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met. The ions used are shown in Table 1.

12.3.5 The baseline to valley between isomers must be <25% of the sum of the two peak heights or the result must be reported as isometric pairs.

12.3.6 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.3.7 The relative intensities of ions must agree within plus or minus 20% between the standard and sample spectra. In addition, the relative intensities of the ions should maximize within one scan of each other.

12.3.8 Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

12.3.9 Refer to Attachment IV for large detections quanting to zero.

12.3.10 Refer to Attachment V for j flag hits vs noise.

12.4 QUANTITATION

12.4.1 Target components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte.

12.4.1.1 The EICP area of characteristic ions of analytes listed in Table 1. The average response factor (RF) from the initial calibration is used to calculate the concentration in the sample.

12.4.1.2 Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a response factor is calculated using the secondary ion.

12.4.2 Calculate the concentration in the sample using the average response factor from the initial calibration and Equation 5.

12.4.3 Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if recovery is within limits and report on appropriate form. (See Equation 8) If recovery is not within internally generated limits, the following is required:

12.4.3.1 Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

12.4.3.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

12.4.3.3 Re-extract and reanalyze the sample if none of the above is a problem.

13 Quality Control

13.1 Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or sand	One per 20 samples	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB that have detection between the MDL and RL will be flagged. Samples can still be reported.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	Reagent water or sand spiked with all target compounds	One per 20 samples; LCS duplicate is performed if there is insufficient sample volume for matrix spike samples	Internally generated limits generated on an annual basis. Per NELAC standards found in the quality manual one analyte can be out for PAH and two analytes can be out for CPAH, unless specified by client.	The LCS/LCSD must pass acceptance criteria for the batch to be accepted. Re-analyze the LCS to confirm results. Check calculation or standard preparation documentation to assure there are no errors; check internal standard and spiking solutions for degradation, contamination, etc; check instrument performance. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the

				associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 20 samples	Individual component recoveries of the matrix spike are calculated using Equation 6. The reproducibility of RPD of the MS is calculated using Equation 7.	MS usually fail due to matrix interference; all failures are flagged in horizon. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately. -If there is insufficient volume for MS/MSD, an LCSD must be performed.
MSD / Duplicate	MS Duplicate *OR* *(alternative)* Sample Dup	One per 20 samples	Individual component recoveries of the matrix spike are calculated using Equation 6. The reproducibility of RPD of the MSD is calculated using Equation 7.	MSD usually fail due to matrix interference, all failures are flagged in horizon.

13.2 Surrogate

13.2.1 Each sample (including MS/MSD, LCS/LCSD, and blanks) are spiked with surrogate compounds prior to extraction. The surrogate spiking compounds shown in Table 2 (Table 4 for PCP only samples) are used to fortify each sample or blank with the proper concentrations. Performance based criteria are generated from laboratory results.

13.2.2 Surrogate spike recovery must be evaluated for acceptance by determining whether the concentration (measured as percent recovery) falls inside the recovery limits established by the laboratory.

13.2.2.1 Reagent Blank Surrogate Recovery

13.2.2.1.1 When the surrogate recovery for any one surrogate compound is outside of the surrogate recovery limits for a reagent blank, the laboratory must take the following actions:

13.2.2.1.1.1 Check calculations to assure there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; also, check instrument performance.

13.2.2.1.1.2 Re-analyze the extract if the above fails to reveal the cause of the non-compliant surrogate recoveries.

13.2.2.1.1.3 If the measures listed in the preceding paragraphs fail to correct the problem, the analytical system must be considered out of control. The problem MUST be corrected before continuing, unless approval to continue is obtained from the client.

13.2.2.1.1.4 This may mean recalibrating the instrument but it may also mean more extensive action. The specific corrective action is left up to the GC/MS supervisor.

13.2.2.2 Sample Surrogate Recovery

13.2.2.2.1 When the surrogate recovery of any one surrogate compound is outside of the recovery limits for a sample, it is the responsibility of the laboratory to establish that the deviation is not due to laboratory problems.

13.2.2.2.2 The laboratory will document deviations outside acceptable quality control limits by taking the following actions:

13.2.2.2.2.1 Check calculations to assure there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; and, check instrument performance.

13.2.2.2.2.2 Re-analyze the sample or extract if the step immediately above fails to reveal a problem. If reanalysis of the sample or extract solves the problem, then only the sample data from the analysis with surrogate spike recoveries within the method limits will be submitted.

13.2.2.2.2.3 Re-extract and re-analyze the sample if the laboratory is unable to identify a definitive problem with the original extraction.

13.2.2.2.2.4 Report the surrogate spike recovery data and the sample data from the original extraction.

13.2.2.2.2.5 All deviations that cannot be corrected by the points listed above will be narrated in a discrepancy report and the client notified.

14 Data Analysis and Calculations

14.1 Response factors (RFs) for each compound can be calculated using Equation 1:

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg /mL).
C_x = Concentration of the compound being measured (µg /mL).

14.2 The percent relative standard deviation (%RSD) is calculated using Equation 2:

Equation 2

$$\%RSD = \frac{SD}{\overline{RF}} x100$$

Where: $\overline{RF}$ = Mean of the Response Factors mentioned above.
SD = Standard Deviation of initial response (see Equation 3)

Equation 3

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_1 - \overline{RF}\right)^2}{n-1}}$$

Where: RF_1 = Each individual response factor
$\overline{RF}$ = Mean of the Response Factors mentioned above.
N = Number of response factors

14.3 The Percent Difference (%D) is calculated as follows:

Equation 4

$$\%Difference = \frac{(RF_i - RF_c)}{RF_i} x100$$

Where: *Rfi* = Average response factor from initial calibration
RF_c = Response factor from current verification check standard

14.4 The concentration of analyte in the sample is calculated as follows:

Equation 5

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF}_i)(W_o)(V_i)}$$

$$\text{Concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF}_i)(W_s)(V_i)\left(\frac{100-M}{100}\right)}$$

Where: A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
W_o = Volume of sample extracted in liters
V_i = Volume of extract injected (µL)
V_t = Volume of total extract (mL)
$\overline{RF}$ = Average response factor from initial calibration
W_s = Weight for soil in kg
M = % Moisture

14.5 The Matrix Spike Percent Recovery as follows:

Equation 6

$$\%Recovery = \frac{SSR - SR}{SA} x100$$

Where: SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

14.6 The laboratory will calculate the relative percent difference between the matrix spike and matrix spike duplicate. The relative percent differences (RPD) for each component are calculated using the following equation:

Equation 7

$$RPD = \frac{|A - B|}{(A + B)/2} x100$$

Where: RPD = Relative Percent Difference
A = First Sample Value
B = Second Sample Value (duplicate)

14.7 Surrogate Standard Recovery:

Equation 8

$$\%\,\mathrm{Recovery} = \frac{Concentrationon\_(amount)\_Found}{Concentration\_(amount)\_added} x100$$

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See table in section 11 and 13.

16 Corrective Actions for Out-of-Control Data

16.1 See table in section 11 and 13.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in the table in section 11 or 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18 Method Performance

18.1 All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4 **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19 Method Modifications

19.1 Not applicable to this SOP.

20 Instrument/Equipment Maintenance

20.1 All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21 Troubleshooting

21.1 The tune should help serve as a troubleshooting guide for column degradation and standard issues. For example: if the system has trouble consistently passing calibrations or recoveries are inconsistent from run to run and PCP is not present the result is more than likely due to a system issue. This would cue the analyst to perform different maintenance including injection port maintenance, clipping column, cleaning split lines, cleaning weldments, or replacing columns.

22 Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24 Pollution Prevention

24.1 The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25 References

25.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update I, Method 8270C

25.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update IV, Method 8270D

25.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update I, Method 8000B

25.4 Contract Laboratory Program Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM01.2)

25.5 Pace Quality Assurance Manual- most current version.

25.6 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.7 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

26 Tables, Diagrams, Flowcharts, and Validation Data

26.1 Table 1 - Analytes and Reporting Limits

26.2 Table 2 - Surrogates Spiking Compounds

26.3 Table 3 – Ion Abundance Criteria for DFTPP

26.4 Table 4 – PCP SIM Surrogate Compound

26.5 Table 5 – PAH SIM Ion and Internal Standard Reference

26.6 Attachment I – SVOA Analyst Checklist (example)

26.7 Attachment II –ICAL Verification Form (example)

26.8 Attachment III – PAH Analyte Lists

26.9 Attachment IV – Large Hits Quanting to Zero

26.10 Attachment V – J Flag Hits vs Noise

27 Revisions

Document Number	Reason for Change	Date
S-MN-O-507-Rev.29	Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. 10.2 – replaced SOP reference with local SOP number that replaced corp SOP. Table 10.4 CPAH ICV section – removed 4-nitropyrene and 3-methylchloroanthrene; updated Vendor for 1,8-dinitropyrene, 1,6-dinitropyrene, and custom PAH mix; updated compound and vendor for custom full SIM PAH Mix. Table 11.1 – in the Comments column of the ICV row, removed "if a second source cannot…(cPAHs only)." New revisions of Attachments I and II added. Added section 11.3.	05Mar2018
S-MN-O-507-Rev.30	Table 13.1 MB row: Added "For WI samples…" exception to Corrective Action column, in Acceptance Criteria column replaced "should be non-detect" with "that have detection between the MDL and RL will be flagged. Samples can still be reported." 10.3 – updated SOP reference number to local SOP instead of corp SOT. Added new Section 9.3. Table 10.1 – added reagents: 1,6- and 1,8-dinitropyrene, Custom PAH Mix, and CPAH Additions Mix (CPAHADD) Tables 10.4: - Working Standard for PAH SIMs – added CPAH Additions Mix to Standards used, replaced Vendors, updated solvent volume and final concentration - SIM ICAL Stock – added (SIM STOCK) to title - SIM ICAL – added to Level column: water RL, soil RL, and CCV - CPAH ICAL Stock – added (CPAH-INT) to title, updated Compounds and Vendors, added CPAH Additions Mix row - Deleted CPAH ICAL Stock for MDH CPAH Ext (CPAH INTEX) table - CPAH ICAL – updated title, added water RL, soil RL, and CCV to Level - CPAH ICV – added rows for 3-methylchloranthene and 4-nitropyrene, updated Vendors - PCP ICAL Stock Intermediate – added (PCP INT2) to title - PCP ICAL Stock – added (PCP INT) to title - Initial Calibration Example for PCP – added RL and CCV to Level - PCP ICV Intermediate – added (PCP ICV INT) to title Table 1 – added rows for Benzo(c)fluorene, benzo(j)fluoranthene, and anthanthrene Attachment III under List 2 – added MDH Primary List and MDH Secondary List Updated to new revision of Attachment I.	25Jun2018

TABLE 1: ANALYTES AND REPORTING LIMITS

Carcinogenic PAH's	CAS Numbers	Quant	Ions Qual	Qual	Liquid Reporting Limit µg/L	Solid Reporting Limit µg/kg
1,4-Dichlorobenzene- d4 IS	53-70-3	149.9	151.9	114.9		
Nitrobenzene-d5 Surr	4165-60-01	82.1	128	54		
Napthalene-d8 IS	1520-96-3	135.9	136.9	108		
Naphthalene	91-20-3	128	127	136.9	0.040	10
Quinoline	91-22-5	129	102		0.040	10
2-Methylnaphthalene	91-57-6	141.9	140.9	114.9	0.040	10
1-Methylnaphthalene	90-12-0	141.9	140.9	114.9	0.040	10
2-Fluorobiphenyl Surr	321-60-8	172	171	170		
2-Chloronaphthalene	91-58-7	162	164	127	0.040	10
Acenaphthylene	208-96-8	152	151	153	0.040	10
Acenapthene-d10 IS	15067-26-2	164	162			
Acenaphthene	83-32-9	153	154	152	0.040	10
Dibenzofuran	132-64-9	168	138.9		0.040	10
Fluorene	96-73-7	166	165		0.040	10
Pentachlorophenol	87-86-5	266	264	268	0.3	10
Phenanthrene-d10 IS	1517-22-2	188	93.9			
Phenanthrene	85-01-8	178.1	179		0.040	10
Anthracene	120-12-7	178.1	179.1		0.040	10
Carbazole	86-74-8	167.1	165.9	138.9	0.040	10
5-Nitroacenaphthene	602-87-9	152	199	153	0.040	10
Fluoranthene	206-44-0	201.9	199.9	101	0.040	10
2-Nitrofluorene	607-57-8	165	211	194	0.040	10
Pyrene	129-00-0	201.9	199.9	101	0.040	10
Terphenyl-d14 Surr	98904-43-9	244.1	243.1	122		
Chrysene	218-01-9	227.9	225.9	112.9	0.040	10
Chrysene-d12 IS	1719-03-5	240.1				
Benz(a)anthracene	56-55-3	227.9	225.9	113.9	0.040	10
4-Nitropyrene	57835-92-4	201	189	247	0.040	10
5-Methylchrysene	3697-24-3	242	241		0.040	10
1-Nitropyrene	5522-43-0	201	189	247	0.040	10
Benzo(b and j)fluoranthene	205-99-2	251.9	249.9	126	0.080	20
Benzo(k)fluoranthene	207-08-9	251.9	249.9	126	0.040	10
7,12-Dimethylbenz(a) anthracene	57-97-6	256	252	241	0.040	10
Total Fluoranthenes					0.120	30
Benzo(e)pyrene	192-97-2	251.9	249.9	125	0.040	10
Perylene-d12 IS	1520-96-3	264.2	265	132		
Benzo(a)pyrene	50-32-8	251.9	252.9	126	0.040	10
6-Nitrochrysene	7496 .02.8	226	273	252	0.040	10
Perylene	198-55-0	252	250		0.040	10
3-Methylcholanthrene	56-49-5	268	252		0.040	10
1,6-Dinitropyrene	42397-64-8	200	292	188	1.0	100
1,8-Dinitropyrene	42397-65-9	200	292	188	1.0	100
Dibenz(a,h)acridine	226-36-8	279	280	139	0.040	10
Dibenz(a,j)acridine	224-42-0	279	280	139	0.040	10
Indeno(1,2,3-cd)pyrene	193-39-5	275.9	137.9	136.9	0.040	10
Dibenz(a,h)anthracene	53-70-3	277.9	138.9	275.9	0.040	10
7H-Dibenzo(c,g)carbazole	194-59-2	267	265		0.040	10
Benzo(g,h,i)perylene	191-24-2	275.9	137.9	136.9	0.040	10
Dibenzo(a,e)pyrene	192-65-4	302	150		0.040	10
Dibenzo(a,l)pyrene	191-30-0	302	150		0.040	10
Dibenzo(a,h)pyrene	189-64-0	302	303	151	0.040	10
Dibenzo(a,l)pyrene	189-55-9	302	303	151	0.040	10
2,4,6-Tribromophenol Surr	118-79-6	329.9	331.9	141		
Benzo(c)fluorene	205-12-9	215	216		0.040	NA
Benzo(j)fluoranthene	205-82-3	251.9	249.9	126	0.100	NA
Anthanthrene	120-12-7	276	277		0.040	NA

TABLE 2: SURROGATE SPIKING COMPOUNDS

Compound	Fraction	SIM Water (µg/L)
Nitrobenzene-d_5	BN	3.0
2-Fluorobiphenyl	BN	3.0
Terphenyl-d_{14}	BN	3.0

TABLE 3: ION ABUNDANCE CRITERIA FOR DFTPP

Mass (M/z)	Relative Abundance Criteria	Purpose of Checkpoint
51	10-80% of the base peak	Low-mass sensitivity
68	< 2% of Mass 69	Low-mass resolution
70	< 2% of Mass 69	Low-mass resolution
127	10-80% of the base peak	Low-to mid-mass sensitivity
197	< 2% of Mass 198	Mid-mass resolution
198	Base peak or > 50% of Mass 442	Mid-mass resolution and sensitivity
199	5-9% of Mass 198	Mid-mass resolution and isotope ratio
275	10-60% of the base peak	Mid- to high-mass sensitivity
365	> 1% of the base peak	Baseline threshold
441	Present and < Mass 443	High-mass resolution
442	Base peak or > 50% of Mass 198	High-mass resolution and sensitivity
443	15-24% of Mass 442	High-mass resolution and isotope ratio

TABLE 4: PCP SIM SURROGATE COMPOUND

Compound	Concentration (µg/mL)
2,4,6-Tribromophenol	1.0

TABLE 5: PAH SIM IONS AND INTERNAL STANDARD REFERENCES

PAH SIM Ions and Internal Standard References

Monitoring Ions*	Analyte Name	Internal Std Reference
128, 127, 137	Naphthalene	Naphthalene-d8
142, 141, 115	2-Methylnaphthalene	Naphthalene-d8
142, 141, 115	1-Methylnaphthalene	Naphthalene-d8
172, 171,170	2-Fluorbiphenyl(S)	Acenaphthene-d10
162, 164, 127	2-Chloronaphthalene	Acenaphthene-d10
152, 151, 153	Acenaphthylene	Acenaphthene-d10
153, 154, 152	Acenaphthene	Acenaphthene-d10
167.95, 138.9	Dibenzofuran	Acenaphthene-d10
166, 165	Fluorene	Acenaphthene-d10
178, 179	Phenanthrene	Phenanthrene-d10
178, 179	Anthracene	Phenanthrene-d10
166.95, 165.9, 138.9	Carbazole	Phenanthrene-d10
202, 200, 101	Fluoranthene	Phenanthrene-d10
202, 200, 101	Pyrene	Chrysene-d12
244.1, 243.05, 121.95	Terphenyl - d14 (S)	Chrysene-d12
228, 226, 114	Benzo(a)anthracene	Chrysene-d12
228, 226, 113	Chrysene	Chrysene-d12
252, 250, 126	Benzo(b)fluoranthene	Perylene-d12
252, 250, 126	Benzo(k)fluoranthene	Perylene-d12
252, 250, 125	Benzo(e)pyrene	Perylene-d12
252, 250, 126	Benzo(a)pyrene	Perylene-d12
276, 138, 137	Indeno(1,2,3-cd)pyrene	Perylene-d12
278, 139	Dibenz(a,h)anthracene	Perylene-d12
276, 138, 137	Benzo(g,h,i)perylene	Perylene-d12

ATTACHMENT I – SVOA Analyst Checklist (example)

Pace Analytical™

SIM Semivolatile Analyst Checklist

Prep Batch # _____ Analytical Batch # _____

☐ SIM – Water ☐ SIM – Soil ☐ CPAH – Water ☐ CPAH – Soil ☐ PCP ☐ 14dioxane

YES ☐ NO ☐ **HOLDING TIME**
Extraction holding times met (7 days from collection for waters and 14 days from collection for soils)? And analysis holding time of 40 days from extraction met for all samples? If not, explain and list affected samples:
☐Review _____

YES ☐ NO ☐ **DFTPP**
Was DFTPP demonstrated every 12 hours and criteria met? If not, explain why samples were analyzed and impact on reported results. Also, discuss any corrective action taken.
☐Review _____

YES ☐ NO ☐ **INITIAL CALIBRATION(S)**
Did the initial calibration meet criteria for all compounds (15% RSD Average, 0.995 Linear, or 0.990 Quadratic)? Is the curve fit the same as the original ICAL? If the ICAL is more than 30 days old, the reporting limit must be verified every 30 days. List Ical ID _____
☐Review

YES ☐ NO ☐ **CONTINUING CALIBRATION(S)**
Was the 20%D criteria met for all compounds? If not, list outliers and discuss any impact on the data. Was a Ccal analyzed every 12 hours?
☐Review _____

YES ☐ NO ☐ **BLANK ANALYSIS**
Was a compliant method blank analyzed on each instrument associated with sample analysis? Were all target analytes present at or below the reporting limit? If not, was the "B" qualifier used? Discuss any impact on the data. Evaluate MB to the MDL for WI. Flag for any detections between MDL and RL.
☐Review _____

YES ☐ NO ☐ **LABORATORY CONTROL SAMPLE**
Was an LCS performed with each extraction batch? Percent recovery (compared to internally generated limits) of each compound in the LCS within control limits? If not, list compounds, any corrective actions, discuss any impact on data and why sample analysis proceeded. RPD within limits? If not, list compounds and discuss impact on data.
☐Review _____

YES ☐ NO ☐ **MATRIX SPIKE/MATRIX SPIKE DUPLICATE**
Was a MS/MSD performed with each extraction batch? Percent recovery (compared to internally generated limits) of each compound in the MS/MSD within control limits? If not, list compounds, any corrective actions, discuss any impact on data and why sample analysis proceeded. RPD within limits? If not, list compounds and discuss impact on data.
☐Review _____

YES ☐ NO ☐ **S.S. AND I.S. RECOVERIES**
Percent recovery of each surrogate compound within control limits? If surrogate outside limits, was sample reanalyzed? List outliers, any corrective action and discuss impact on data.

YES ☐ NO ☐
All samples met internal standard criteria? If not, list outliers, discuss impact on data and note if sample was reanalyzed.
☐Review _____

YES ☐ NO ☐ **MANUAL INTEGRATIONS**
Reason for manual integrations: ☐Retention Time Shift ☐ Baseline Correction ☐ Split Peak
☐ Peak Assignment ☐ Other (indicate why)_____
☐Approved

ADDITONAL COMMENTS: _____

"To the best of my knowledge all of the above information is correct and all supporting documentation has been provided."
Analyst: _____ Date: _____
Reviewer: _____ Date: _____

ATTACHMENT II – ICAL Verification Form (example)

Pace Analytical®	Document Name: **SIM Initial Calibration Verification Form**	Document Revised: 26Mar2018 Page 1 of 1
	Document No.: **F-MN-O-123-rev.16**	Issuing Authority: Pace Minnesota Quality Office

SIM Initial Calibration Verification Form

☐ SIM ☐ CPAH ☐ PCP ☐ HVI ☐ 14Dioxane

ICAL Identification _____

ICAL Exp Date_____ICV_____

Instrument _____

Date of Analysis _____

Analyst _____

Date of Review_____

Reviewed By_____

CRITERIA

Reason for ICAL: Why was this ICAL performed?_____

YES	NO	
☐	☐	Average response factor used? Was the ≤15% RSD for 8270C or ≤20% for 8270D criteria met for all compounds? PCP 8270C requires ≤30% on average before switching curve fit. No outliers should be allowed for compounds of interest.
☐	☐	Linear response factor used? Was the >0.99 criteria met for all compounds?
☐	☐	Quadratic response factor used? Are there a minimum of six points used? Was the >0.99 criteria met for all compounds?
	☐ **Review**	If ICAL criteria were not met, list outliers, explain why samples were analyzed and impact on reported results. Also discuss corrective actions taken to bring Ical into compliance.
☐	☐	Was a standard at the reporting limit (PQL) analyzed and used as part of the calibration? Level 2 for PAH and CPAH waters and PCP. Level 4 for PAH and CPAH soils. Level 3 for 14dioxane.
	☐ **Review**	
☐	☐	Does the PRL standard meet ±40% (for all curve fits) criteria for MN or ±50% for non MN for each analyte set to linear or quadratic and ±30% of the true value for all levels of a linear or quadratic curve above the RL? All extended CPAH compounds get ±50% of the true value for all levels above the RL for linear or quadric curves. If not, was the reporting limit raised? If the ICAL is more than 30 days old, the reporting limit must be verified every 30 days.
	☐ **Review**	
☐	☐	Was ICAL verified against a second source standard(ICV)? Was ICV processed as a sample in original target folder? Apply S-MN-Q-275 criteria (Ccal criteria on a client specific basis, 70-130%) for acceptance. List any outliers:
	☐ **Review**	
☐	☐	Reason for Manual Integrations:
	☐ **Approved**	☐ Retention Time Shift ☐ Baseline Correction ☐ Split Peak ☐ Peak Assignment ☐ Other
☐	☐	Complete curve uploaded into Gandalph and correct Cal Time showing on all files?
	☐ **Review**	List Cal Time_____
☐	☐	Are concentrations set correctly in the method? PAH concentrations are 0.02, 0.04, 0.1, 0.3, 1, 3, 5, 10. Surrogate concentrations are 0.024, 0.048, 0.12, 0.36, 1.2, 3.6, 6, 12.
	☐ **Review**	

ATTACHMENT III – PAH Analyte Lists

List 1 – Short PAH List

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
Napthalene-dS*
2-Chloronapthalene
Acenaphthylene
Acenaphthene
Dibenzofuran
Fluorene
Acenaphthene*
Phenanthrene
Anthracene
Carbazole
Fluoranthene
Phenanthrene-d10*
Pyrene
Benzo(a)anthracene
Chrysene
Chrysene-d12*
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene
Benzo(a)pyrene
Perylene-d12*
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

List 2 – Extended PAH List

Quinoline
5-Nitroacenaphthene
2-Nitrofluorene
4-Nitropyrene
5-Methylchrysene
1-Nitropyrene
7,12-Dimethybenz(a)anthracene
Benzo (j) fluoranthene
6-Nitrochrysene
Perylene
3-Methylcholanthrene
1,6-Dinitropyrene
1,8-Dinitropyrene
Dibena(a,h)acridine
Dibenz(a,j)acridine
7H-Dibenzo(c,g)carbazole
Dibenzo(a,e)pyrene
Dibenzo(a,h)pyrene
Dibenzo(a,i)pyrene
Dibenzo(a,l)pyrene

MDH Primary List

Anthanthrene
Benz(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(c)fluorene
Benzo(g,h,i)perylene
Benzo(j)fluoranthene
Benzo(k)fluoranthene
Chrysene
Dibenz(a,h)anthracene
Dibenzo(a,e)pyrene
Dibenzo(a,h)pyrene

Dibenzo(a,i)pyrene
Dibenzo(a,l)pyrene
Fluoranthene
Indeno(1,2,3-cd)pyrene
5-Methylchrysene
6-Nitrochrysene

MDH Secondary list
Dibenz(a,h)acridine
Dibenz(a,j)acridine
7H-Dibenzo(c,g)carbazole
7,12-Dimethylbenz(a)anthracene
3-Methylchloranthrene
5-Nitroacenaphthene
2-Nitrofluorene
1-Nitropyrene
4-Nitropyrene

List 3 – Pentachlorophenol
Phenanthrene-d10*

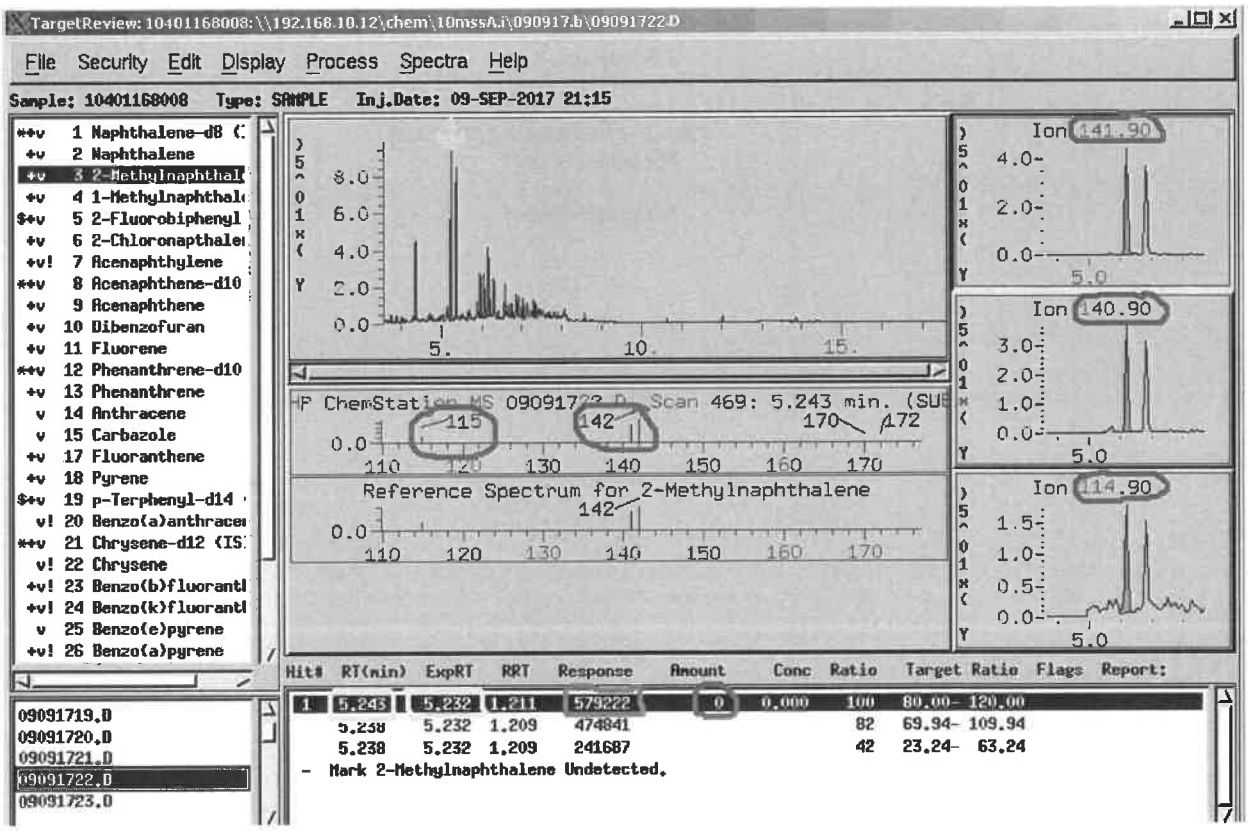
* Internal Standard

Attachment IV: Large Hits Quanting to Zero

Pace Analytical	Document Name: **Large Hits Quanting to Zero**	Document Revised: 18Dec2017 Page 1 of 2
	Document No.: **F-MN-O-338-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

Large Hits Quanting to Zero

How to determine if Amount is really zero or if it is so far above calibration range its quanting to zero



If response is a high number, usually >1000 (579222)

If the peak is in the right RT, <0.06 from ExpRT (5.243RT, 5.232ExpRT)

If all ions are present (141.90, 140.9 and 114.90). They do not need to necessarily match the reference spectrum but they need to present (other matrix in the sample may be skewing the ion ratio height and they may appear to be smaller than reference spectrum).

Amount is (0) but all other criteria is met so peak should be left in and a dilution should be ran to capture peak area. Dilution should be started at 50x to determine what the peak area is. A higher or lower dilution may be required depending on calibration curve range.

The Blue arrow is pointing to the total ion on the chrome which shows the peak is very large compared to the rest of the peaks on the chrome which should trigger you to look at the response not the amount.

Attachment IV: Large Hits Quanting to Zero (Continued)

Pace Analytical®	Document Name: **Large Hits Quanting to Zero**	Document Revised: 18Dec2017 Page 2 of 2
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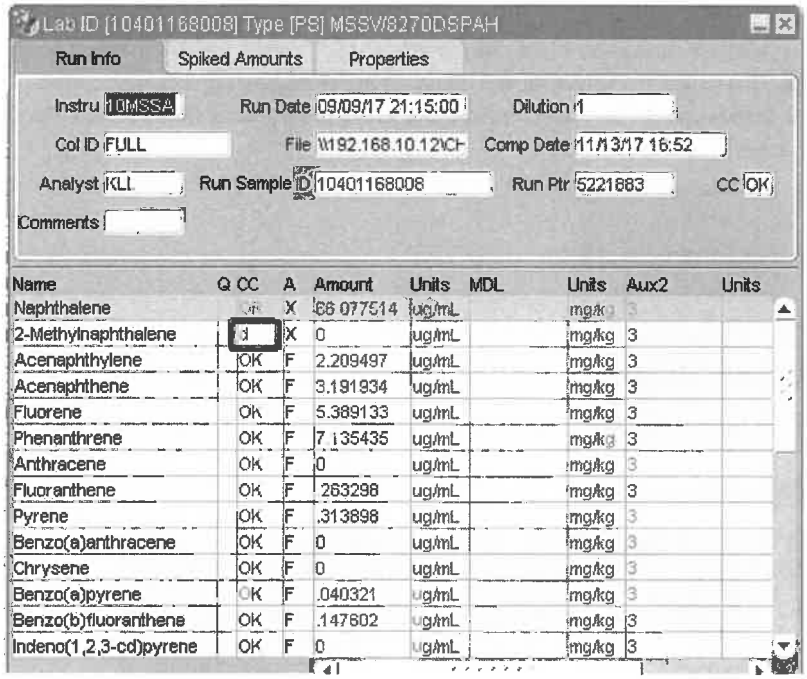
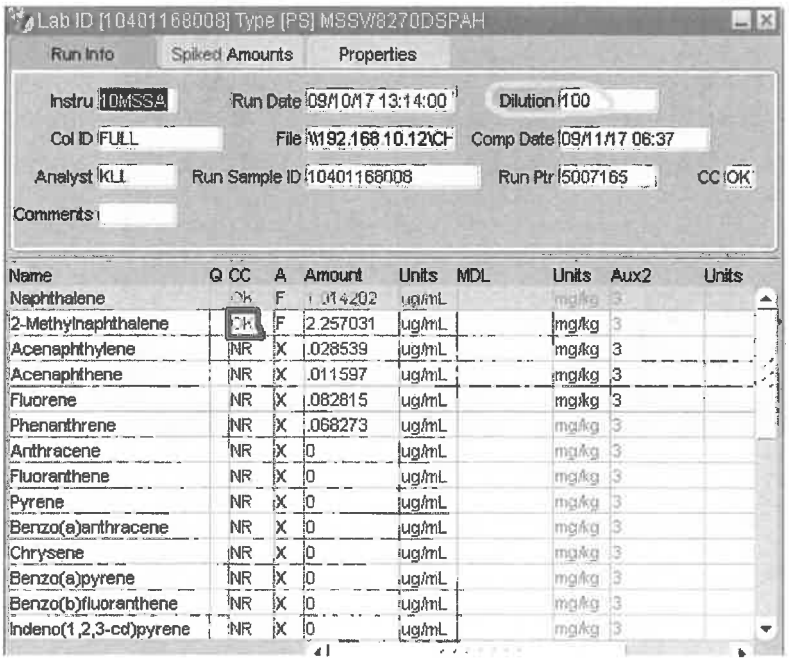
When uploading the data, horizon will not recognize that a dilution is needed. In the CC (condition code) column you will have to change "OK" to d (don't pick this result to report).



Name	Q	CC	A	Amount	Units	MDL	Units	Aux2	Units
Naphthalene			X	66.077514	ug/mL		mg/kg	3	
2-Methylnaphthalene		d	X	0	ug/mL		mg/kg	3	
Acenaphthylene		OK	F	2.209497	ug/mL		mg/kg	3	
Acenaphthene		OK	F	3.191934	ug/mL		mg/kg	3	
Fluorene		OK	F	5.389133	ug/mL		mg/kg	3	
Phenanthrene		OK	F	7.135435	ug/mL		mg/kg	3	
Anthracene		OK	F	0	ug/mL		mg/kg	3	
Fluoranthene		OK	F	.263298	ug/mL		mg/kg	3	
Pyrene		OK	F	.313898	ug/mL		mg/kg	3	
Benzo(a)anthracene		OK	F	0	ug/mL		mg/kg	3	
Chrysene		OK	F	0	ug/mL		mg/kg	3	
Benzo(a)pyrene		OK	F	.040321	ug/mL		mg/kg	3	
Benzo(b)fluoranthene		OK	F	.147602	ug/mL		mg/kg	3	
Indeno(1,2,3-cd)pyrene		OK	F	0	ug/mL		mg/kg	3	

When uploading the dilution in horizon you will have to change the NR to OK



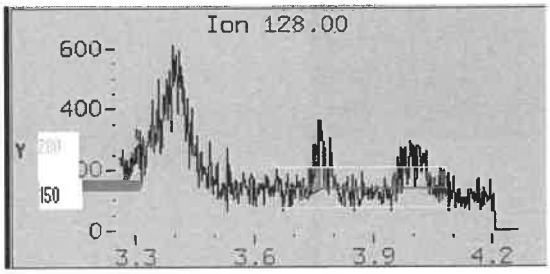
Name	Q	CC	A	Amount	Units	MDL	Units	Aux2	Units
Naphthalene		OK	F	1.014202	ug/mL		mg/kg	3	
2-Methylnaphthalene		OK	F	2.257031	ug/mL		mg/kg	3	
Acenaphthylene		NR	X	.028539	ug/mL		mg/kg	3	
Acenaphthene		NR	X	.011597	ug/mL		mg/kg	3	
Fluorene		NR	X	.082815	ug/mL		mg/kg	3	
Phenanthrene		NR	X	.068273	ug/mL		mg/kg	3	
Anthracene		NR	X	0	ug/mL		mg/kg	3	
Fluoranthene		NR	X	0	ug/mL		mg/kg	3	
Pyrene		NR	X	0	ug/mL		mg/kg	3	
Benzo(a)anthracene		NR	X	0	ug/mL		mg/kg	3	
Chrysene		NR	X	0	ug/mL		mg/kg	3	
Benzo(a)pyrene		NR	X	0	ug/mL		mg/kg	3	
Benzo(b)fluoranthene		NR	X	0	ug/mL		mg/kg	3	
Indeno(1,2,3-cd)pyrene		NR	X	0	ug/mL		mg/kg	3	

Attachment V: J Flag Hits vs Noise

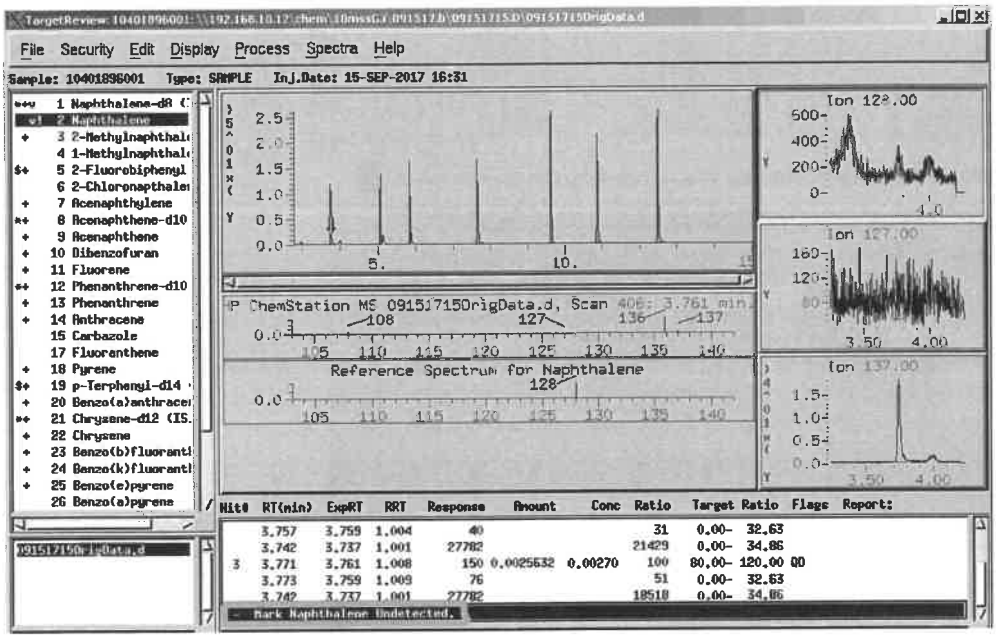
Pace Analytical	Document Name: **J Flag Hits vs Noise**	Document Revised: 19Dec2017 Page 1 of 2
	Document No.: **F-MN-O-339-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

J Flag Hits vs Noise

Identify signal to noise. To do this you'll have to visualize where the minimum and maximum noise lines would be and then take the average of that noise. That would be the base of your signal. The apex of the peak should be 3 times greater than the base of the signal. In this example the base of the signal is 150, 3 times greater would be 450. The apex of the peak is around 280 which does not meet the criteria.



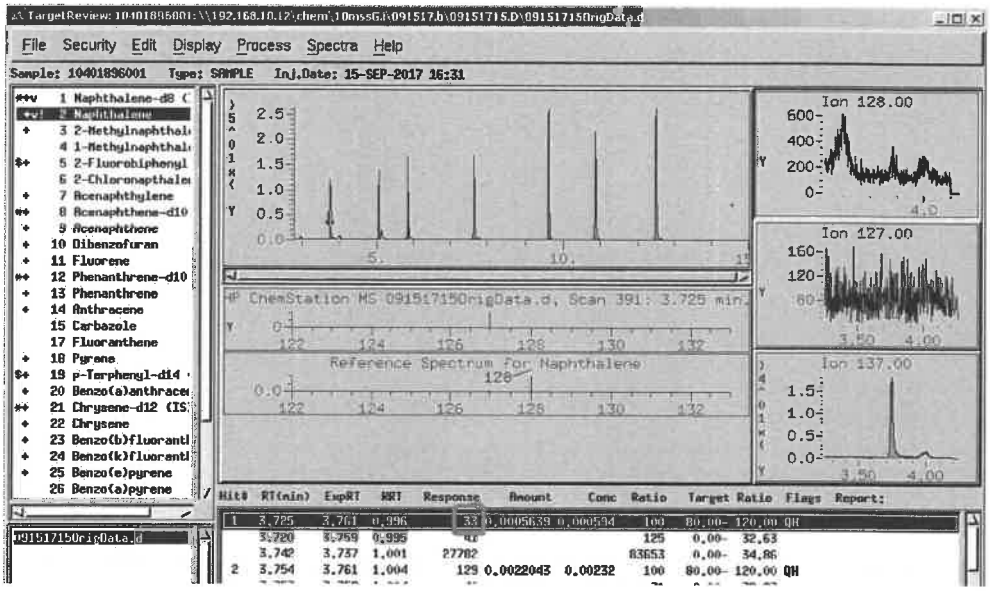
This detection should be undetected or ND(see below).



Attachment V: J Flag Hits vs Noise (Continued)

Pace Analytical®	Document Name: **J Flag Hits vs Noise**	Document Revised: 19Dec2017 Page 2 of 2
	Document No.: **F-MN-O-339-Rev.00**	Issuing Authority: Pace Minnesota Quality Office

Response should be ≥100 (33)-this peak should be un-detected



Document Information

Document Number: ENV-SOP-MIN4-0069 **Revision:** 00

Document Title: 1,4-Dioxane Extraction and Analysis in Liquid Matrices by GC/MS: Capillary Column Technique

Department(s): SVOA

Previous Document Number: S-MN-O-591-rev.02

Date Information

Effective Date: 11 Jul 2018

Next Review Date: 11 Jul 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

1,4-DIOXANE EXTRACTION AND ANALYSIS IN LIQUID MATRICES BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE

Reference Methods: SW846 Modified 8270D

Local SOP Number:	S-MN-O-591-rev.02
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-591-rev.01

APPROVALS

Laboratory General Manager — Date: 11 Jul 2018

Laboratory Quality Manager — Date: 11 Jul 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-591-rev.02

TABLE OF CONTENTS

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. The purpose of this Standard Operating Procedure (SOP) is to set forth the procedure used for the determination of 1,4-dioxane in liquids that are partitioned into an organic solvent and are amenable to gas chromatography/mass spectrometry by EPA method 8270D by SIM.

2. SUMMARY OF METHOD

2.1. This standard operating procedure describes the preparation and analysis of 1,4-dioxane in water per extraction method 3510. A 100 mL aliquot of sample is extracted with solvent Methylene Chloride.

3. SCOPE AND APPLICATION

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method process.

3.2. **Parameters**: This method is used to determine the concentration of 1,4-Dioxane.

4. APPLICABLE MATRICES

4.1. This SOP is applicable to liquid matrices only.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. Analyte and quantitation limits are shown in Table I. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. INTERFERENCES

6.1. Matrix interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Table 7.1 - Sample Collection, Preservation and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	100mL amber glass bottles	Unpreserved	<6°C but above freezing	Must be extracted within 7 days from collection and analyzed within 40 days of extraction.

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Gas Chromatograph/Mass Spectrometer	GC/MS with autosampler	Agilent/7890**B**/5977B and autosampler 7693 or equivalent

Column	ZB-Semivolatiles, 30 m x 0.25 mm bonded-phase silocone coated fused silica capillary column, 0.5 um film thickness	Phenomenex Part #7HG-G027-17
Liner	Liner for Agilent (4mm ID x 78.5mm L x 6.3mm OD), Split/Splitless, Dual Taper, 5/Pk	Phenomenex/AGO-8173
Acquisition Software	Used for acquisition of data	MassHunter
Processing Software	Used for data processing	Target, see master list for current version
Reporting Software	Laboratory Information Management System	Horizon, see master list for current version
Data Package Software	Used for data review and level 4 data packages	Gandalf, see master list for current version
Syringes	10 µL- 1.0 mL	Hamilton or equivalent
Sodium Sulfate	Anhydrous Sodium Sulfate baked at 400°C for minimum of 4 hours (see prep SOP S-MN-O-500 or equivalent replacement)	Fisher Scientific or equivalent replacement
Sodium Chloride	NaCl baked at 400*C for 4 hours	Fisher Scientific Cat.#S-271-10
Sep Funnels	250 mL	ThermoScientific or equivalent
Glass Wool		Fisher Cat.# 10-368B or equivalent replacement
Metal Funnel	Stainless Steel	Fisher Cat.# 10-368B or equivalent replacement
Erlenmeyer	250 mL	Fisher Cat.# 07250090 or equivalent replacement
Buchi	Concentrator with F-108 recirculating chiller, concentrator vessels with V-855 vacuum controller	Buchi, or equivalent replacement
pH strip	Wide range(0-14)	Fisher Catalog #09-876-17 or equivalent
Pipettes	Disposable 1 mL and 2 mL	Fisher Catalog #13-678-31E or equivalent
Centrifuge	Top loading centrifuge	IBC CentraGP8 or equivalent
Auto Sampler Vials	Amber 2 mL autosampler vials with Teflon lined crimp seal caps	Fisher Catalog #03-375-3S or equivalent

9.2. Example GC/MS operating conditions:

Dwell Time per ion:	30 to 100 µS
Temperature Program:	50°C, hold for 2 minutes 25°C/min to 325°C, hold for 3.25 minutes
Injection Temperature:	250°C
Transfer Line Temperature:	300°C
Sample Volume:	2 uL, pulsed splitless injection
Carrier Gas:	Helium
Pressure	10 PSI, Ramped Pressure
Pressure Program	Value psi 10 hold for 2 min Ramp rate psi/min 10, value psi 10 hold 18 min
Scanning range	m/z 50 to 500

10. REAGENTS AND STANDARDS

10.1. Table 10.1 - Reagent and Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Solvent	Methylene chloride and Acetone – optima grade	Fisher Scientific or equivalent replacement

1,4-dichlorobenzene-d4 Internal Standard Mix	4000 µg/mL	Phenomenex Cat# AL0-130249
1,4-dioxane Initial Calibration Stock Standard	2000 µg/mL	O2Si Cat # 020223-01-02
1,4-dioxane (ICV) Solution	2000µg/mL	Phenomenex CAT# AL0-101313
DFTPP	1000 µg/mL	Accustandard Cat #M-625-TS-20X
1,4-dioxane-d8	2000 µg/mL	Phenomenex Cat# AL0-130235

10.2. 14DIOX-SPK (brought to volume with acetone):

Compound	Conc.	Amount	Final Volume	Final Conc.
1,4-dioxane (O2Si)	2000 µg/mL	100 uL	1 mL	200 µg/mL

10.3. 14DIOX-SS is utilized as an internal standard in calculations (brought to volume with acetone):

Compound	Conc.	Amount	Final Volume	Final Conc.
1,4-dioxane-d8 (Phenomenex)	2000 µg/mL	100 uL	1 mL	200 µg/mL

10.4. Recovery Standard – 10 µL 14DIOX-IS added to all calibrations and extracts (samples and quality control) Recovery standard is brought to volume with methylene chloride.

Solution	Parent Conc.	Amount	Final Volume	Final Conc.
1,4-dichlorobenzene-d4 (Phenomenex)	4000 µg/mL	0.250 mL	10 mL	100 µg/mL

10.5 14diox-INT - brought to volume with methylene chloride.

Compound	Conc.	Amount	Final Volume	Final Conc.
1,4-dioxane (O2Si)	2000 µg/mL	0.025 mL	5 mL	10 µg/mL

10.6 Initial Calibration Example for 1,4-dioxane (see section 10.5 for stock/parent solution). Note: all levels of the initial calibration are brought to volume with methylene chloride.

Level	Parent Conc.	Amount	Final Volume	Methylene Chloride amount	Final Concentration
14DX-CAL1	1 µg/mL (CAL6)	0.004 mL	1 mL	0.996 mL	0.004 µg/mL
14DX-CAL2	10 µg/mL	0.001 mL	1 mL	0.999 mL	0.01 µg/mL
14DX-CAL3 (RL)	10 µg/mL	0.002 mL	1 mL	0.998 mL	0.02 µg/mL
14DX-CAL4	10 µg/mL	0.010 mL	1 mL	0.990 mL	0.1 µg/mL
14DX-CAL5	10 µg/mL	0.020 mL	1 mL	0.980 mL	0.2 µg/mL
14DX-CAL6 (CCV)	10 µg/mL	0.100 mL	1 mL	0.900 mL	1 µg/mL
14DX-CAL7	10 µg/mL	0.200 mL	1 mL	0.800 mL	2 µg/mL
14DX-CAL8	10 µg/mL	0.500 mL	1 mL	0.500 mL	5 µg /mL

* Final concentration based on 100mL extraction

Note: 5 µL of the 14DIOX-SS (section 10.3) as well as 10 µL 14DIOX-IS (section 10.4) is added to each level of the calibration (resulting in a concentration of 1 µg/mL at a constant concentration in all levels of the calibration).

10.7 Initial Calibration Verification Standard ICV Intermediate (14dioxICVINT)-brought to volume with methylene chloride.

Compound	Conc.	Amount	Final Volume	Final Conc.
1,4-dioxane (Phenomenex)	2000 µg/mL	0.010 mL	1 mL	20µg/mL

10.8 Initial Calibration Verification (14DX-ICV) – brought to volume with methylene chloride.

Solution	Parent Conc.	Amount	Final Volume	Final Conc.
14dioxICVINT	20 µg/mL	0.050 mL	1 mL	1 µg/mL

Note: 5 µL of the 14DIOX-SS (section 10.3) as well as 10µL 14DIOX-IS (section 10.4) is added to the ICV solution.

10.9 MDL 14diox-SPK (14dioxSPKMDL) – brought to volume with methylene chloride.

Solution	Parent Conc.	Amount	Final Volume	Final Conc.
14diox-SPK	200 µg/mL	0.05 mL	1 mL	10 µg/mL

Spike 2.5uL into each MDL rep=0.25 on column for true value.

10.10 DFTPP – brought to volume with methylene chloride.

Solution	Parent Conc.	Amount	Final Volume	Final Conc.
DFTPP (Restek or equiv)	1000 µg/mL	0.05 mL	1 mL	50 µg/mL

11. CALIBRATION AND STANDARDIZATION

11.1 Table 11.1 - Calibration and Standardization

Calibration Metric	Parameter / Frequency	Criteria	Comments
Tune	Analyzed at the beginning of every 12 hour sequence	See Table II	It not met, perform system maintenance including injection port maintenance (septa, liner, gold seal, split lines, etc) up to and including re-preparing solutions.
Initial Calibration Curve Fit (ICAL)	Average Response Factor Linear Regression Non-linear Regression Following an acceptable tune; conducted as needed, following repeat continuing calibration failures	≤15% $r \geq 0.990$ COD ≥ 0.990 with a minimum of 6 levels	If not met, try non-linear regression fit. If still not met, remake standards and recalibrate and verify before sample analysis. Perform and document maintenance conducted to return to compliance.
Second Source Verification Standard (ICV)	Immediately after each ICAL; must be a second source standard.	% Diff ±20%	If the criteria is not met, verify the preparation of the standard. Analyze one additional time. If it fails a second time, perform any necessary maintenance and recalibrate the instrument followed by another ICV prior to conducting sample analysis.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 20 injections thereafter. Samples must be bracketed with a closing CCV standard.	% Diff ±20% compared to the response factor of the ICAL.	If the requirements for continuing calibration are not met, verify standard preparation or injection error. Reanalyze one additional time. If the second injection fails, perform necessary instrument maintenance and recalibrate the instrument. Samples that were not bracketed by an acceptable CCV must be reanalyzed.

Recovery Standard (1,4-dichlorobenzene-d4)	Evaluated in each analytical run for retention time and EICP area	Retention time must not change more than ±30 seconds from the last check calibration; EICP must not exceed -50% to +100% from the last daily calibration standard check.	If the criteria are not met, the analytical system must be inspected for malfunctions and corrections must be made. Reanalyze and flag if second analysis fails criteria.
Internal Standard (1,4-dioxane-d8)	Each sample and QC.	40-100%	Re-extract and qualify data if the second re-extraction/re-analysis fails to meet criteria.

12. PROCEDURE

12.1 SAMPLE PREPARATION

12.1.1 Samples should be removed from the refrigerator shortly before extraction, and should not be allowed to warm to room temperature before the extraction solvent is added. Samples should be prepared one at a time to the point of solvent addition (i.e., do not prepare a number of samples then add the solvent). Pay particular attention to minimizing the exposure of the sample and/or extract to air.

12.1.2 Sample IDs should be verified by another lab personnel.

12.1.3 Measure the initial volume and record the amount of sample in the 250 mL amber glass container on the extraction sheet.

12.1.4 If more than 5% of the entire sample volume is sediment present in the bottom of the glass container, follow procedure in S-MN-L-142 (or equivalent replacement).

12.1.5 Using pH paper, pipet a small amount of the sample onto a pH strip and record the pH of each sample on the extraction sheet.

12.1.6 Pour 100 mL optima water into 250 mL sep. funnel for MB, LCS and LCSD. Record amount on extraction sheet.

12.1.6.1 The state of WI requires a set of MS/MSD in a batch if the sample is from WI. If there is insufficient sample for a MS/MSD the extra container from the state of WI must be split into 3 portions (parent, MS and MSD). A placeholder must be requested from the PM and used as the parent sample. The volume will then be brought up for extraction purposes.

12.1.7 Add 12g of NaCl to all QC, blanks and sample separatory funnel.

12.1.8 Add 5uL of 14diox-SS (table 10.3) to all QC and blanks and samples. Add 5uL of 14diox-SPK (table 10.2) to all LCS, LCSD, MS or MSD.

12.1.9 Pour each sample into their respective separatory funnel.

12.1.10 Add 6 mL of methylene chloride to each sample jar (unless the sediment is greater than 5% of the sample volume- if so, see section 12.1.2), cap, shake and decant rinsate into the appropriate separatory funnel. Load sep funnels into tumbler and tumble for about 5 rotations. Stop the tumbler with the seps in the upside-down position and vent each separatory funnel to release the pressure. Set timer; funnels are tumbled for 4 minutes at a speed of 55 RPM. Alternatively, shake the samples vigorously by hand for two minutes.

12.1.11 After 4 minutes stop the tumbler and allow the layers to separate for 10 minutes. Drain the solvent layer through a stainless steel funnel that contains a glass wool plug and sodium sulfate all having been rinsed with methylene chloride, into an Erlenmeyer flask that has also been rinsed with methylene chloride. Rinse funnel with Methylene Chloride after sample has been allowed to drain.

12.1.12 If an emulsion has formed, make a note on the extraction sheet.

12.1.13 Repeat the extraction two more times using a 6 mL aliquot of methylene chloride for each tumble. Collect the solvent in the same flask. Rinse funnel again after all extract is through the sulfate.

12.1.14 Cover flask with foil and store in cooler above freezing but below 6 °C until the concentration procedure or follow Buchi concentration procedure below.

12.1.15 Buchi Concentration:

12.1.15.1 Make sure the receiving flask and secondary receiving flask are free of solvent before use.

12.1.15.2 Turn on the F-108 recirculating chiller. Set the temperature to 10 °C and press the start button. Wait approximately 5 minutes for the chiller to reach the set temperature.

12.1.15.3 Turn on the Syncore® platform. Set the temperature to 60°C and press the start button.

12.1.15.4 Allow approximately 20 minutes for the platforms to reach the set temperature.

12.1.15.5 Add approximately 10 mL of DI water to each cell of the rack to help with heat transfer. The platform will not concentrate correctly without water in the cells. Make sure there is enough water causing overflow when the vessel is placed in the cell.

12.1.15.6 All Buchi concentration vessels must be cleaned by soap wash. Refer to SOP S-MN-O-465 for glassware cleaning procedure section 12.3.

12.1.15.7 Add baked sodium sulfate to all extracts to remove any water.

12.1.15.8 Label the vessels with batch prep labels or Sharpie.

12.1.15.9 Transfer the extract from the Erlenmeyer to the concentration vessel. Rinse the Erlenmeyer with approximately 10 mLs of methylene chloride three times and transfer to the vessel each time. Make sure no sodium sulfate is transferred into the concentration vessel.

12.1.15.10 Insert the vessels into the cells and evenly tighten down the vacuum cover.

12.1.15.11 Turn the rotation dial and set the speed to 250 RPM.

12.1.15.12 Select the 1,4 DIOX program that is set in the V-855 vacuum controller.

12.1.15.12.1 To select the desired program select "Menu".

12.1.15.12.2 Arrow down and select "program".

12.1.15.12.3 Arrow down to "open" and turn the dial to select desired program. Once program is highlighted select "ok".

12.1.15.12.4 Press "start" to run the program.

12.1.15.13 Once the program is finished select "Stop" and allow the vacuum to return to 1000 mbar. Remove the vacuum cover and take out the vessels. If a sample is higher than 1 mL place it on the N-EVAP and bring down to 1 mL.

12.1.15.14 Finalize using a calibrated disposable pipet into a 2 mL vial. All samples should be stored in the freezer.

12.1.16 Extracts should be stored in a freezer.

12.2 GC/MS ANALYSIS

12.2.1 Add 10 µL of the recovery standard, 1,4-dichlorobenzene-d4 (14diox-IS) (100 µg/mL) to each extract.

12.2.2 Analyze sample using a GC/MS system with the parameters outlined in section 9.2. A typical run sequence includes: instrument blank, tune, initial or continuing calibration, up to 20 injections of samples and quality control samples (method blank, laboratory control spikes, matrix spike/duplicates).

12.2.3 Perform all qualitative and quantitative measurements as described in section 12.3 - Data Interpretation.

12.2.4 Samples following high standards or over-range samples are to be monitored for carryover.

12.2.5 Internal Standards Evaluation (1,4-dioxane-d8) - Internal standard responses and retention times in all samples must be evaluated.

12.2.6 Evaluate Recovery Standard (1,4-dichlorobenzene-d4). Criteria are defined in Table 11.1 and corrective actions in 16.3.

12.2.7 Each analytical run must also be checked for saturation (the level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound). The initial method calibration requires the system should not be saturated for high response compounds.

12.3 DATA INTERPRETATION

12.3.1 Qualitative Analysis – The target compounds shall be identified by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: 1) elution of the sample component at the same GC relative retention time as the standard component, and 2) correspondence of the sample component and standard component mass spectra.

12.3.2 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For comparison purposes, the RRT should be evaluated against the standard analyzed prior to the sample.

12.4 QUANTITATION

12.4.1 Target components identified shall be quantified by the isotope dilution method.

12.4.2 The EICP area of characteristic ions of analytes listed in Table I. The average response factor (RF) from the initial calibration is used to calculate the concentration in the sample.

12.4.3 Calculate the concentration in the sample using the average response factor from the initial calibration and Equation 5.

12.4.4 Calculate surrogate standard recovery (1,4-dioxane-d8 (S)) on all samples, blanks and spikes. Determine if recovery is within limits; see Equation 6.

13. QUALITY CONTROL

13.1 Table 13.1 - Quality Control Criteria

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples or less	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be evaluated to the same criteria and data should be flagged appropriately.	Investigate the source of the detection. Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.

Laboratory Control Sample (LCS)	DI water spiked with all target compounds	One per 20 samples less	Internally generated limits generated on an annual basis RPD is 20%	Repeat the analysis of the LCS; If problem persists, check spike solution; Perform system maintenance prior to new LCS run. RPD failures will be flagged but samples do not need to be re-run. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 20 samples or less	Internally generated limits generated on an annual basis	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One per 20 samples or less	Individual component recoveries of the matrix spike are calculated using Equation 6. The reproducibility of RPD of the MSD is calculated using Equation 7. RPD is 20%	MSD usually fail due to matrix interference, all failures are flagged in horizon. RPD failures will be flagged but samples do not need to be re-run.
Internal Standard/Surrogate(1,4-dioxane-d8)	Spiking standard prepared in acetone	Each sample and QC.	Internally generated limits generated on an annual basis. Internal standard portion of 1,4-dioxane-d8 will usually fail the recovery standard criteria because it is injected before extraction. This is acceptable.	If surrogate fails internally generated limits, re-extract and qualify data if the second re-extraction/re-analysis fails to meet criteria.
Recovery Standard (1,4-Dichlorobenzene -d4	Spiking standard prepared in acetone	Each sample and QC.	Area Count -50 – +100% relative to the continuing calibration verification	Re-analyze and qualify the second analysis if it fails criteria.

14. DATA ANALYSIS AND CALCULATIONS

14.1 Calculate response factors (RFs) for each compound as follows:

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg /mL).
C_x = Concentration of the compound being measured (µg /mL).

14.2 The percent relative standard deviation (%RSD) is calculated as follows:

Equation 2

$$\%RSD = \frac{SD}{\overline{RF}} x100$$

Where: $\overline{RF}$ = Mean of the Response Factors mentioned above.

SD = Standard Deviation of initial response (see Equation 3).

14.3 Standard Deviation of Initial Response

Equation 3

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_1 - \overline{RF}\right)^2}{n-1}}$$

Where: RF_1 = Each individual response factor
$\overline{RF}$ = Mean of the Response Factors mentioned above.
N = Number of response factors

14.4 The Percent Difference (%D) is calculated as follows:

Equation 4

$$\%Difference = \frac{\left(RF_i - RF_c\right)}{RF_i} x100$$

Where: Rfi = Average response factor from initial calibration
RFc = Response factor from current verification check standard

14.5 The concentration of analyte in the sample is calculated as follows:

Equation 5

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF}_i)(W_o)(V_i)}$$

Where: A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
W_o = Volume of sample extracted in liters
V_i = Volume of extract injected (µL)
V_t = Volume of total extract (mL)
$\overline{RF}$ = Average response factor from initial calibration

14.6 Calculate the Matrix Spike Percent Recovery as follows:

Equation 6

$$\%Recovery = \frac{SSR - SR}{SA} x100$$

Where: SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

14.7 The laboratory will calculate the relative percent difference between the laboratory control spike and control spike duplicate, the matrix spike and matrix spike duplicate, and/or between laboratory samples and sample duplicates. The relative percent differences (RPD) for each component are calculated using the following equation:

Equation 7

$$RPD = \frac{|A - B|}{(A + B)/2} x100$$

Where: RPD = Relative Percent Difference
A = First Sample Value

B = Second Sample Value (duplicate)

14.8 The quantitation of this method is using isotope dilution technique by adding 1,4-Dioxane-d8 prior to extraction. The 1,4-Dioxane-d8 is designated as an internal standard in the quantitation software (Target) in order to correct for any losses seen in the preparation of the sample (if 20% recovery is lost for 1,4-Dioxane-d8, 1,4-Dioxane will be corrected/compensated for this loss and adjusted back up 20% by this technique, giving a better representative of the true amount of 1,4-Dioxane in samples). The internal standard report should have 1,4-Dioxane-d8 and the recovery standard of 1,4-Dichlorobenzene-d4. If the 1,4-Dioxane-d8 recovery (based on the recovery standard) is outside of acceptance criteria not due to an obvious matrix interference, re-analysis needs to occur. If the re-analysis confirms that 1,4-Dioxane-d8 is outside of acceptance limits, and sufficient sample remains, re-extract the sample.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1 See tables in section 11 & 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Quantitation ions - If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution may take place or the client may be contacted to see if a dilution is warranted (dilutions may become estimated due to the nature of isotope dilution). If a dilution is necessary, utilize the following procedure:

16.1.1 The sample extract needs to be analyzed as an undiluted run in order to report the extraction efficiency for 1,4-dioxane-d8.

16.1.2 Dilute the sample and refortify with recovery standard to the constant level of 1.0 ug/mL. If the internal standard is still at least 10x the noise; quantitate as normal and include the dilution factor used.

16.1.3 If the signal to noise ratio is less than 10, and if sufficient sample volume remains, re-extract utilizing a lower volume, targeting a value of the mid point or higher of the 1,4-dioxane concentration obtained from the original extraction.

16.2 Internal Standards Evaluation (1,4-dioxane-d8) - Internal standard responses and retention times in all samples must be evaluated.

16.2.1 If the retention time for any internal standard changes by more than 30 seconds, then the analytical system must be inspected for malfunctions and corrections made as required.

16.2.2 If the extracted ion current profile (EICP) area for any internal standard changes by more than - 40-100% from the latest daily calibration standard, the MS system must be inspected for malfunction and corrections made as appropriate.

16.2.3 14diox-IS recovery (based on the recovery standard) is outside of acceptance criteria not due to an obvious matrix interference, re-analysis needs to occur. If the re-analysis confirms that 14diox-IS is outside of acceptance limits, and sufficient sample remains, re-extract the sample. If re-extraction confirms, report the original extraction with a qualifier that the sample was re-extracted and the results confirmed.

16.3 Recovery Standard (1,4-dichlorobenzene-d4) Evaluation – If the EICP area for any of the recovery standard changes by a factor of two, (-40% to +100%) from the calibration standard check (CCV), re-analyze the sample. If the second analysis confirms the original analysis, qualify the data accordingly using the Horizon footnotes.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable

qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. METHOD PERFORMANCE

18.1 All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269, Method Detection Limit Studies (or equivalent replacement) for each matrix per instrument.

18.3 **Demonstration of Capability (DOC)**: An initial demonstration of capability (IDC) must be performed. A record of the IDC will be maintained in his/her file with written authorization from the Laboratory Manager and Quality Manager. Results are stored in the QA office. Every analyst who performs this method must continue to document acceptable accuracy and precision by passing an annual demonstration of capability study (DOC) per S-MN-Q-279, Training and Employee Orientation (or equivalent replacement).

19. METHOD MODIFICATIONS

19.1 We are using a modified 3510D extraction procedure. Instead of using 1L of samples, 100mL of samples is used.

19.1.1 12g of NaCl is added to all QC, blanks and samples in the separatory funnel.
19.1.2 6mL instead of 60mL of methylene chloride for each shake.
19.1.3 Spiked surrogate also has an internal standard present and extraction is performed as an isotope dilution.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1 Please refer to the GC/MS 7890 instrument manual for maintenance procedures performed by the lab.

20.2 All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3 Additional activities are outlined in SOP S-MN-L-114, Preventive, Routine and Non-Routine Maintenance and SOP S-MN-Q-264, Support Equipment or equivalent replacements.

21. TROUBLESHOOTING

21.1 The tune should help serve as a troubleshooting guide for column degradation and standard issues. Inlet cleaned such as replacing the liner and cleaning or replacing the gold seal should be done after running samples with matrix.

22. SAFETY

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

23.1 Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. POLLUTION PREVENTION

24.1 The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

25.1 Pace Quality Assurance Manual- most current version.

25.2 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update IV, Method 8270D.

25.5 Contract Laboratory Program Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM01.2)

25.6 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, July 2014, Revision 4, Method 8000D.

25.7 Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), February 2007, Revision 4.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1 Attachment I: 1,4-Dioxane Water Extraction Sheet (example)

26.2 Attachment II: 1,4-Dioxane SVOA Analyst Checklist (example)

26.3 Attachment III: Initial Calibration Verification Checklist – 1,4-Dioxane Water (example)

26.4 Table I - Analytes and Quantitation Limits

26.5 Table II - Ion Abundance Criteria for DFTPP

27. REVISIONS

SOP Number	Revisions	Date
S-MN-O-591-Rev.00	Original SOP	27Mar2018
S-MN-O-591-Rev.01	Added section 12.1.6.1 for state of WI. Added "For WI samples…" exception to Table 13.1, MB row, Corrective Action column. Removed section 12.3.3, left in from Sulfolane SOP (used as template) New revision of Attachment II.	10May2018
S-MN-O-591-Rev.02	Added method modifications to section 19.1 and all subsections.	10Jul2018

Attachment I: 1,4-Dioxane Water Extraction Sheet (example)

Pace Analytical® Prep Log Report

Template Version: F-ATL-O-001-Rev.00 (19Jan2018)

Batch Information: OEXT 396801 14 DIOXANE

Prep Method	EPA 3511D	Analysis Method	EPA 8270D by SIM	Extracted By	JLR	Extracted Date/Time	03/27/2018 13:02:34.875
Dispenser ID 1		Syringe ID 1		Syringe ID 2		Syringe ID 3	
Disp. Pipet Lot #		Amber Vial Lot #		Concentrated By		Concentrated By Date/Time	
Conc. Method		Methylene Chloride	73384	Sodium Sulfate	56298	Sodium Chloride	None Added
Glass Wool	None Added	Copper Powder	None Added	Sulfur Cleaned By	N/A	Sulfur Cleaned By Date	
Reviewed By		Reviewed By Date		Batch Notes			

Sample Information:

QC Rule	Sample Type	Lab Sample ID	Client ID	Sample ID Verified By	Spike Verified By	Sample Volume (mL)	Sediment Volume (mL)	Initial Volume (mL)	pH	Final Volume (mL)	Sample Notes	14DIOX-SPK (uL)	14DIOX-SS (uL)
8270DW14DP	BLANK	2172683						100	7	1			73883 (5)
8270DW14DP	LCS	2172684						100	7	1		73886 (5)	73883 (5)
8270DW14DP	LCSD	2172685						100	7	1		73886 (5)	73883 (5)
8270DW14DP	PS	10335634001	PASI-MINN					100	7	1			73883 (5)
8270DW14DP	PS	10335634002	PASI-MINN					100	7	1			73883 (5)

Standard Notes:

Tue, 27 Mar 2018 13:03:17 -0500

Attachment II: 1,4-Dioxane SVOA Analyst Checklist

SIM Semivolatile Analyst Checklist

Prep Batch # _____ Analytical Batch # _____

☐ SIM – Water ☐ SIM – Soil ☐ CPAH – Water ☐ CPAH – Soil ☐ PCP ☐ 14dioxane

YES ☐ NO ☐ **HOLDING TIME**
Extraction holding times met (7 days from collection for waters and 14 days from collection for soils)? And analysis holding time of 40 days from extraction met for all samples? If not, explain and list affected samples:
☐Review

YES ☐ NO ☐ **DFTPP**
Was DFTPP demonstrated every 12 hours and criteria met? If not, explain why samples were analyzed and impact on reported results. Also, discuss any corrective action taken.
☐Review

YES ☐ NO ☐ **INITIAL CALIBRATION(S)**
Did the Initial calibration meet criteria for all compounds (15% RSD Average, 0.995 Linear, or 0.990 Quadratic)? Is the curve fit the same as the original ICAL? If the ICAL is more than 30 days old, the reporting limit must be verified every 30 days. List Ical ID _____
☐Review

YES ☐ NO ☐ **CONTINUING CALIBRATION(S)**
Was the 20%D criteria met for all compounds? If not, list outliers and discuss any impact on the data. Was a Ccal analyzed every 12 hours?
☐Review

YES ☐ NO ☐ **BLANK ANALYSIS**
Was a compliant method blank analyzed on each instrument associated with sample analysis? Were all target analytes present at or below the reporting limit? If not, was the "B" qualifier used? Discuss any impact on the data. Evaluate MB to the MDL for WI. Flag for any detections between MDL and RL.
☐Review

YES ☐ NO ☐ **LABORATORY CONTROL SAMPLE**
Was an LCS performed with each extraction batch? Percent recovery (compared to internally generated limits) of each compound in the LCS within control limits? If not, list compounds, any corrective actions, discuss any impact on data and why sample analysis proceeded. RPD within limits? If not, list compounds and discuss impact on data.
☐Review

YES ☐ NO ☐ **MATRIX SPIKE/MATRIX SPIKE DUPLICATE**
Was a MS/MSD performed with each extraction batch? Percent recovery (compared to internally generated limits) of each compound in the MS/MSD within control limits? If not, list compounds, any corrective actions, discuss any impact on data and why sample analysis proceeded. RPD within limits? If not, list compounds and discuss impact on data.
☐Review

YES ☐ NO ☐ **S.S. AND I.S. RECOVERIES**
Percent recovery of each surrogate compound within control limits? If surrogate outside limits, was sample reanalyzed? List outliers, any corrective action and discuss impact on data.

YES ☐ NO ☐
All samples met internal standard criteria? If not, list outliers, discuss impact on data and note if sample was reanalyzed.
☐Review

YES ☐ NO ☐ **MANUAL INTEGRATIONS**
Reason for manual integrations: ☐Retention Time Shift ☐ Baseline Correction ☐ Split Peak
☐ Peak Assignment ☐ Other (indicate why)_____
☐Approved

ADDITONAL COMMENTS:

"To the best of my knowledge all of the above information is correct and all supporting documentation has been provided."

Analyst: _____ Date: _____

Reviewer: _____ Date: _____

Attachment III: Initial Calibration Verification Checklist – 1,4-Dioxane Water (example)

Pace Analytical®	Document Name: SIM Initial Calibration Verification Form	Document Revised: 26Mar2018 Page 1 of 1
	Document No.: F-MN-O-123-rev.16	Issuing Authority: Pace Minnesota Quality Office

SIM Initial Calibration Verification Form

☐ SIM ☐ CPAH ☐ PCP ☐ HVI ☐ 14Dioxane

ICAL Identification _____

ICAL Exp Date_____ICV_____

Instrument _____

Date of Analysis _____

Analyst _____

Date of Review_____

Reviewed By_____

CRITERIA

Reason for ICAL: Why was this ICAL performed?_____

YES	NO	
☐	☐	Average response factor used? Was the ≤15% RSD for 8270C or ≤20% for 8270D criteria met for all compounds? PCP 8270C requires ≤30% on average before switching curve fit. No outliers should be allowed for compounds of interest.
☐	☐	Linear response factor used? Was the >0.99 criteria met for all compounds?
☐	☐	Quadratic response factor used? Are there a minimum of six points used? Was the >0.99 criteria met for all compounds?
	☐ **Review**	If ICAL criteria were not met, list outliers, explain why samples were analyzed and impact on reported results. Also discuss corrective actions taken to bring Ical into compliance.
☐	☐	Was a standard at the reporting limit (PQL) analyzed and used as part of the calibration? Level 2 for PAH and CPAH waters and PCP. Level 4 for PAH and CPAH soils. Level 3 for 14dioxane.
	☐ **Review**	
☐	☐	Does the PRL standard meet ±40% (for all curve fits) criteria for MN or ±50% for non MN for each analyte set to linear or quadratic and ±30% of the true value for all levels of a linear or quadratic curve above the RL? All extended CPAH compounds get ±50% of the true value for all levels above the RL for linear or quadric curves. If not, was the reporting limit raised? If the ICAL is more than 30 days old, the reporting limit must be verified every 30 days.
	☐ **Review**	
☐	☐	Was ICAL verified against a second source standard(ICV)? Was ICV processed as a sample in original target folder? Apply S-MN-Q-275 criteria (Ccal criteria on a client specific basis, 70-130%) for acceptance. List any outliers:
	☐ **Review**	
☐	☐	Reason for Manual Integrations: ☐ Retention Time Shift ☐ Baseline Correction ☐ Split Peak
	☐ **Approved**	☐ Peak Assignment ☐ Other
☐	☐	Complete curve uploaded into Gandalph and correct Cal Time showing on all files?
	☐ **Review**	List Cal Time_____
☐	☐	Are concentrations set correctly in the method? PAH concentrations are 0.02, 0.04, 0.1, 0.3, 1, 3, 5, 10. Surrogate concentrations are 0.024, 0.048, 0.12, 0.36, 1.2, 3.6, 6, 12.
	☐ **Review**	

TABLE I: Analytes and Quantitation Limits

1,4-Dioxane by SIM	CAS Numbers	Quant	Qual Ions	Liquid Reporting Limit µg/L
1,4-Dioxane-d8 (Internal Std)	1520-96-3	96		
1,4-Dioxane	123-91-1	88	58	0.25
1,4-Dichlorobenzene-d4 (rec std)	3855-82-1	152	150,115	

TABLE II: Ion Abundance Criteria for DFTPP

Mass (M/z)	Relative Abundance Criteria	Purpose of Checkpoint
51	10-80% of the base peak	Low-mass sensitivity
68	< 2% of Mass 69	Low-mass resolution
70	< 2% of Mass 69	Low-mass resolution
127	10-80% of the base peak	Low-to mid-mass sensitivity
197	< 2% of Mass 198	Mid-mass resolution
198	Base peak or > 50% of Mass 442	Mid-mass resolution and sensitivity
199	5-9% of Mass 198	Mid-mass resolution and isotope ratio
275	10-60% of the base peak	Mid- to high-mass sensitivity
365	> 1% of the base peak	Baseline threshold
441	Present and < Mass 443	High-mass resolution
442	Base peak or > 50% of Mass 198	High-mass resolution and sensitivity
443	15-24% of Mass 442	High-mass resolution and isotope ratio

Document Information

Document Number: ENV-SOP-MIN4-0070 **Revision:** 00

Document Title: Analysis of Volatile Organic Compounds by GC/MS Method 8260

Department(s): VOA

Previous Document Number: S-MN-O-521-rev.35

Date Information

Effective Date: 12 Jul 2018

Next Review Date: 12 Jul 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY GC/MS

Reference Methods: SW846 Method 8260B

Local SOP Number:	S-MN-O-521-rev.35
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-521-rev.34

APPROVALS

Laboratory General Manager — Date: 12 Jul 2018

Laboratory Quality Manager — Date: 12 Jul 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-521-rev.35

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to define the process for the determination of volatile organic compounds by gas chromatography/mass spectrometry (GC/MS), capillary column technique per SW-846 method 8260B.

2. Summary of Method

2.1. The volatile organic compounds are introduced into the gas chromatograph by the purge-and trap method. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and back flushed with helium to desorb trapped sample components. The analytes are directly desorbed onto a narrow bore capillary column connected to a split/splitless injection port. The column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph.

2.2. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant mass spectra and GC retention times. Each identified component is quantitated by relating the MS response for an appropriate selected ion produced by that compound to the MS response for an appropriately selected ion produced by an internal standard.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method process.

3.2. **Parameters**: This SOP applies to compounds listed in Attachment I. Additional compounds may be analyzed if all quality control criteria are met.

3.3. Method 8260B can be used to quantitate most volatile organic compounds that have boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately two to ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitrites, acetates, acrylates, ethers, and sulfides.

3.4. Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

4. Applicable Matrices

4.1. This SOP is applicable to a variety of matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, surface water, aqueous sludges, soils, and sediments.

5. Limits of Detection and Quantitation

5.1. All current MDLs and LOQs are listed in the LIMS and are available by request from the Quality Manager.

5.2. Reporting Limits will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

6. Interferences

6.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such

materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should investigate the source of contamination and correct it. Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks results in what the laboratory feels is a false positive for a sample, this should be fully explained in text accompanying the uncorrected data.

6.2. Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing of the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more method blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with methanol, rinse it with organic-free reagent water, and then dry the purging device in an oven less than 120°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning, typically a methanol back flush followed by a DI water back flush. Screening the sample prior to analysis is recommended to prevent system contamination. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique using a flame ionization detector or by analyzing the sample at a dilution by purge and trap GC/MS.

6.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank is prepared using organic-free water (or pre-tested, boiled and/or purged DI water) and carried through the sampling and handling protocol or pre tested, boiled, deionized water can serve as a check on such contamination. Trip blanks may also be purchased premade, refer to the Bottle Preparation SOP, S-MN-C-003, or equivalent replacement.

6.4. Holding blanks consisting of VOA vials of DI water or methanol are placed in the refrigerators and freezers used for the storage of samples for volatile analysis. These blank samples are removed every two weeks and analyzed for the target analytes to determine if cross-contamination has occurred during sample storage.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Sample Collection, Preservation and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	3 VOA vials, approximately 42 mL total volume when capped with no headspace (no headspace is considered less than 6 mm bubble present in container	Acidified with 1:1 hydrochloric acid (HCl) to pH<2; no headspace	<6°C, but above freezing	Must be analyzed within 14 days of collection when properly preserved; 7 days if the pH is >2
Aqueous for Acrolein and Acrylonitrile	3 VOA vials, approximately 42 mL total volume when capped with no headspace (no headspace is considered less than 6 mm bubble present in container	Adjust pH to pH 4-5	<6°C, but above freezing	Must be analyzed within 14 days of collection when properly preserved; 3 days in no pH adjustment is made per 40 CFR Part 136 guidance

Solids – Low level by 5035	2 unpreserved tared 40 mL vial with stirbar and 5 mL DI water	DI water	<6°C, but above freezing	Must be frozen within 48 hours of collection to -10 to -20 °C and analyzed within 14 days of collection
Solids – Medium level by 5035	2 Tared 40 mL vial or wide mouth jar – 25 g capacity Encore, or similar approved sample container and storage device (Terracore)	Methanol	<6°C, but above freezing	Must be analyzed within 14 days of collection when properly preserved
TCLP/SPLP Leachate	2 VOA vials, approximately 42 mL total volume when capped with no headspace (no headspace is considered less than 5-6 mm bubble present in container	The leachate solution provides a pH generally <2 so no additional chemical preservative is added	<6°C, but above freezing	Must be analyzed within 14 days of the end of the leaching process to analysis

7.2. Acceptable sample collection options for low level 5035 soil samples are listed below:

7.2.1. 40mL tared vial preserved with Sodium Bisulfate. Note- Sodium Bisulfate preserved vials are not recommended or generally supplied by Pace. The reason why sodium bisulfate is not recommended is due to the possibility of ketones contamination/formation. Depending on the soil matrix, the sodium bisulfate can react to the soil to form ketones. Therefore if the client chooses to use sodium bisulfate as the preservative there may be ketones detections. Also sodium bisulfate is destructive to the autosamplers and concentrators causing additional maintenance and down time.

7.2.2. 5g capacity Encore or similar approved sample collection and storage device (i.e. Terracore).

7.2.3. Method criteria states that the sample weight should be 5 ± 0.5g, but due to field sampling the weights may vary. Pace will qualify samples that are received with greater than 7.5 grams of sample. There may also be times due to matrix, such as an ash, that weights lower than 5 grams result in the lab not being able to perform an adequate purge. Pace will notify the clients of the matrix difficulties, analyze and qualify accordingly.

7.3. Samples collected in Encore or similar devices must be extruded within 48 hours of collection. Alternatively, samples may be frozen. The extrusion time and date must be recorded in the extrusion logbook.

7.3.1. For low-level soil – extrude the sample into a tared 40mL vial containing 5mL of organic free reagent water and a clean stir bar. Record the weight in the extrusion logbook. Record the date and time of extrusion in the extrusion logbook. Cap the vial and freeze at -10 to -20oC until analysis.

7.3.2. For medium-level soils – extrude the sample into a tared 40mL vial. Record the weight in the extrusion logbook. Record the date and time of extrusion in the extrusion logbook. Add the appropriate ratio of methanol to the weight of the soil (e.g. 10mL methanol to 10g of sample) and cap.

7.3.3. If a client is collecting and freezing in the field, Pace assumes that the samples are frozen within the 48 hour requirement if they are received frozen if no associated paperwork is received indicating otherwise. The laboratory will not qualify the data if the samples are received frozen.\

7.3.3.1. If the samples were anticipated to be received frozen, and are received in a manner indicating that the samples were not completely frozen or thawed on transport, sample receiving will mark the containers received thawed with an "X" on the cap to alert the laboratory.

7.3.3.2. If there is volume available that was received frozen, that container should be used for analysis. If there is no frozen volume available and the lab must used a "X" container for analysis, the lab must qualify the data as being performed by a container that had not been completely frozen.

7.4. Multiple states allow for packed jars for soil analysis. For samples that are received in a pack jar, there may be client specific requirements that direct the subsampling and preserving within 48 hours as directed by Method 5035.

7.4.1. If the samples were not preserved within 48 hours, document the information on the associated preparation logbook. Qualify the data according to client specific instructions as necessary.

7.4.2. A packed jar will be considered to contain headspace if the soil falls below the threads of the vial. If there is headspace, the data will be qualified to indicate that analysis was conducted on a sample that contained headspace.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies.

Supply	Description	Vendor/ Item # / Description
Autosampler	Varian Archon 5100 and EST Archon 8100, Centurion w/s (or equivalent), or Atomx	Varian, Tekmar or Centurion
Sample Concentrator	EST Encon Evolution(EV) Concentrator, Tekmar Atomx, Tekmar (Lab Sample Concentrator) LSC 3100, LSC 3000, OI analytical Eclipse or equivalent	Encon, OI or Tekmar
Purging Chamber	The purging chamber is designed to accept 5mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap should be minimized. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3mm at the origin. The purge gas must be introduced no more than 5mm from the base of the water column	Encon,Tekmar, OI analytical or equivalent replacement
Traps	Trap Packing - A variety of traps are available from manufacturers. Any of these traps may be used if the trap packing materials do not introduce contaminants into the analysis and the data generated using the trap meets the initial and continuing calibration technical acceptance criteria of this method. Some traps used include, but are not limited to a tenax/silica gel/carbon trap, tenax/silica gel/carbon/OV-1 trap, and a Vocarb 3000 trap.	E7300-K03, EVO K trap E07300-L03, EVO MoRT trap Supelco 24920-u, Tekmar K traps for 3000/3100 Supelco 24910-U, Tekmar A traps for 3000/3100 14-9908-403,#9 (U-shaped) 14-9908-003, #9 (Straight Trap) straight trap H

Desorber	EST EV, Tekmar LSC-3100, LSC 3000 or equivalent should be capable of rapidly heating the trap to the manufacturer's recommended temperature for desorption, typically 180°C to 260°C, depending on the trap chosen	Encon, OI, Tekmar, or equivalent replacement
Sample Heater	Should be capable of maintaining the purging chamber at 40°	Varian, Tekmar, Centurion, OI or equivalent replacement
Gas Chromatograph	An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection (Hewlett Packard HP 6890 or equivalent)	Agilent/Hewlett Packard, or equivalent replacement
Column	20 m x 0.18 mm ID x 1.0 µm film thickness capillary column	RestekVMS-Rtx, or equivalent replacement
Mass Spectrometer	Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria in Table III when 5-50 ng of the GC/MS tuning standard (BFB) are directly injected onto the column. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC (HP5973MSD or equivalent)	Agilent/Hewlett Packard, or equivalent replacement
Transfer Line	GC/MS interface - The GC is interfaced to the MS with an all glass enrichment device and an all glass transfer line. Any GC-to-MS interface that gives acceptable calibration points at 50ng or less per injection for each of the analytes and achieves all acceptable performance criteria may be used. If a 0.18-0.32 mm ID capillary column is used, it is positioned directly into the ion source and this GC/MS interface acts only as a heated connection, not as an enrichment device	Agilent/Hewlett Packard, or equivalent replacement
Shaker Table	A shaker table is used to mix samples thoroughly prior to being analyzed on the instrument.	Precision Scientific 360P Orbital Shaker. CAT#67127, or equivalent replacement
Data System	A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.	Hewlett-Packard Chemserver
Data Processing Software		Target 4.1, or equivalent replacement

Data Package and Review Software	Compiles pdf images of all the data to be available for package generation and secondary review	Gandalf, see master software list for revision
Data Reporting Software	Laboratory information management system (LIMS)	Horizon, see master software list for revision
Microsyringes	10 , 25, 50, 100, 250, 500, and 1000µL	Hamilton, or equivalent replacement
Syringes	5, 10, 25mL, 50 mL or gas-tight with shutoff valve	Hamilton, or equivalent replacement
Eppendorf Pipettor	1000µL	Eppendorf, or equivalent replacement
Balance	Analytical, 0.0001g, and top-loading, 0.1g	
2mL Screw Top Vials	Clear glass vials, 2mL with Teflon-lined screw cap	Fisher Scientific C40131500 or equivalent replacement
Disposable pipettes	Glass pasteur pipettes	Fisher Scientific 13-678-31J or equivalent replacement
Volumetric Flasks	Class A - 5mL, 10mL, 25mL, 50mL, and 100mL, 200mL, 250mL, and 1000mL with ground-glass stoppers	Fisher Scientific or equivalent replacement
Spatula	Stainless steel	Fisher Scientific equivalent replacement
VOA vials	40 mL VOA Vials actual volume without headspace = 42 mL	C&G Unpreserved Vials NC9879693 or equivalent replacement

10. Reagents and Standards

10.1 Table 10.1 Reagents and Standards.

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Organic-free Water (DI)	De-ionized water, may be boiled and/or purged to further remove volatile contaminants	Verify that background levels of volatile compounds are acceptable by analysis
Methanol (MeOH)	CH_3OH - Fisher Purge and Trap grade or equivalent, demonstrated to be free of analytes. Store apart from other solvents	Fisher Scientific A453-1 or equivalent replacement
Stock Standard	Stock solutions are typically purchased as certified solutions. Multiple stock standards can be combined (diluted) to yield one working standard. 1000-40,000mg/L	O_2Si - 121369-02-02 (8260 Gases) O_2Si - 020986-02-02 (Freon 21) O_2Si - 121370-02-02 (Custom Mix 98-1370) O_2Si - 125875-05 (Reactive 5-81) or equivalent
Surrogate Standard	10,000mg/L	O_2Si -120330-03-P (8260 IS/SS Soln)
Initial Calibration Verification Stock Standard	1000-40,000mg/L	O_2Si - 121369-02-02-SS (8260 Gases SS) O_2Si - 020986-02-02-SS (Freon 21 SS) O_2Si - 121370-02-02-SS (Custom Mix 98-1370SS) O_2Si - 125875-05-SS (Reactive 45-81SS) or equivalent
Internal Stock Standard	10,000mg/L; 10,000-100,000mg/L	O_2Si -120330-03-P (8260 IS/SS Soln); Chem Service Inc. - SP-89304710CSZ (1,4-dioxane d8 and acetone d6)
Tuning Standard	10,000mg/L	O_2Si -120330-03-P (8260 IS/SS Soln)
Anti-Foaming Agent	Dow Corning 1520-US Antifoam (or equivalent replacement). Add 2g to 42 mL of DI water and mix vigorously. The solution must be gently shaken solution	Dow Corning 1520-US

	prior to use. If anti-foam is used for samples, injection 100-200uL of anti-foam solution into the vial	

10.2 Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Diluent Diluent Volume	Final Total Final Volume	Final Concentration
Intermediate Tune Solution	500 µL	Methanol	100 mL	50 µg/mL	Intermediate Tune Solution
Tune	5 µL	Water	5mL	50 µg/L	Tune
Calibration Working Standard	0.5mL of 1000mg/L-40,000mg/L	Methanol	9.5mL methanol	10mL	50-2000 µg/mL(nominal conc 100ug.mL)
Calibration Std 1	0.5 µL	Methanol	249.999 water	250 mL	0.2 µg/L
Calibration Std 2	1.0 µL	Methanol	249.9995 water	250 mL	0.4 µg/L
Calibration Std 3	1.0 µL	Methanol	99.999 water	100 mL	1 µg/L
Calibration Std 4	4.0 µL	Methanol	99.996 water	100 mL	4 µg/L
Calibration Std 5	10.0 µL	Methanol	99.99 water	100 mL	10 µg/L
Calibration Std 6	20.0 µL	Methanol	99.98 water	100 mL	20 µg/L
Calibration Std 7	50.0 µL	Methanol	99.95 water	100 mL	50 µg/L
Calibration Std 8	100.0 µL	Methanol	99.90 water	100 mL	100 µg/L
Calibration Std 9	250.0 µL	Methanol	99.75 water	100 mL	250 µg/L
Surrogate Working Standard for Archon	0.5mL of 10,000mg/L	Methanol	19.5mL methanol	20mL	250 µg/mL
Surrogate Working Standard for Centurion	0.5mL of 10,000mg/L	Methanol	99.5mLmethanol	100mL	50 µg/mL
Internal Standard Working Standard for Archon	0.5mL of 10,000mg/L; 0.5mL of 10,000 to 100,000mg/L	Methanol	19.5mL methanol	20mL	250 µg/mL (1,4-dioxane-d8 is at 5000 µg/mL)
Internal Standard Working Standard for Centurion	0.5mL of 10,000mg/L; 0.5mL of 10,000 to 100,000mg/L	Methanol	99.5mLmethanol	100mL	50 µg/mL (1,4-dioxane-d8 is at 1000 µg/mL)
Continuing Calibration Verification Standard at 50ppb	100uL of working std	Water	199.9mL of water	200mL	50ug/mL
Continuing Calibration Verification Standard at 20ppb	50uL of working std	Water	249.95mL of water	250mL	20ug/mL

10.2.1 Calibration standards for SIM/SCAN simultaneous acquisition are prepared by diluting calibration std 5 (10ug/L final concentration-see above) with an appropriate volume of organic free DI water. Dilutions are performed in 100mL volumetric flasks and diluted to a final concentration of 0.005ug/L, 0.01ug/L, 0.05ug/L, and 0.1ug/L. For SIM/SCAN analysis the calibration curve is named as calibration std 1 through calibration std 13 beginning with the lowest concentration.

10.3 All standards, blanks, spikes, and samples must be analyzed using the same conditions. A set of at least five calibration standards containing the method analytes and surrogates are needed (six standards are necessary for quadratic curve fits). One calibration standard should contain each analyte at a concentration at or below the reporting limit for that compound; the other calibration standards should contain analytes at concentration that define the range of the method. To prepare a calibration standard, add an appropriate volume of standard solution to organic-free reagent water in a volumetric flask. Using a microsyringe, rapidly inject the standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times. Transfer the standard to a 40 mL VOA vial and load into the Autosampler. If ICAL or CCVs are not used immediately they must be stored at 0 to 6 degrees Celsius in a cooler which does not house samples for up to one day from the day they were made.

10.4 The following is an example of standard preparation for an initial calibration. Standard preparation is determined by client and project requirements. Unless a reporting limit of 0.2µg/L or lower is required, the low standard will typically be prepared at 0.4µg/L. A "soil" curve to reflect the chromatography conditions of medium level soils (1:50 ratio of MeOH:water) is prepared by adding 2 mL of methanol into the calibration standards and reducing the volume of reagent water accordingly. Additional levels may be performed.

10.5 For the centurions autosamplers, a 5 µL aliquot of the intermediate tune solution is added to the sample loop that is transferred to the sparge tube, at this point it is a 5 mL solution at a concentration of 50 µg/L on column. The method criteria of 5-50 ng is met as the split ratio is set at 30:1 to 50:1 depending on instrument resulting in >5 ng at the mass spectrometer.

11. Calibration and Standardization

11.1. Table 11.1 Calibration and Standardization.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Tune (BFB)	Every 12 hours	See below in 11.2	If the tune criteria is not met, evaluate the standard preparation. Perform any vendor recommended maintenance including hardware tune using perfluorotirbutylamine (PFTBA). The tune must pass prior to any analysis being conducted.
Calibration Curve Fit	Average Response Factor Linear Regression Non-linear Regression(quadratic)	% RSD: 30% for CCCs, 15% for all others $r \geq 0.995$ $COD \geq 0.99$ *See 11.3 and 11.4 for CCCs and SPCCs*	If not met, try non-linear regression fit. If still not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	after each initial calibration	CCV criteria + 10% unless otherwise specified in a QAPP	If the requirements for ICV are not met, verify the standard preparation and if there are any apparent issues with the initial analysis. Reanalyze one more time. Only two injections of the same standard are permitted back to back prior to recalibrating the instrument. May be used as a CCV if CCV requirements are met.

			Secondary source standard is also here-in referred to as non-calibration source standard to note that although the primary and secondary source standards are comparable in concentration and used interchangeably, The calibration is prepared from a separate source than is used for ICV, CCV, and QC spiked samples. (i.e. A calibration curve may be prepped with secondary source so long as the affiliated ICV,CCV, LCS,LCSD, MS, and MSD are spiked using the primary (non-calibration) source.)
Reporting Limit Verification	Initially evaluated from the Ical levels and every 30 days after or until next Ical is necessary	The reporting limit must be ± 40% of the true value for MN originating samples	Requantitate the reporting limit standard in the Ical once the linear regression has been established to verify the recoveries. If the criteria is not met, review for any calculation errors. Evaluate for recalibration. Depending on data quality objectives, the reporting limit may be raised to the next passing Ical level.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and be verified every 12 hours following the tune Prepared using non-calibration source standard	% Diff ±20% for CCCs, and ±40% for non-CCCs If a CCV is evaluated using a sublist that does not include all the CCCs, CCC criteria applies to all the compounds in the list. Client, QAPP, or state requirements may supersede this requirement.	If the requirements for CCV are not met, verify the standard preparation and if there are any apparent issues with the initial analysis. Reanalyze one more time. Only two injections of the same standard are permitted back to back prior to recalibrating the instrument.
Internal Standards	All analytical runs	Retention time must be ±30 seconds of any internal standard from the ccv; the response factor must be within -50% to 200%. Client, QAPP, or state requirements may supersede this requirement.	The chromatographic system must be inspected for malfunctions and corrections must be made.

11.2. Each GC/MS system must be hardware-tuned using perfluorotributylamine (PFTBA) and must also meet the criteria below for a 5-50ng injection of 4-bromofluorobenzene. Analyses must not begin until these criteria are met.

BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
95	Base Peak, 100% relative abundance
50	15.00 - 40.00% of m/z 95
75	30.00 - 60.00% of m/z 95
96	5.00 - 9.00% of m/z 95
173	Less than 2.00% of m/z 174
174	50.00-120% of m/z 95
175	5.00 - 9.00% of m/z 174
176	95.00 to 101.00% of m/z 174
177	5.00 - 9.00% of m/z 176

Note: All ion abundance must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent.

The mass spectrum of BFB should be acquired in the following manner. Three scan (the peak apex and the scans immediately preceding and following the apex) and acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Part of the BFB peak must not be background subtracted

11.3. CCCs include the list below:

1,1-Dichloroethene	1,2-Dichloropropane
Chloroform	Toluene
Ethylbenzene	Vinyl Chloride
NOTE: Additional CCCs may be specified on a client and project-specific basis. If a sublist is analyzed all analytes are treated as CCCs	

11.4. System Performance Check Compounds (SPCCs) are checked for a minimum average response factor. The SPCCs and the minimum response is listed below:

Compound	Minimum Response
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachlorethane	0.30

11.5. For water and low level soil analysis, the CCV and LCS/LCSD solutions are the same solution (prepared at the same, using the same standards, etc.). The CCV and LCS/LCSD analyses are interchangeable and can be used for both sample types (the CCV can also be used as the LCS and the LCS can also be used as the CCV) provided that 2 CCV's didn't already fail in a row (which would trigger an initial calibration). When these analytical runs are used as the same file, they should be named as 2 separate files to distinguish the sample types for reporting the appropriate reports and so that there is a unique file name associated with each sample type.

11.6. For medium level soils, LCS/LCSD's are not interchangeable with the CCV's in the run sequences as the LCS/LCSD's are prepared and extracted with the associated sample on the day of preparation while the CCV's and initial calibration solutions are prepared by using a ratio of 1 mL methanol into 50 mL of DI water (to matrix match the calibrations to the sample matrix) and are not extracted.

11.7. INITIAL CALIBRATION VERFICATION (ICV)

11.7.1. To ensure internal standard recoveries from samples that run after the Ical under the same folder go off the Ical and not the ICV, process the ICV as a sample. To see if the ICV meets

passing requirements pull the ICV file into a batch.b along with the method and requant the ICV as a continuing calibration.

11.8. CONTINUING CALIBRATION VERIFICATION (CCV)

11.8.1. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition.

11.8.2. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration, the analytical system must be inspected for malfunctions and corrections must be made.

11.8.3. If the EICP area for any of the internal standards changes by a factor of two, (-50% to +100%) from the last daily calibration standard check, the system must be inspected for malfunctions and corrections must be made.

12. Procedure

12.1 Water/Leachate Sample Analysis.

12.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Screening can be accomplished by using a headspace GC PID or by analyzing the sample at a dilution by GC/MS. When available, historical data may be used to perform dilutions prior to analysis.

12.1.2 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples (see 11.2)

12.1.2.1 The BFB and calibration verification standard may be combined into a single analysis as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

12.1.3 Sample vials are loaded onto the autosampler. All leachate samples are analyzed at 1:25 based on action limits.

12.1.3.1 If there is more than one ZHE batch they may be combined, however, all ZHE blanks must be tested and proven to be free of contamination. If contamination is found in the ZHE blank, the other vial must be analyzed to confirm contamination. The affected samples must be footnoted appropriately. Only one is reported if both are proven free of contamination.

12.1.3.2 All leachate samples (including the Blank, LCS, MS/MSD, and sample duplicate (DUP) are diluted 1:25 based on action limits. The leachate is diluted by adding 2000 µL of the leachate extract to a 50 mL volumetric flask containing DI water. Dilute to a final volume of 1:25 using DI water. Fill a 40 mL unpreserved VOA vial with the prepared sample for analysis. If foaming is observed during dilution of the extract, 200 µL of anti-foaming agent (see 9.8) may be added to the 50mL volumetric flask to prevent foaming of the concentrator.

12.1.4 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample vial is sealed.

12.1.4.1 Dilutions may be made in volumetric flasks of various sizes. Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions. Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask. See Attachments V and VI.

12.1.4.2 Inject the proper aliquot of sample into the flask. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert three times. For samples requiring pH determination; once sample dilution is completed, the pH of the un-diluted sample must be taken with pH paper. If the pH is greater than 2 the sample must be footnoted to the nearest whole number. Repeat above procedure for additional dilutions.

12.1.4.3 Fill the vial with diluted sample and load onto the autosampler.

12.1.4.4 The autosampler adds the internal standard spiking solution and the surrogate spiking solution to the 5mL sample aliquot. The amount added by the autosampler should be equivalent to the concentration of 50 µg/L of each surrogate, 50 µg/L for the internal standards 1,4 dioxane d8 is at a concentration of 2000 µg/L and acetone d6 is at concentration of 100 µg/L). The archon accomplishes this by adding the internal standard and surrogate solution utilizing a 1 µL loop, the centurion w/s adds 5 µL of the solution. Analyze the samples using the same autosampler and GC conditions used to pass BFB, standard, and blank criteria.

12.1.5 If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. Alternately, samples loaded on an autosampler can be accepted after a subsequent sample is shown to be free of carry-over contamination or if the detection is 10x greater than the carryover detection. Carryover in p&t systems can vary for instrument to instrument depending on the condition of the equipment. Analysts review the carryover after the upper level of the initial calibrations and after the ICV, in addition they monitor the carryover daily on the system blanks ran which is generally ran after QC samples.

12.1.5.1 It is routine for the laboratory to run multiple blanks after an initial calibration to monitor the carryover and ensure the ICV does not have carryover affecting the % recoveries. Daily, it is common for the laboratory to run system blank before the method blank. The system blank analyzed serves as a carry over assessment. It helps us determine how our system is performing from sample to sample and gives us a measure of how well our procedures for clean up are functioning on our instrumentation including rinses, baking, etc. As a secondary assessment, this allows us to determine if low level detections on our system are due to system contamination or from carryover which has become increasingly more important as we have a significant amount of clients that request evaluation and reporting to the statistical MDL levels. The second blank is used as the method blank but we feel that there is a benefit to us and the client in the analysis of 2 blanks for better assessment of system performance and data evaluation. All samples must be thoroughly reviewed when sample concentrations exceed 50µg/L to ensure low-level carryover is not occurring into subsequent analyses. Samples that need to be evaluated to the MDL are not re-analyzed if there are j-flagged detections in the method blank or the detections from possible sample carryover.

12.1.6 For matrix spike analysis, add 8.4 µL or 21 µL of a 100 µg /mL non-calibration source standard to the aqueous sample vial (42 mL actual volume). This is equivalent to a concentration of 20 µg/L or 50 µg/L, respectively, of each non-calibration source standard. Add the spiking solution through the septa of the vial as the vial should not be opened to maintain sample integrity.

12.1.7 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper calibration range for compounds which were previously over range.

12.1.8 Once sample analysis is completed, the pH of the sample must be taken using pH paper and recorded in the instrument run logbook. If the pH is greater than 2 and the holding time is past 7 days, the sample will be footnoted accordingly. The report number must be documented in the instrument run log and the sample data footnoted.

12.2 Sediment/Soil and Waste Samples

12.2.1 It is recommended that all samples of this type be screened prior to analysis. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and trap system, and require extensive cleanup and instrument downtime. Use the screening data to determine whether to use the low-concentration method (0.004-0.2 mg/kg) or the mid-concentration method (>0.2 mg/kg). For both low level soils (LLS) and medium level soils (MLS) the pace label is placed on all containers during the log-in process which is performed by sample receiving. The weight of the label is subtracted from all soil samples to reflect the weight of the soil weight. The weight of the label is determined annually (unless specified) by weighing out 10 labels and determining an average weight. This subtracting of the label weight is performed in the soil prep logbook.

12.2.2 Low-Level Soils (LLS) - This is designed for samples containing individual purgeable compounds of <0.2 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. Analyze all blanks and standards under the same conditions as the samples.

12.2.2.1 A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed by low level soil method. Follow the initial and daily calibration instructions, except the ICAL, CCVs, LCS/LCSD, Blanks are prepared by adding 5 mL of the solution into 40 mL unpreserved vial containing 5±0.1 g of sand and a stir bar purged at a temperature of 45-50°C.

12.2.2.2 The majority of the samples received are sampled in Terracore kits (5035 closed system); therefore, the exact weight of the soil sample must be determined. Weigh the vial and record to the nearest 0.01 grams. Subtract the vial weight prior to sampling and record the final weight. The final weight is entered into Horizon to reflect to the soil weight. Refer to 7.2.3.4 for weight acceptance policy. The soil volume is critical to the purging of the sample. If there is too much soil in the vial, approximately more then ½" high in the vial, the autosampler purging needle can't purge the sample. The sample will be rejected for low level soil analysis and medium level analysis will occur instead. If there is soil in the threads of the capped vial, the sample will not seal properly and purging efficiency will be reduced which is generally indicated by monitoring the internal standards and can result in unreportable LLS analysis.

12.2.2.3 When a sample arrives and low level analysis is required (non- 5035), a 5 g aliquot of the sample is weighed directly into a tared unpreserved 40 mL vial. Note the weight to the nearest 0.01 g; add 5 mL of organic free DI water, and a stir bar.

12.2.2.4 The method blank is prepared by adding 5 mL of organic free DI water with 5+/-0.1g of sand and a stir bar. The LCS/LCSD is prepared by adding 5 mL of a mid-level calibration CCV solution to 5+/-0.1g of sand and a stir bar. The weight of the BLK, LCS, and LCSD are recorded as 5 grams so long as the actual weight is 5+/-0.1grams.

12.2.2.5 For matrix spike analysis, add 2.5 µL of the 100 µg/mL non-calibration source standard through the septa of the vial as the vial should not be opened to maintain sample integrity. This is the equivalent to a nominal concentration of 50 µg/kg for the majority of the analytes. Alternatively, if the MS/MSD is being prepared from a packed jar or encore, the MS/MSD may be prepared by adding 5 mL of a mid- level calibration CCV solution instead of spiking 2.5 µL of the 100 µg/mL non-calibration source standard through the septa of the vial. Which method is chosen it must be documented on the prep logbook.

12.2.2.6 Samples and QC are placed on a shaker table for 2 minutes to ensure samples are thoroughly mixed prior to purging.

12.2.2.7 The QC and samples are loaded onto the autosampler. The autosampler adds the internal standard spiking solution and the surrogate spiking solution to the soil sample with the addition of 10 mLs of DI water. The amount added by the autosampler should be equivalent to the concentration of 100µg/kg of each surrogate, 100µg/kg for the internal standards, and low level soils have 4,000 µg/kg for the 1,4-Dioxane d8 internal standard 200 µg/kg for acetone d6 (wet weight). The autosampler heats the sample at 45°C throughout the purge cycle and stirs the magnetic stir bar.

12.2.2.8 If saturated peaks occurred the sample is either E-flagged or the medium level soil is prepared and analyzed for the analytes which exceeded the calibration range. If a non-closed system (5035) sample is provided, a smaller aliquot, 1 g or larger, may be used to dilute the analytes that exceeded the calibration range.

12.2.3 Medium Level Soil (MLS) - A sample is either extracted or diluted with methanol, depending on its solubility. An aliquot of the extract is added to organic-free reagent water. The autosampler adds the internal standard and surrogate spiking solution to the 5mL sample aliquot. The amount added by the autosampler should be equivalent to the concentration of 50 µg/L of each internal and surrogate standard. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/kg should be analyzed by this method.

NOTE: The following steps must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

12.2.4 To prepare the laboratory method blank (BLK), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) weigh out 10+/-0.1 grams of Ottawa sand and add 10mL of methanol to a 40 mL VOA vial (42 mL actual volume). The weight of the BLK, LCS, LCSD are recorded as 10 grams so long as the actual weight is 10+/-0.1grams. They should be uniquely labeled by QC batch numbers to ensure they are analyzed with the correct batch of samples. The LCS/LCSD are spiked with the 100 µL of 100ug/mL of the non-calibration source standard to achieve a final concentration of 1000 µg/kg after the 1:50 dilution.

12.2.5 To prepare the matrix spike (MS) and matrix spike duplicate (MSD), weigh the vial and record the weight to the nearest 0.01g. Subtract the vial weight prior to sampling and record the final weight. If the weight is greater than the expected 5g, 10g, or 25g weight, the addition of methanol is necessary in order to maintain the 1:1 sample to solvent ratio. Add the appropriate amount of the 100 µg/mL of the non-calibration source standard to achieve a final concentration of 1000 µg/kg after the 1:50 dilution. If insufficient sample volume was received to prepare the MS/MSD, the project must be footnoted.

12.2.6 To prepare samples that arrive preserved in methanol, weigh the vial and record to the nearest 0.01g. Subtract the vial weight prior to sampling and record the final weight. If

the weight is greater than the expected 5g, 10g, or 25g weight, the addition of methanol is necessary in order to maintain the 1:1 sample to solvent ratio. If the weight is less than expected 5g, 10g or 25g weight, record the difference.

12.2.7 To prepare samples that are not preserved in methanol, the sample consists of entire contents of sample container. Using a top-loading balance, weigh 10 grams (wet weight) of the sample into a tared 40 mL vial. Record the weight to 0.01g. Samples not field preserved should be preserved within 48 hours of collection. Client, QAPP, or state requirements may supersede this requirement.

12.2.8 Oily, solid waste or product samples are generally not field preserved due to the unknown solubility. If the sample is not soluble in water, a waste dilution will be performed by weighing out 1gram of the sample into a tared 40 mL VOA vial. Record the weight to 0.01g. Dilute with the addition of 10 mL of P&T grade methanol. Note the prep method as a waste dilution on the prep log.

12.2.9 The pace label that is applied in sample receiving is subtracted from the soil weight to eliminate the label weight from biasing the soil weight. The label weight was determined to be 0.18 grams by averaging 10 labels and all samples have this subtraction done in our prep sheets. If additional labels are applied the lab is unable to determine the weight of those labels and therefore unable to determine the potential bias in the weight/reporting limits.

12.2.10 The LCS/LCSD, MS/MSD, BLK, and all associated samples within the batch must be shaken for two minutes, and then sonicated for 20 minutes. After sonicating, prepare the samples by adding 1000 µL either by syringe or eppendorf pipetter of the methanol extract to a 50 mL volumetric flask containing DI water. Dilute to a final volume of 1:50 using DI water. Fill a 40 mL VOA vial with the prepared sample for analysis.

12.2.11 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample vial is sealed.

12.2.11.1 Dilutions may be made in volumetric flasks of various sizes. Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

12.2.11.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask. See attachment III for common dilution factors.

12.2.11.3 Inject the proper aliquot of sample extract into the flask and the proper amount of P&T methanol, so that the same amount of methanol is added to all samples and QC. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert three times.

12.2.12 The extracts must be stored above freezing but <6°C.

12.3 Data Interpretation

12.3.1 Qualitative Analysis: An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the user's GC/MS. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component, and (2) correspondence of the sample component and the standard component mass spectrum.

12.3.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

12.3.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of ions specified in (1) must agree within ± 30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.

12.3.2 For samples containing components not associated with the calibration standards, a library search using the most recent NIST/EPA Library may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

12.3.2.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.3.2.2 The relative intensities of the major ions should agree within ± 20%.

12.3.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.3.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.3.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.2.6 Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

13. Quality Control

13.1. Table 13.1 Quality Control.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per batch of 20 samples or less	Target analytes must be less than 1/2 reporting limit. If results are reported to MDL, target analytes in MB should be non-detect to ½ PRL. The lab is not able to routinely achieve MB less than the MDLs due to common p&t	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and the sample cannot be reanalyzed, report sample with appropriate qualifier. Client must be alerted and

			carryover and common lab contamination.	authorize this condition. If there is insufficient sample volume B flag the detections present in the samples associated with the contaminated blank. For WI samples, evaluate the MB to the MDL. If detections are present between the MDL and RL, qualify appropriately. For detections above the RL, data is acceptable to report only if sample concentrations are 10x greater, otherwise re-prep and re-analyze.
Laboratory Control Sample (LCS)	DI water spiked with all target compounds for waters and Low level soils. Medium level soils are extracted in 10mLs of methanol. LCS/LCSDs prepared using non-calibration source standard	One per batch of 20 samples. A LCSD is not required per method requirement and generally only performed if insufficient volume for MS/MSD or MS/DUP.	Internally generated limits updated annually. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of the control limits. This does not necessarily mean the system is out of control, and therefore no corrective action would be required beyond footnoting the data. Client, QAPP, or state requirements may supersede this requirement. See Quality Manual section 4.2 for more information.	The number of allowable exceedance is as follows: ->90 analytes in LCS- 5 outliers -71-90 analytes in LCS- 4 outliers -51-70 analytes in LCS- 3 analytes -31-50 analytes in LCS- 2 outliers 11-30 analytes in LCS- 1 outlier -<11 analytes in LCS- no outliers allowed Evaluate the LCS to determine the cause of the outliers; verify calculations and standard preparation. Perform any necessary system maintenance prior to reanalyzing the LCS. For waters, this is the same as the CCV solution so the associated samples will have to be reanalyzed accordingly. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds Prepared using non-calibration source standard	One per batch of 20 samples or less	Internally generated limits updated annually	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Determine that there was no system error causing the outlier, reanalyze if necessary per client or regulatory QAPP.
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup Prepared using non-calibration source	One per batch of 20 samples or less MS/MSD is the method requirement if	%Diff ≤ 30%	Report results with an appropriate footnote. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the

	standard	sufficient sample volume is available. If insufficient vials are available perform a MS of one sample and a Dup of another in the batch. For MPCA/Admin Contract clients, if there is insufficient volume for the MS/MSD, perform and MS/LCSD		original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately. For Minnesota Admin Contract clients, if there is insufficient volume for MS/MSD, qualify the batch for the insufficient volume even if a MS/DUP is performed as the MS/MSD are required.
Surrogate	1,2 Dichloroethane d4 Toluene d8 4-Bromofluorobenzene (BFB)	In every analytical run. Surrogates are spiked into the sample utilizing the autosampler using a single point calibration for waters, low level soils and medium level soils.	Internally generated limits updated annually	Check to make sure there are no calculation errors, surrogate solutions or internal standard errors. Recalculate accordingly if found. Check instrument performance, if it is correct and reanalyze accordingly. Reanalyze to confirm outlier. If confirmed, report the initial analysis and indicate confirmed by second analysis. If it doesn't confirm, report the second analysis if within holding time. If the methanol preserved soil is methanol corrected see section 14.9 for outliers details. ***Exception:*** if the surrogates are out biased high and the sample is non-detect, report the data with the surrogates qualified accordingly or if visual matrix affecting the surrogate recovery data may be reported with footnotes.

14. Data Analysis and Calculations

14.1. The following calculation can be used to calculate the LCS or MS percent recovery (where SampleConc would be equal to 0 for the LCS and MSConc would be the LCS concentration):

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

Where:
MSConc = MS Concentration
SampleConc = Sample Concentration of the MS parent sample

14.2. %REC = % Recovery The RF is calculated as follows:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

14.3. The percent relative standard deviation (%RSD) for CCCs:

$$\%RSD = \frac{SD}{\overline{X}} x100$$

Where: **RSD** = Relative standard deviation.
SD = Standard deviation of average RFs for a compound
$\overline{X}$ = Mean of initial RFs for a compound

14.4. Standard deviation is calculated as below:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_1 - \overline{RF}\right)^2}{n-1}}$$

Where: $\mathbf{RF_1}$ = Each individual response factor
$\overline{\mathrm{RF}}$ = Mean of the Response Factor
n = The total number of values

14.5. Percent Difference (%D) for daily CCV evaluation:

$$\%Difference = \frac{RF_1 - RF_c}{RF_1} x100$$

Where: $\mathbf{RF_1}$ = Average response factor from initial calibration.
$\mathbf{RF_c}$ = Response factor from current verification check standard

14.6. Quantitative Analysis: When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used should be the one nearest the retention time of that of a given analyte or as specified in the method.

Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste:

$$\text{Concentration}(\mu g/L) = \frac{(A_x)(I_s)(DF)}{(A_{is})(RRF)}$$

Where:

$\mathbf{A_x}$ = Area of characteristic ion for compound being measured.

$\mathbf{I_s}$ = Amount of internal standard injected (µg/L).

$\mathbf{A_{is}}$ = Area of characteristic ion for the internal standard.

RRF = Average Relative Response factor for compound being measured.

DF = Dilution Factor

Sediment/Soil, Sludge, and Waste:

$$High\ \text{Conc.}\ (\mu g/kg) = \left[\frac{(A_x)(I_s)(DF)(V_t)}{(A_{is})(RRF)(W_s)}\right] \bullet 50$$

$$Low\ \text{Conc.}\ (\mu g/kg) = \frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)}$$

Where:

A_x, I_s, A_{is}, RRF = Same as in water and water-miscible waste above
V_t = Volume of total extract (mL)
V_i = Volume of extract added (mL) for purging
W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.
S_v = Volume of diluted extract
DF = Dilution factor

Note: All methanol extracts are diluted 1:50

14.7. Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The percent dry weight of the sample should be reported along with the data in either instance.

14.8. When applicable, an estimate of concentration for non-calibrated components in the sample can be made. The formula given above should be used with the following modifications: The areas Ax and Ais should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentrations. Use the nearest internal standard free of interferences.

14.9. Methanol Correction: This will only be performed if specifically requested by a regulatory agency or by the client. When methanol correction is requested, the volume needs to be adjusted for the amount of soil moisture present in the solid sample. This is done through Horizon once the % moisture analysis has been performed. The analyst enters the sample weight and volume into the prep batch in Horizon. The condition code needs to be changed to "mc" for methanol corrected instead of the standard "ok". Once this is done, Horizon will perform the calculation. The final volume will change in Horizon to reflect the methanol correction. Note: the lab will spike the surrogate solution (if applicable) based on the known actual volume methanol in the sample, not the methanol corrected volume. Depending on the % moisture in the sample(s) the lab has seen circumstances where the methanol corrected volume is artificially higher than it visually appears. It is not uncommon to have the surrogate recoveries fail once moisture is corrected. The lab will not re-run for surrogate confirmation if non-methanol corrected surrogate recoveries are within control limits.

14.10. Methanol Correction Formula:

14.11. Methanol Corrected Volume = Volume of methanol(mL) + ((%_percent_moisture * sample weight(g))/100))

14.12. The methanol corrected volume value obtained becomes Vt in the calculation referenced in section 14.6.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 & 13.

16.2. SPCC Criteria Failed: If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. SPCCs are used to check compound instability and to check degradation caused by contaminated lines or active sites in the system. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. Chloromethane is most likely compound to be lost if the purge flow is too fast. Bromoform is one of the compounds most likely to be purged very poorly in the purge flow is too slow. Cold spots and/or active sites in the transfer line may adversely affect response. Response of quantiation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z may improve bromoform response. 1,1,2,2 Tetrachloroethane and 1,1 dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

16.3. CCC Failure: If the percent difference for any non-CCC analyte is greater than 40%, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (>20% difference), for any one CCC, corrective action should be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration should be generated. This criterion should be met before quantitative sample analysis begins.

16.4. CCV Failure: If the first CCV fails, the analyst should try to determine the root cause for the failure. Some possible reasons for failures may include but not limited to: bad CCV solution, bad spike of CCV standard, standard mix degradation, internal standard fluctuation/change, analytical system not conditioned, active sites and/or cold sites in the trap or concentrator, contaminated system due to dirty samples, or analytical conditions changed over time. Corrective action for a failed CCV will be case by case depending on the root cause. The analyst's expertise in determining the root cause will help determine the corrective action. Some common corrective actions may include not limited to: making a new CCV solution, running a different vial of a CCV solution, using a different standard to make the CCV solution, making new standards and new CCVs, baking out the concentrator, or replacing transfer lines on concentrator. For volatiles there is generally no major maintenance performed on a daily basis to maintain calibration. If major maintenance is required the system then needs to be recalibrated. If the 2nd ccv fails to meet criteria, a new initial calibration curve must be performed.

16.5. Internal Standard Failures: Internal standard recoveries out low (high bias) – if compounds associated with the internal standard(s) that are outside the control limits are non-detect, the sample can be reported without re-analysis, however, if the outlier is not indicative of a system drift (i.e. If only one sample has internal standard drift, which is dissimilar from other samples around the injection time), re-analysis should be performed to rule out matrix effects. Internal standard recoveries out high (low bias) – re-analysis should be performed assuming there is sufficient sample volume remaining. Appropriate footnoting practices are also observed.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269, Method Detection Limit Studies for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279, Training and Employee Orientation.

18.4. **Periodic proficiency testing (PT)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Results are filed in the Quality office.

19. Method Modifications

19.1. 8260B is a performance based method from SW-846. Some modifications include the use of Nitrogen as purge gas, faster GC ramp parameters reduced purge times, increased purge flow rates, reduced bake times, reduced desorb times, and some systems use a heated purge at 40°C for water,

Below are the typical GC parameters and Concentrator parameters

Typical GC/Concentator paramaters	
Start Temp (°C):	45
Initial Time (min):	2.75
Initial Rate (°C/min):	30/min to 155, hold for 0.5 min
Final Temp (°C):	32/min to 220 hold for 0.5min
Final Time (min):	0.29
Total GC Run Time(min)	~9.5 min
Inlet Temp (°C):	250
Detector Temp (°C):	180
Purge Temp (°C):	ambient-50*
Purge Time (min):	5.0-11.0
Purge Flow (mL/min):	~40-60
Desorb Time (min):	0.5-2.0
Desorb Temp (°C):	250-260
Bake Time (min):	2.0-7.0
Bake Temp (°C):	270
Column Type:	Restek RTX-VMS
Column Dimensions:	20m/0.18mmID/1um df
Column Flow (mL/min):	0.8
Split Flow	30:1 to 60:1
* EVOs that run waters purge temp 30-40C; tekmar 3000/3100/atomx are ambient; MLS are ambient; LLS are 50C via soil cup of autosampler.	

These parameters are for the typical GC/MS system. Some systems may differ depending on the analyte list/matrix/RL needed for the analysis that runs on each different instrument.

19.2. The tuning criteria follow the guidelines indicated in Appendix C: CLP/SOW OLC02.1/Low Concentration Volatile Organic Analysis Method QC criteria, Equations and Definitions.

19.3. The primary ion used for 2-Butanone and 4-Methyl-2-Pentanone are based on the Appendix A: CLP/SOW OLM03.2/Volatile Organic Analysis. In addition, the ion selected for 2-Butanone is due to the fact that 2-Butanone coelutes with 1,1-Dichloropropene. Ion 43 is being utilized as that is not a common ion with the co-eluting compound.

19.4. The lab compares the internal standard to the daily mid-point standard level (CCV) and the CCV is compared to the initial calibration for retention time. This is a modification of the method as written due to client data quality objectives.

19.5. Pace utilizes a single point calibration for surrogates as noted in section 13. This is a deviation of Method 8000B and 8000C based on the guidance in EPA 8260C section 11.3.3. It is demonstrated through acceptable initial calibration, ICV, CCV, sample and batch QC performance.

19.6. The CCV is prepared using non-calibration source standard to allow for LCS/CCV interchangeability, the ICV is also commonly used as CCV/LCS when sample analysis follows calibration.

20. Instrument/Equipment Maintenance

20.1 Please refer to the GC/MS 6890 instrument manual for maintenance procedures performed by the lab.

20.2 All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. The purge and trap concentrator must be leak free in order to ensure properly sample purge efficiency and desorbation. If analyst notices significant decrease in response and suspects a possible leak, one can leak check the concentrator to ensure the p&t concentrator is free for leaks. This can be done through the software or manually by capping the vent valve of the concentrator and purging a blank.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 8260B.

25.5. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 5035.

25.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 5030B.

25.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, Method 8000B.

25.8. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, Method 8260C.

25.9. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, Method 5035A.

25.10. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, Method 5030C.

25.11. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Online, Method 8000C.

25.12. Appendix C: CLP/SOW OLC02.1/Low Concentration Volatile Organic Analysis Method QC criteria, Equations and Definitions.

25.13. Appendix A: CLP/SOW OLM03.2/Volatile Organic Analysis

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Method 8260B Analyte List, PRL, Characteristic Mass (m/z), and associated IS for Restek VMS- Rtx Column.

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27. Revisions

Revision Number	Reason for Change	Date
S-MN-O-521-Rev.35	Added Sections 11.7 and 11.8 and all subsections.	11Jul2018

Attachment I: Method 8260B Analyte List, PRL, Characteristic Mass (m/z), and associated IS for Restek VMS- Rtx Column

Analyte	CAS Number	Low Level Waters (µg/L)	Waters (µg/L)	Low-Level Soils (µg/kg)**	Medium Level Soils (µg/kg)***	Primary Ion	Secondary Ion(s)	Internal Standard used for Quantitation
Propylene	115-07-01	1	1	4	50	41	39,42	1
Dichlorodifluoromethane	75-71-8	1	1	10	50	85	87	1
Chloromethane	74-87-3	4	4	10	200	50	52	1
Vinyl Chloride	75-01-4	0.2	0.4	4	20	62	64	1
1,3- Butadiene	106-99-0	1	1	4	50	54	39	1
Bromomethane	74-83-9	4	4	20	500	94	96	1
Chloroethane	75-00-3	1	1	10	500	64	66	1
Trichlorofluoromethane	75-69-4	0.5	1	10	200	101	103	1
Dichlorofluoromethane	75-43-4	1	1	4	500	67	69	1
Diethyl Ether	60-29-7	4	4	10	200	59	45, 74	1
Ethanol	64-17-5	80	80	400	NA	45	46	4
1,1-Dichloroethene	75-35-4	0.5	1	4	50	96	61, 63	1
Carbon Disulfide*	75-15-0	1	1	4	50	76	78	1
Trichlorotrifluoroethane	76-13-1	1	1	4	50	101	151, 103	1
Iodomethane	74-88-4	4	4	10	200	142	127,141	1
Acrolein	107-2-8	10	10	100	2000	56	55	1
Allyl Chloride	107-05-1	4	4	10	200	41	76,39	1
Acetone d6 (IS#2)	666-52-4	IS	IS	IS	IS	46	64	
Isopropanol (2-Propanol)	67-63-0	100	100	NA	NA	45	43	4
Methylene Chloride*	75-09-2	4	4	20	200	84	86	1
Acetone*	67-64-1	20	20	20	1000	58	43	2
trans-1,2-Dichloroethene	156-60-5	0.5	1	4	50	96	61,98	1
Methyl Acetate	79-20-9	5	5	Na	Na	74	43	
Hexane (n-Hexane)*	110-54-3	10	10	500	500	86	57,56	2
Methyl-tert-butyl Ether	1634-04-4	0.5	1	4	50	87	57	1
Tert Butyl Alcohol (2-Methyl-2-propanol) (TBA)	75-65-0	10	40	100	5000	59	41	4
Acetonitrile*	75-05-8	100	100	NA	NA	41	40,39	1
Isopropyl Ether (Diisopropyl ether)	108-20-3	1	1	4	200	45	87,59	1
Chloroprene	126-99-8	1	1	NA	NA	53	88,90	1
1,1-Dichloroethane	75-34-3	0.5	1	4	50	63	65, 83	1
Acrylonitrile	107-13-1	10	10	100	2000	53	52,51	1
ethyl tert-butyl ether	637-92-3	0.5	1	4	200	59	87	1
Vinyl Acetate	108-05-4	10	10	10	500	43	86	1
cis-1,2-Dichloroethene	156-59-2	0.5	1	4	50	96	61,98	1

Analyte	CAS Number	Low Level Waters (µg/L)	Waters (µg/L)	Low-Level Soils (µg/kg)**	Medium Level Soils (µg/kg)***	Primary Ion	Secondary Ion(s)	Internal Standard used for Quantitation
2,2-Dichloropropane	594-20-7	1	4	10	200	77	97	1
Cyclohexane*	110-82-7	5	5	10	250	56	84,41	1
Bromochloromethane	74-97-5	1	1	4	50	130	49, 128	1
Chloroform	67-66-3	0.5	1	4	50	83	85	1
Carbon Tetrachloride	56-23-5	1	1	4	50	117	119	1
Tetrahydrofuran	109-99-9	10	10	40	2000	72	71,42	2
Ethyl acetate	141-78-6	5	5	NA	NA	43	61,70	1
1,1,1-Trichloroethane	71-55-6	0.5	1	4	50	97	99,61	1
Dibromofluoromethane (S)	1868-53-7	SS	SS	SS	SS	113		1
Sec-Butyl alcohol	78-92-2	40	40	NA	NA	45	59	4
1,1-Dichloropropene	563-58-6	0.5	1	4	50	75	110,77	1
2-Butanone (MEK)*	78-93-3	5	5	20	250	43	72	1
2,2,4-trimethylpentane *	540-84-1	4	4	-	-	57	56	1
Benzene	71-43-2	0.5	1	4	20	78	77	1
Propionitrile	107-12-0	40	40	NA	NA	54	55,52	1
Methacrylonitrile	126-98-7	4	5	NA	NA	41	67,39	1
Pentafluorobenzene (IS#1)	363-72-4	IS	IS	IS	IS	168		
tert-amyl methyl ether	994-05-8	0.5	1	4	200	73	87,55	1
1,2 Dichloroethane d4 (S)	17060-07-0	SS	SS	SS	SS	65	67,51	1
1,2-Dichloroethane	107-06-2	0.5	1	4	50	62	98	1
Isobutanol	78-83-1	80	80	400	4000	43	41,42	4
tert-amyl alcohol	75-85-4	10	10	100	5000	59	73,55	4
methylcylohexane	108-72-2	1	1	Na	Na	98	83,55	1
Trichloroethene	79-01-6	0.4	0.4	4	50	130	95, 132	3
1,4 Difluorobenzene (IS #3)	540-36-3	IS	IS	IS	IS	114		
Tert-amyl ethyl ether	919-94-8	0.5	1	4	200	59	87, 73	3
Dibromomethane	74-95-3	0.5	4	4	50	174	95,93	3
n-Butanol	71-36-3	200	200	NA	NA	56	41,43	4
1,2-Dichloropropane	78-87-5	4	4	4	50	63	112	3
Bromodichloromethane	75-27-4	0.5	1	4	50	83	85,127	3
Ethyl Acrylate	140-88-5	4	5	NA	NA	55	56	3
1,4 Dioxane-d8 (IS #4)	17647-74-4	IS	IS	IS	IS	96	64	
1,4-Dioxane	123-91-1	200	200	400	10000	88	58,57	4
Methyl Methacrylate	80-62-6	4	5	NA	NA	69	41,100	3
3-Pentanone	96-22-0	4	4	NA	NA	57	86	3
2-Chloroethyl Vinyl Ether	110-75-8	10	10	25	500	63	106, 65	3
cis-1,3-Dichloropropene	10061-01-5	0.5	4	4	50	75	77, 39	3

Analyte	CAS Number	Low Level Waters (µg/L)	Waters (µg/L)	Low-Level Soils (µg/kg)**	Medium Level Soils (µg/kg)***	Primary Ion	Secondary Ion(s)	Internal Standard used for Quantitation
Toluene d8 (S)	2037-26-5	SS	SS	SS	SS	98	100	5
Toluene	108-88-3	0.5	1	4	50	92	91	5
2-Nitropropane	79-46-9	10	10	NA	NA	43	41, 39	5
Tetrachloroethene	127-18-4	0.5	1	4	50	166	168, 129	5
4-Methyl-2-Pentanone (MIBK)*	108-10-1	5	5	20	250	43	58, 85	5
trans-1,3-Dichloropropene	10061-02-6	0.5	4	4	50	75	77,39	5
1,1,2-Trichloroethane	79-00-5	0.5	1	4	50	97	83, 85	5
4-Methyl-2-pentanol	108-11-2	40	40	NA	NA	45	69,87	4
Ethyl Methacrylate	97-63-2	4	5	NA	NA	69	41,99	5
Dibromochloromethane	124-48-1	0.5	1	4	50	129	127	5
1,3-Dichloropropane	142-28-9	0.5	1	4	50	76	78	5
1,2-Dibromoethane	106-93-4	0.5	1	4	50	107	109, 188	5
2-Hexanone*	591-78-6	5	5	20	250	43	58, 57	5
Chlorobenzene d5 (IS#5)	3114-55-4	IS	IS	IS	IS	117		
Chlorobenzene	108-90-7	0.5	1	4	50	112	77, 114	5
Ethylbenzene	100-41-4	0.5	1	4	50	91	106	5
1,1,1,2-Tetrachloroethane	630-20-6	0.5	1	4	50	131	133, 119	5
m&p-Xylene	7816-60-0	1	2	8	100	106	91	5
o-Xylene	95-47-6	0.5	1	4	50	106	91	5
Bromoform	75-25-2	4	4	20	200	173	175,254	5
Styrene	100-42-5	0.5	1	4	50	104	78	5
Isopropyl benzene (Cumene)	98-82-8	0.5	1	4	50	105	120	5
4-Bromofluorobenzene (BFB) (S)	460-00-4	SS	SS	SS	SS	95		6
Bromobenzene	108-86-1	0.5	1	4	50	156	77,158	6
Cis-1,4-Dichloro-2-butene	1476-11-5	4	4	NA	NA	53	77, 75	6
n-Propylbenzene	103-65-1	0.5	1	4	50	91	120	6
1,1,2,2-Tetrachloroethane	79-34-5	0.5	1	4	50	83	131, 85	6
2-Chlorotoluene	95-49-8	0.5	1	4	50	91	126	6
1,2,3-Trichloropropane	96-18-4	4	4	4	200	110	75, 112	6
4-Ethyltoluene	622-96-8	1	1	4	50	105	120	6
1,3,5-Trimethylbenzene	108-67-8	0.5	1	4	50	105	120	6
Trans-1,4-Dichloro-2-butene	110-57-6	10	10	50	500	53	88, 75	6
4-Chlorotoluene	106-43-4	0.5	1	4	50	91	126	6
tert-Butylbenzene	98-06-6	0.5	1	4	50	119	91,134	6
1,2,4-Trimethylbenzene	95-63-6	0.5	1	4	50	105	120	6
sec-Butylbenzene	135-98-8	0.5	1	4	50	105	134	6

Analyte	CAS Number	Low Level Waters (µg/L)	Waters (µg/L)	Low-Level Soils (µg/kg)**	Medium Level Soils (µg/kg)***	Primary Ion	Secondary Ion(s)	Internal Standard used for Quantitation
Dicyclopentadiene	77-73-6	4	4	4	200	66	39,132	6
p-Isopropyltoluene	99-87-6	0.5	1	4	50	119	134, 91	6
1,3-Dichlorobenzene	541-73-1	0.5	1	4	50	146	111, 148	6
1,4-Dichlorobenzene-d4 (IS#6)	3855-82-0	IS	IS	IS	IS	152		
1,4-Dichlorobenzene	106-46-7	0.5	1	4	50	146	111, 148	6
1,2,3-Trimethylbenzene	526-73-8	1	1	NA	NA	105	120	6
n-Butylbenzene	104-51-8	0.5	1	4	50	91	92, 134	6
1,2-Dichlorobenzene	95-50-1	0.5	1	4	50	146	111, 148	6
1,2-Dibromo-3-chloropropane	96-12-8	4	4	10	500	75	155,157	6
Hexachloro-1,3-butadiene	87-68-3	1	1	10	250	225	227,223	6
1,2,4-Trichlorobenzene	120-82-1	0.5	1	4	50	180	182, 145	6
Naphthalene	91-20-3	1	4	10	200	128		6
1,2,3-Trichlorobenzene	87-61-6	0.5	1	4	50	180	182, 145	6
2-Methylnaphthalene*	91-57-6	5	5	20	250	142	141	6
Xylene (total)	1330-20-7	1.5	3	12	150	NA	NA	5
1,2-Dichloroethene (total)	540-59-0	1	2	8	100	NA	NA	1
BTEX (total)	N/A	1	2	NA	NA	NA	NA	1,5
Total 1,3-Dichloropropene	NA	1	8	NA	NA	NA	NA	3,5

*Can be lab contaminate and therefore MDL set to ½ PRL and not statistical MDL.**Reporting limit for a 5g soil preserved with 5mL DI water ***Reporting limit for a 25g/10g soil preserved with 25mL/10mL methanol

1,4 Dioxane-d8 internal standard is used to quantify the water soluble compounds. Acetone d6 is used to quantify acetone, THF and hexane.

Note: Hexane uses 86 as the primary ion due to co-elution with MTBE.

Important NOTE: Reporting Limits may vary. For the most current reporting limits, refer to HORIZON.

Attachment II: Characteristic Ions for TCLP Target Compounds

Parameter	Primary Ion	Secondary Ions
Vinyl chloride	62	64
1,1-Dichloroethene	96	61, 63
Chloroform	83	85
1,2–Dichloroethane	62	98
2-Butanone	43	72
Carbon tetrachloride	117	119, 121
Trichloroethene	130	95, 132
Benzene	78	77
Tetrachloroethene	166	168, 129
Chlorobenzene	112	77, 114
1,4- Dichlorobenzene	146	111, 148

Attachment III: Analytes, Quantitation Limits and Regulatory Levels for TCLP Compounds

Parameter	CAS Number	Quantitation Limit (µg/L)	Regulatory Level (µg/L)
Vinyl chloride	75-01-4	10	200
1,1-Dichloroethene	75-35-4	25	700
Chloroform	67-66-3	25	6,000
1,2-Dichloroethane	107-06-2	25	500
2-Butanone	78-93-3	100	200,000
Carbon tetrachloride	56-23-5	25	500
Trichloroethene	79-01-6	25	500
Benzene	71-43-2	25	500
Tetrachloroethene	127-18-4	25	700
Chlorobenzene	108-90-7	25	100,000
1,4- Dichlorobenzene	106-46-7	25	7,500

Attachment IV: Standard 8260B Volatile Target List

ANALYTE NAME	ANALYTE NAME
1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,1,1-Trichloroethane
1,1,1,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane	1,1,2-Trichlorotrifluoroethane
1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,2,3-Trichlorobenzene
1,2,3-Trichloropropane	1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene	1,3-Dichlorobenzene
1,3-Dichloropropane	1,3,5-Trimethylbenzene
1,4-Dichlorobenzene	2,2-Dichloropropane
2-Chlorotoluene	4-Chlorotoluene
Acetone	Allyl chloride
Bromochloromethane	Benzene
Bromobenzene	Bromoform
Bromomethane	Cis-1,2-Dichloroethene
Cis-1,3-Dichloropropene	Carbon Tetrachloride
Chlorodibromomethane	Chlorobenzene
Chloroethane	Chloroform
Chloromethane	1,2-Dibromo-3-chloropropane
Dibromomethane	Dichlorodifluoromethane
Dichlorofluoromethane	1,2-Dibromoethane
Ethylbenzene	Ethyl ether
Hexachlorobutadiene	Isopropylbenzene
Methylene chloride	Methyl ethyl ketone
Methyl isobutyl ketone	Methyl tertiary butyl ether
n-Butylbenzene	Naphthalene
n-Propylbenzene	o-Xylene
p&m-Xylene	p-Isopropyltoluene
Sec-Butylbenzene	Styrene
Tert-Butylbenzene	Trans-1,2-Dichloroethene
Trans-1,3-Dichloropropene	Trichloroethene
Trichlorofluoromethane	Tetrachloroethene
Tetrahydrofuran	Toluene
Vinyl chloride	Bromodichloromethane

Attachment V: Common Dilution Factors for Water Samples

Water Dilution Factors		
Dilution	**Into 50 mL**	**Into 100 mL**
2x	25 mL	n/a
5x	10 mL	20 mL
10x	5 mL	10 mL
20x	2.5 mL	5 mL
25x	2 mL	4 mL
50x	1000 uL	2 mL
100x	500 uL	1000 uL
200x	250 uL	500 uL
500x	100 uL	200 uL
1000x	50 uL	100 uL
10000x	5 uL	10 uL

Attachment VI: Common Dilution Factors for TCLP Samples

Pace Analytical	Document Name: **TCLP Dilution Factors**	Document Revised: 28Feb2018 Page 1 of 1
	Document No.: **F-MN-O-138-rev.04**	Issuing Authority: Pace Minnesota Quality Office

TCLP Dilution Factors		
Dilution	**Into 50 mL**	**Into 100 mL**
1x	2 mL	4 mL
2x	1 mL	2 mL
5x	400 µL	800 µL
10x	200 µL	400 µL
20x	100 µL	200 µL
50x	40 µL	80 µL
100x	20 µL	40 µL

Attachment VII: Common Dilution Factors for Medium Level Samples

Soil Dilutions into 50mLVolumetric		
Dilution Factor	Volume of Soil Extract (uL)	Volume of P&T Methanol (uL)
1	1000	0
2	500	500
5	200	800
10	100	900
20	50	950
25	40	960
50	20	980
100	10	990
200	5	995
500	2	998
1000	1	999
Beyond 1000x Serial Dilutions are performed.		

Soil Dilutions into 100mLVolumetric		
Dilution Factor	Volume of Soil Extract (uL)	Volume of P&T Methanol (uL)
1	2000	0
2	1000	1000
5	400	1600
10	200	1800
20	100	1900
25	80	1920
50	40	1960
100	20	1980
200	10	1990
500	4	1996
1000	2	1998
Beyond 1000x Serial Dilutions are performed.		

Form O139Rev.01 30Jan2008

ATTACHMENT VIII: 1,4 Dioxane by 8260B SIM

Analyte	CAS Number	Reporting Limit in ug/L	Primary Ion	Secondary Ion	Internal Standard	Surrogate
1,4 Dioxane by SIM	123-91-1	1	88	58	1,4 dioxane d8	Toluene-d8

Standard	Standard(s) Amount	Solvent	Solvent Diluent Volume	Final Total Volume	Final Concentration
Intermediate Tune Solution	500 µL	MeOH	100 mL	50 µg/mL	Intermediate Tune Solution
Tune	5 µL	Water	5 mL	50 µg/L	Tune
Calibration Working Standard	0.5 mL of 1000 – 40,000 mg/L	MeOH	9.5 mL of MeOH	10.0 mL	50 - 2000 µg/mL (nominal conc. 100 µg/mL)
Calibration Std 1	0.5 µL		99.999 H_2O	100 mL	0.5 µg/L
Calibration Std 2	1.0 µL		99.997 H_2O	20 mL	1.0 µg/L
Calibration Std 3	3.0 µL		99.994 H_2O		3.0 µg/L
Calibration Std 4	6.0 µL		99.99 H_2O		6.0 µg/L
Calibration Std 5	10.0 µL		99.98 H_2O		10.0 µg/L
Calibration Std 6	20.0 µL		99.95 H_2O		20.0 µg/L
Calibration Std 7	50.0 µL		99.90 H_2O		50.0 µg/L
Calibration Std 8	100 µL		99.76 H_2O		100 µg/L
Calibration Std 9	0.250 µL		98.75 H_2O		250 µg/L
Surrogate Working Standard for Achon	0.5 mL of 10,000 mg/L		19.5 mL MeOH		250 µg/mL
Surrogate Working Standard for Centurion	0.5 mL of 10,000 mg/L		99.5 mL MeOH	100 mL	50 µg/mL
Internal Standard Working Standard for Archon	0.5 mL of 10,000 mg/L; 0.5 mL of 10,000 – 100,000 mg/L		19.5 mL MeOH	20 mL	250 µg/mL (1,4-dioxane-d8 is at 5000 µg/mL)
Internal Standard Working Standard for Centurion	0.5 mL of 10,000 mg/L; 0.5 mL of 10,000 – 100,000 mg/L		99.5 mL MeOH	20 mL	50 µg/mL (1,4-dioxane-d8 is at 5000 µg/mL)
Continuing Calibration Verification at 50ppb	100 µL of working standard	H_2O	199.9 mL H_2O	200 mL	50 µg/mL
Initial/Continuing Calibration Verification Standard at 20 ppb	50 µL of working standard	H_2O	249.95 mL H_2O	250 mL	20 µg/mL

Sample Preparation: Water samples are purged in the vial on the auto-sampler for SIM Analysis. The sample is prepared by adding 10mls of the sample using a 10ml syringe into a 40ml vial. All standards are treated in the same manner as the samples.

Suggested Purge Parameters: 6 minutes, 90 degrees heated purge at 120ml/mn

Attachment IX: Analytes using SIM acquisition for 8260B SIM/SCAN simultaneous acquisition

Analyte name	CAS Number	RL	acquisition method	Primary (Quant) Ion	Secondary (Qualifier) Ion
1,2,3-Trichloropropane	96-18-4	0.01	SIM	110	112
1,2-Dibromo-3-chloropropane	96-12-8	0.5	SIM	157	155
1,2-Dibromoethane (EDB)	106-96-4	0.05	SIM	107	109
1,2-Dichloroethane	107-06-2	0.2	SIM	62	98
1,4-Dioxane (p-Dioxane)	123-91-1	1	SIM	88	58
Carbon tetrachloride	56-23-5	0.05	SIM	117	119
cis-1,3-Dichloropropene	10061-01-5	0.4	SIM	75	77
Dibromochloromethane	124-48-1	0.4	SIM	129	127
Hexachloro-1,3-butadiene	87-68-3	0.2	SIM	225	227
trans-1,3-Dichloropropene	10061-02-6	0.4	SIM	75	77
Trichloroethene	79-01-6	0.05	SIM	130	132
Vinyl chloride	75-01-4	0.01	SIM	62	64

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STANDARD OPERATING PROCEDURE

SOIL EXTRACTION FOR PAH ANALYSIS BY GC/MS:SIM

Reference Methods: SW846 EPA Method 3550C

Local SOP Number:	S-MN-O-540-rev.15
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-540-rev.14

APPROVALS

Laboratory General Manager — Date: 27 Jun 2018

Laboratory Quality Manager — Date: 27 Jun 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-540-rev.15

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1. Purpose/Identification of Method

1.1. Standard Operating Procedure (SOP) describes the extraction by sonication of solid samples by EPA Method 3550C for the determination of polynuclear aromatic hydrocarbons by gas chromatography/mass spectrometry utilizing selected ion monitoring as delineated in EPA Method 8270C/D.

2. Summary of Method

2.1. A 30 gram sample aliquot may be extracted in a sonicator. After the extraction process, the extract is concentrated to a final volume of one milliliter.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method process.

3.2. **Parameters**: This SOP applies to compounds extracted for semi-volatile compounds (BNAs).

4. Applicable Matrices

4.1. This SOP is applicable to solid samples.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) and method detection limits (MDLs) for all analytes are available in the laboratory information management system (LIMS) and are available by request from the Quality Manager.

6. Interferences

6.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Interferences by phthalate esters can pose a major problem in organic analysis when using the electron capture detector. These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. Phthalates are easily extracted or leached from materials during laboratory operations. Cross- contamination of clean glassware routinely occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

6.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the site being sampled.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Collection, Preservation and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	4 oz glass amber jar with Teflon-lined lid	Thermal preservation only	<6°C but above freezing	Samples must be extracted within 14 days of collection and must be analyzed within 40 days of extraction

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Beakers	400 mL glass beakers	Fisher Scientific or equivalent replacement
Erlenmeyer flasks	500 mL	Fisher Scientific or equivalent replacement
Autosampler vials	2 mL, clear glass	Fisher Scientific or equivalent replacement
Microsyringes	Various sizes	Hamilton or equivalent replacement
Funnels		Fisher Scientific or equivalent replacement
Glass wool		Fisher Scientific or equivalent replacement
Filter paper	Fluted	Reeve Angel, 802 Fluted, 5802-185, or equivalent replacement
Silica gel	60-100 mesh pre-activated	Supelco 236799-IKG or equivalent
Pasteur pipettes	5 ¾ inch	Fisher Scientific 13-678-20B or equivalent vendor
Glass Jar	16 oz	C&G or equivalent replacement
Tilting dispenser	To transfer solvent to the beakers	Fisher Scientific or equivalent replacement
Kuderna-Danish Flasks (KD)	500 mL with ground glass joints	Fisher Scientific or equivalent replacement
KD Concentrator Tubes	10 mL, graduated with ground glass fittings	Fisher Scientific or equivalent replacement
Snyder columns	2-ball and 3-ball varieties	Fisher Scientific or equivalent replacement
Heated water bath	Capable of holding temperatures at 95 C	Fisher Scientific or equivalent replacement
Boiling chips		Fisher Scientific or equivalent replacement
Balance	Analytical balance capable of weighing 0.01 gm, top loading	Fisher Scientific or equivalent replacement
Sonicator		Fisher Scientific or equivalent replacement
KimWipe		Fisher Scientific or equivalent replacement
Laboratory Information System (LIMS)	Epic Pro used to create and track batches	Horizon
Butter Knife		Dollar store

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards.

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Sand	Ottawa 20-30 mesh	Fisher Scientific or equivalent replacement
Methylene Chloride	Pesticide quality or equivalent	Fisher Scientific or equivalent replacement
Acetone	Pesticide quality or equivalent	Fisher Scientific or equivalent replacement
Base Neutral Surrogate Mix	5000 ug/mL	Restek 31086 (or equivalent)
Custom Full SIM PAH Mix	250 ug/mL	Restek 555193 (or equivalent)
Na_2SO_4	Baked per SOP S-MN-O-500 (or equivalent replacement) - Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes	Fisher Scientific(or equivalent) S415-200LB

10.2. Table 10.2 Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount (mL)	Solvent	Solvent Volume	Final Total Volume	Final Concentration
SIM Intermediate Surrogate Solution	Base Neutral Surrogate Mix	1.5 mL	Methylene Chloride	3.5 mL	5.0 mL	1500 µg/mL
SIM Surrogate Spiking Solution	SIM Intermediate Surrogate Solution	1.0 mL	Acetone	499 mL	500 mL	3.0 µg/mL
SIM Matrix Spike Solution	Custom Full SIM PAH Mix	.800 mL	Acetone	199.2 mL	200 mL	1.0 µg/mL

11. Calibration and Standardization

11.1. See SOP S-MN-Q-264 - Support Equipment (or equivalent replacement) for support equipment calibration information.

12. Procedure

12.1. See S-MN-O-465 Glassware Cleaning, or equivalent replacement, on proper glassware and separatory funnel cleaning practices. Rinse and label all glassware appropriately.

12.2. Group samples by fraction to a maximum of 20 paying samples per extraction batch. An extraction batch consists of a method blank, appropriate quality control samples, and associated paying samples.

12.3. Transfer sample to 16 oz. jar and refer to S-MN-L-147, or equivalent replacement, Sample Homogenization and Sub-sampling SOP for homogenization process.

12.4. Weigh approximately 30 g of sample to the nearest 0.1g into a 400 mL beaker. Record this value on the extraction batch sheet (see Attachment I).

12.4.1. Add enough granular sodium sulfate to make sample free-flowing.

12.4.2. Mix well with a scoopula. Allow the samples to dry and mix again if needed.

12.5. A matrix spike (MS) and matrix spike duplicate (MSD) should be weighed out to 30 g (record weight to the 0.1 g, which should be to the same weight as the original sample, on extraction benchsheet. The client, supervisor, or project manager may designate the MS/MSD sample, but one set should be completed per batch. A laboratory control sample (LCS) should also be prepared by weighing 30 g of Ottawa sand into a 400 mL beaker.

12.5.1. If there is insufficient sample remaining to perform an MS/MSD or additional quality control information is needed (program specific), a laboratory control sample duplicate (LCSD) may be prepared.

12.6. Prepare a laboratory control sample (LCS) with each group of soil/sediment samples to be extracted. An LCS consists of weighing 30 g of Ottawa sand (to the nearest 0.1g) into a 400 mL beaker.

12.7. Prepare a method blank with each group of soil/sediment samples to be extracted. A method blank consists of weighing 30 g of Ottawa sand (to the nearest 0.1 g) into a 400 mL beaker.

12.8. To all quality control samples (MS/MSD/LCS/LCSD) 1.0 mL SIM matrix spiking solution (Table 10.2) should be added.

12.9. Add 1 mL of SIM surrogate standard (Table 10.2) to all samples, blanks, and quality control samples.

12.10. All spiking should be verified by a second person and recorded on the extraction bench sheet (Attachment I) according to SOP S-MN-O-497, or equivalent replacement.

12.11. Immediately after the spiking solution has been added to the sample, 60 mL of 80:20 Methylene Chloride/Acetone is added to the sample. The verification person may add this. Alternate volumes may be needed to completely cover or submerge the sample. Note if alternate volumes than 60 mL are utilized.

12.11.1. Move tilting dispenser or solvent pump hose in a circular motion to rinse walls of beaker with solvent.

12.12. Cover with aluminum foil to prevent solvent evaporation.

12.13. The sonicator should be tuned prior to sample contact according to SOP S-MN-O-414, or equivalent replacement.

12.14. Clean sonicator probe by washing with Methylene Chloride and wiping with a KimWipe or paper towel.

12.15. Place sample in the sonicator.

12.15.1. Lift probe

12.15.2. Place sample in the middle of the platform

12.15.3. Lower the probe

12.15.3.1. The probe tip should be approximately half way between the top of the soil and the top of the solvent. The platform may be adjusted by turning the lab jack.

12.15.3.2. Note: It may be easy to keep track of sonications 1, 2, and 3 by pointing the spout of the beaker first towards 3 o'clock, then 6 o'clock, and finally 9 o'clock.

12.16. Close the door and latch

12.17. Turn on the sonicator.

12.17.1. Directions will appear on the screen.

12.17.2. Press 'clear'

12.17.3. Press 'prog'

12.17.4. Note: It should be set to pulse for 3 minutes at 1:15 setting at 2.0 pulses/second

12.17.5. Press 'start'

12.18. When pulsing stops, remove the sample.

12.18.1. Open the door

12.18.2. Lift the probe

12.18.3. Rinse the probe with Methylene Chloride into the sample.

12.19. Filter

12.19.1. Assemble funnel and filter paper.

12.20. Repeat procedure two more times- add 60 mL of solvent and mix, followed by sonication and filtering.

12.21. After the third sonication, pour all of the soil into the funnel

12.21.1. Two methods that may be used:

12.21.1.1. Use a butter knife to push the soil into the funnel. Rinse the beaker and butter knife with Methylene Chloride.

12.21.1.2. Use wash bottle and a minimal amount of Methylene Chloride to wash the soil into the funnel.

12.22. Clean the sonicator probe (12.13)

12.23. Turn power off.

12.24. Remove funnel; throw soil into trash.

12.25. Cover flask and side arm (if applicable) with tinfoil and store in cooler, or follow with concentration (refer to SOP S-MN-O-504, or equivalent replacement).

12.26 Optional Sample Clean-up for CPAH soil – if requested by the client

12.26.1 The cleanup procedure can either take place after the initial CPAH extract has been analyzed and client evaluates the results or it can be silica gel cleaned before the extracts are analyzed. It

is important that the original extract in the case of silica gel clean up being requested after client evaluates the results be used to minimize the variability of non-homogeneous samples.

12.26.2 The micro column is created by filling a 5 ¾ inch Pasteur pipet ¾ full with silica gel. The column is then placed into a 1 liter jar containing Methylene Chloride and allowed to soak. Do not use the silica columns until the silica gel has been fully saturated with methylene chloride. Once saturated place the columns in the metal column holder stand located in O-prep.

12.26.3 Uncap the vials containing the sample and transfer the extract to the top of the column with a disposable pipet. Rinse the extract vial with 1 mL of MeCl and transfer to the top of the column. Repeat 3 times. If silica gel clean up is requested up front then transfer extract from concentrator tube and follow directions from 12.26.3.

12.26.4 Elute with additional MeCl to obtain a total of 7 mL of solvent collected into a 7mL vial.

12.26.5 Concentrate down to a final volume of 1 mL on the N-Evap.

13. Quality Control

13.1. Table 13.1 Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Baked Ottowa sand	One per 20 samples; Each shift of 8 hours that samples are extracted	Refer to appropriate analytical SOP.	Refer to appropriate analytical SOP.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	Baked Ottowa sand spiked with all target compounds	One per 20 samples. If an MS/MSD is prepared, only an LCS is required.	Refer to appropriate analytical SOP.	Refer to appropriate analytical SOP.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Client sample spiked with all target compounds	One per 20 samples. If an MS/MSD pair is not available, a laboratory control spike and laboratory control spike duplicate (LCS/LCSD) may be prepared.	Refer to appropriate analytical SOP.	Refer to appropriate analytical SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3550C "Separatory Funnel Extraction".

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: SIM Surrogate Compound.

26.2. Attachment II: SIM Matrix Spike Compounds

26.3. Attachment III: SIM Intermediate PCP Surrogate Compounds

27. Revisions

Document Number	Reason for Change	Date
S-MN-O-540-rev.14	Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Table 13.1 MB and LCS/LCSD rows, Component column updated to baked Ottowa sand instead of water. Removed Attachment IV.	21Sept2017
S-MN-O-540-rev.15	Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Table 9.1 – added Silica gel and Pasteur pipettes rows Added new section 12.26 and all subsections.	25Jun2018

Attachment I: SIM Surrogate Compounds

Compounds	Concentration (µg/mL)
Nitrobenzene	3.0
2-Fluorobiphenol	3.0
p-Terphenyl-d14	3.0

Attachment II: SIM Matrix Spike Compounds

Compounds	Concentration (µg/mL)
Naphthalene	1.0
2-Methylnaphthalene	1.0
Acenaphthylene	1.0
Acenaphthene	1.0
Fluorene	1.0
Phenanthrene	1.0
Anthracene	1.0
Fluoranthene	1.0
Pyrene	1.0
Benzo(a)anthracene	1.0
Chrysene	1.0
Benzo(b)fluoranthene	1.0
Benzo(k)fluoranthene	1.0
Benzo(a)pyrene	1.0
Indeno(1,2,3-cd)pyrene	1.0
Dibenz(a,h)anthracene	1.0
Benzo(g,h,i)perylene	1.0
1-Methylnaphthalene	1.0
2-Chloronaphthalene	1.0
Dibenzofuran	1.0

Attachment III: SIM Intermediate PCP Surrogate Compound

Compounds	Concentration (µg/mL)
2,4,6-Tribromophenol	1.0

Document Information

Document Number: ENV-SOP-MIN4-0085 **Revision:** 00

Document Title: Separatory Funnel Extraction

Department(s): Organic Prep

Previous Document Number: S-MN-O-566-rev.06

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Effective Date: 27 Jun 2018

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

SEPARATORY FUNNEL EXTRACTION

Reference Methods: SW846 EPA Method 3510C

Local SOP Number:	S-MN-O-566-rev. 06
Effective Date:	Date of Final Signature
Supersedes:	S-MN-O-566-rev.05

APPROVALS

Laboratory General Manager — Date: 27 Jun 2018

Laboratory Quality Manager — Date: 08 Jun 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-O-566-rev.06

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to provide a laboratory specific procedure for extracting semi-volatile organic compounds from aqueous samples in a separatory funnel while meeting the requirements specified in EPA method 3510 followed by EPA SW-846 8270C/D and EPA Method 625 analysis.

2. Summary of Method

2.1. A measured volume of sample, usually about 1 liter, is serially extracted with solvent in a separatory funnel. Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup or analysis.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method process.

3.2. **Parameters**: This SOP applies to compounds extracted for semi-volatile compounds (BNAs).

4. Applicable Matrices

4.1. This SOP is applicable to aqueous samples.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) and method detection limits (MDLs) for all analytes are available in the laboratory information management system (LIMS) and are available by request from the Quality Manager.

6. Interferences

6.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2. Interferences by phthalate esters can pose a major problem in organic analysis.. These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. Phthalates are easily extracted or leached from materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

6.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the site being sampled.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Collection, Preservation and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid (preferably 1L wide mouth).	Thermal preservation only	<6°C but above freezing	Samples must be extracted within 7 days of collection and must be analyzed within 40 days of extraction

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Separatory funnels	Teflon, able to hold 2 L with PTFE stopcocks and lids	Fisher Scientific or equivalent replacement
Mechanical shaker	A mechanical tumbling apparatus capable of holding and rotating 2 L sized separatory funnels	
Erlenmeyer flasks	250mL	Fisher Scientific Cat.# 07250090 or equivalent replacement
Autosampler vials	2mL, clear glass	Fisher Scientific or equivalent replacement
Microsyringes	Various sizes	Hamilton or equivalent replacement
Funnels	Stainless Steel	Fisher Scientific Cat.# 10-368B or equivalent replacement
Glass wool		Fisher Scientific Cat.#11-388 or equivalent replacement
Graduated cylinder	1L Class A or equivalent	Fisher Scientific Cat.# 08-548-207or equivalent replacement
Metal measuring device	To be utilized in determining volume of C&G amber 1L containers	
Kuderna-Danish Flasks (KD)	500mL with ground glass joints	Fisher Scientific or equivalent replacement
KD Concentrator Tubes	10mL, graduated with ground glass fittings	Fisher Scientific or equivalent replacement
Snyder columns	2-ball and 3-ball varieties	Fisher Scientific or equivalent replacement
Heated water bath	Capable of holding temperatures at 95 C	Fisher Scientific or equivalent replacement
Boiling chips		Fisher Scientific Cat.#02215521
Pipettes	Disposable for dispensing acid or base	Fisher Scientific or equivalent replacement
pH paper		Fisher Scientific or equivalent replacement
Heating mantles		Fisher Scientific or equivalent replacement
250mL vials	Used to centrifuge emulsions	Fisher Scientific or equivalent replacement
Laboratory Information System (LIMS)	Epic Pro used to create and track batches	Horizon

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards.

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular	See S-MN-O-500, or equivalent replacement
Methylene Chloride	Extraction solvent	Pesticide quality or equivalent
Acetone	Extraction solvent	Pesticide quality or equivalent
Sulfuric acid solution	1:1 solution – Slowly add 400mL of concentrated H2SO4, 10mL at a time and stir for 2 minutes until all 400mL have been added to 400mL of DI water. Place in an iced water bath during preparation, solution gets hot. Record all preparation in an appropriate logbook.	Fisher grade
Sodium Hydroxide pellets		Fisher grade
Sodium Hydroxide solution (10N)	Dissolve 240g sodium hydroxide pellets into 600mL of reagent water. Place in an iced water bath during preparation, solution gets hot.	Fisher grade
Restek 8270 Megamix	500-1000ug/mL	Restek, Order # 31850
Restek 605 Benzidines mix	2000ug/mL	Restek Order # 31030
Benzoic Acid Solution	2000ug/mL	Restek Order # 31879
Acid Surrogate Mix	10,000ug/mL	Restek Order # 31087
Base Surrogate Mix	5,000ug/mL	Restek Order # 31086

10.2. Table 10.2 Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount (mL)	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Working Matrix Spike Standard	5.0mL 8270 Megamix, 2.5mL 605 Benzidines, 2.5mL Benzoic acid	Acetone	40mL	50mL	100 ug/mL
Working BNA Surrogate Standard	5.0mL Acid surrogate, 7.5mL Base surrogate	Acetone	37.5mL	50mL	1000 and 750 ug/mL

11. Calibration and Standardization

11.1. See Support Equipment SOP S-MN-Q-264, or equivalent replacement, for calibration of pipettes and syringes.

12. Procedure

12.1. See S-MN-O-465 Glassware Cleaning, or equivalent replacement, on proper glassware and separatory funnel cleaning practices. Rinse and label all glassware appropriately.

12.2. Measure the initial volume and pH of each sample. Record the volumes on the extraction sheet.

12.2.1. If there is greater than 5% sediment, refer to SOP S-MN-L-142 (or equivalent replacement) for procedure.

12.2.2. Use 1L of DI water for the Method Blank and Laboratory Control Sample (LCS) for 8270 and 625 – pour directly into the separatory funnel. Use 100 mL of LB tumbled water for TCLP for Method blank and LCS.

12.2.2.1. The state of WI requires a set of MS/MSD in a batch if the sample is from WI. If there is insufficient sample for a MS/MSD the extra container from the state of WI must be

split into 3 portions (parent, MS and MSD). A placeholder must be requested from the PM and used as the parent sample. The volume will then be brought up for extraction purposes.

12.3. Add 100µL of surrogate solution to each QC and sample container, and 500µL of matrix spike solution to the LCS/MS/MSD. Transfer the samples to their labeled separatory funnel.

12.3.1. The technique used for spiking samples is referenced in SOP S-MN-O-498, or equivalent replacement.

12.4. All spiking should be verified by a second person and recorded on the extraction bench sheet (Attachments II and III) according to SOP S-MN-O-497, or equivalent replacement. Use 1L of DI water for the Method blank and Laboratory Control Sample (LCS) – pour directly into the separatory funnel. All samples and QC samples must be adjusted to a pH of <2 with 1:1 sulfuric acid (add 2mL of 1:1 sulfuric; cap and shake the separatory funnel for 8270/625. Add 0.2 mL 1:1 sulfuric for TCLP). Check the pH by using a disposable pipette to drop a small amount onto a pH strip. If additional acid is required, note it on the extraction sheet. (See Attachment I).

12.5. Rinse each glass liter that contains 5% sediment or less with approximately 60mL of $MeCl_2$

12.5.1. If sample contains more than 5% sediment the first aliquot of $MeCl_2$ will not be added to the glass liter. Notify the PM if a sample contains greater than 5% sediment.

12.6. Transfer the $MeCl_2$ to the funnel and shake the funnel for 2 minutes, making sure to properly vent the funnel initially into a hood (after first shake all aliquots of $MeCl_2$ will be added directly to the separatory funnel).

12.7. After the samples have been shaken (2 minutes for hand shake, 4 minutes on tumbler), wait 10 minutes.

12.8. Drain the $MeCl_2$ layer into a 500ml Erlenmeyer flask.

12.9. Drain through a funnel containing baked and rinsed sodium sulfate (Na_2SO_4) and a glass wool plug. Repeat 2 more times. See S-MN-O-500 (or equivalent replacement) for drying information.

12.10. Drain through a funnel containing baked and rinsed sodium sulfate (Na_2SO_4) and a glass wool plug. Repeat 2 more times. See S-MN-O-500 (or equivalent replacement) for drying information.

12.11. All samples and QC samples must be adjusted to a pH of >11 with 4.5 mL of 10N sodium hydroxide for 8270/625. Add 0.45 mL 10N sodium hydroxide for TCLP. Check the pH by using a disposable pipette to drop a small amount onto a pH strip. If additional sodium hydroxide is required, note it on the extraction sheet.

12.12. Drain through a funnel containing baked and rinsed sodium sulfate (Na_2SO_4) and a glass wool plug. Repeat 2 more times. See S-MN-O-500 (or equivalent replacement) for drying information..

12.13. Add approximately 60mL of $MeCl_2$ to each separatory funnel. Shake and drain $MeCl_2$ layer as listed above. Repeat two more times.

12.14. Extraction is complete when a total of 6 shakes are completed: 3 acid and 3 basic.

12.15. Assemble a KD/concentrator tube apparatus. Add baked sodium sulfate to each extract and transfer only the extract to the KD apparatus. Concentrate on the water bath per procedure S-MN-O-504 – sample concentration (or equivalent replacement).

13. Quality Control

13.1. Table 13.1 Quality Control.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than reporting limit.	Re-analyze associated s Re-extract and re-analyze associated samples if blank result is greater than RL

			If results are reported to MDL, target analytes in MB should be non-detect	samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)	DI water spiked with all target compounds	One per 20 samples LCSD is performed when there is insufficient sample volume for a MS/MSD or MS/Dup pair	Internally generated limits %RPD ± 20%	Re-extract and re-analyze a new LCS if original LCS is outside acceptance limits. If the precision limits are not met, but the recoveries are within limits treat as below. ***Exceptions:*** If LCS recovery is > QC limits and target analytes are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)/ Matrix Spike Duplicate (MSD)/ Duplicate (DUP)	Client sample spiked with all target compounds MS Duplicate *OR (alternative)* Sample Dup	One per 20 samples	Internally generated limits, %RPD ± 30%	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Reanalyze accordingly for client specific data quality objectives to confirm matrix interference. For Minnesota Admin Contract Clients- all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Surrogates	All applicable surrogate compounds	Added to each sample, blank and QC sample	Internally generated limits	Surrogates above limits but no hits- report samples with footnote. Surrogate limits above limits but with hits- re-extract if possible or report as biased high. Surrogates below limits: re-extract if possible or report as biased low.

14. Data Analysis and Calculations

14.1. See the Quality Manual for example calculations.

14.2. %RPD calculations:

$$\%RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where: RPD = relative percent difference
D_1 = first sample result

D_2 = second sample result

14.3. The following calculation can be used to calculate the LCS and MS percent recovery (where *SampleConc* would be equal to 0):

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-S-MN-Q-269, Method Detection Limit Studies for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279, Training and Employee Orientation.

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Reports are stored in the QA office.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. If sample emulsions appear after shaking, drain the emulsion into a clean 40mL vial and put in centrifuge. Then pour solvent from 40mL vial back into the separatory funnel and drain through the funnel. If an emulsion has formed, make a note on the extraction sheet. It may be handled in one of the following ways.

21.1.1. Tap on the side of the separatory funnel at the emulsion layer using the handle of a screwdriver until the emulsion separates. Then drain solvent layer into sodium sulfate funnel.

21.1.2. Centrifuge the emulsion in 250-mL centrifuge tubes with Teflon caps for 5 minutes at 1500 rpm.

21.1.3. Slowly poor the contents back into the separatory funnel and drain the entire solvent layer into the sodium sulfate funnel set up.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510 "Separatory Funnel Extraction".

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Separatory Funnel Extraction Procedure.

26.2. Attachment II: Example 8270 Extraction Sheet

26.3. Attachment III: Example 625 Extraction Sheet

27. Revisions

Document Number	Reason for Change	Date
S-MN-O-566-Rev.05	18.3 – updated to new local SOP number. Added 12.2.2.1 for state of WI	10May2018

S-MN-O-566-Rev.06	12.2.2 – added "for 8270 and 625" and "Use 100 mL of LB tumbled water for TCLP for Method blank and LCS." 12.4 – added "for 8270/625. Add 0.2 mL 1:1 sulfuric for TCLP" 12.11 – added "for 8270/625. Add 0.45 mL 10N sodium hydroxide for TCLP"	07Jun2018

Attachment I: Separatory Funnel Procedure

Pace Analytical	Document Name: Analysis: 8270C/D and 625 – Sep Funnel Extraction Procedure	Document Revised: 8Dec2015 Page 1 of 1
	Document No.: F-MN-O-188-rev.02	Issuing Authority: Pace Minnesota Quality Office

ANALYSIS: 8270C and 625 – Sep funnel Extraction Procedure

Holding Time: Samples should be extracted within 7 days from sample collection.

QC Requirements: A method blank (MB) and LCS/MS/MSD (if sufficient sample is available) must be performed each day or every 20 samples, whichever is more frequent.

Extraction Solvent: Methylene Chloride ($MeCl_2$)

Extraction:
•Rinse all glassware and sep funnels according to SOP S-MN-0-465. Label rinsed 2L separatory funnels with the sample ID.
•Measure the initial volume and pH of each sample and record on the extraction sheet.
-If more than 5% sediment is present in the sample container, refer to SOP MN-L-142.
- Use 1L of DI water for the MB and LCS – pour directly into the funnel.
• Add 100 µL of surrogate solution to each QC and sample container, and 500 µL of matrix spike solution to the LCS/MS/MSD. Pour samples into designated separatory funnels.
• All samples and QC samples will be adjusted to a pH of <2 with 1:1 sulfuric acid (add ~2 mL of 1:1 sulfuric acid; cap and shake sep. funnel) Check pH's using disposable pipets and pH paper. If additional acid is required to achieve a pH of <2, record it on extraction sheet.
•Rinse each glass liter that contains 5% sediment or less with approximately 60 mL of $MeCl_2$.
-If sample contains more than 5% sediment the first aliquot of $MeCl_2$ will not be added to the glass liter. Notify the PM if a sample contains greater than 5% sediment.
Transfer to the funnel and shake the funnel for 2minutes, making sure to properly vent the funnel initially into a hood (after first shake all aliquots of $MeCl_2$ will be added directly to the sep funnel).
•After the samples have been shaken (2 minutes for hand shake, 4 minutes on tumbler), wait 10 minutes.
•Drain the $MeCl_2$ layer into a 500ml Erlenmeyer flask.
•Drain through a funnel containing baked and rinsed sodium sulfate (Na_2SO_4) and a glass wool plug. Repeat 2 more times. See S-MN-O-500 for drying information.
•Adjust pH of all samples and QC to a pH of ≥11 by adding approximately 4.5 mL of a 10N sodium hydroxide solution to each sep funnel. Cap and shake each sep; take pH using disposable pipets and pH paper.
-If additional amounts of 10N sodium hydroxide are needed to reach the desired pH, record the amounts on the extraction sheet.
•Add approximately 60 mL of $MeCl_2$ to each sep funnel. Shake and drain $MeCl_2$ layer as listed above. Repeat two more times.
•Extraction is complete when a total of 6 shakes are completed: 3 acid and 3 basic.

Finalization: Assemble a KD/concentrator tube apparatus. Add baked sodium sulfate to each extract and transfer only the extract to the KD apparatus. Concentrate on the waterbath per procedure S-MN-O-504 –sample concentration.

Final Volume: 1.0 mL
Final Solvent: Methylene Chloride

Reviewed by:	Date:

Attachment II: Example 8270 Extraction Sheet

Pace Analytical®	Document Name: Extraction Sheet – 8270 Water Sep. Funnel	Document Revised: 04 Mar 2014 Page 1 of 1
	Document No.: F-MN-O-116 Rev.04	Issuing Authority: Pace Minnesota Quality Office

SS:		**Amt:** 100 µL	**Analyst:**	**Ext. Date/By:**	
MS:		**Amt:** 500 µL	**Analyst:**	**Batch:**	
BNA SS 750/1500 µg/mL		**BNA MS** 100 µg/mL		**Syringe ID:**	

	Sample ID	I.V. 1000mL	pH	Spike Ver.	pH <2	pH >11	F.V. 1.0mL Lot #	Comments
1	MB-	1000						
2	LCS-	1000						
3	LCSD-	1000						
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample ID Verified by:_____ $MeCl_2$ (Ext):_____ Date Conc. /By:_____

☐ Acid Extraction completed. 1:1 H_2SO_4 Lot:_____ $MeCl_2$ (F.V.):_____

☐ Base/Neutral Extraction completed. 10N NaOH Lot:_____ Bath Temp (90°) Read:_____

Glass Wool Lot:_____ Na_2SO_4 Lot:_____ Bath Temp (90°) Corrected:_____

Pipettor ID:_____ Thermometer ID:_____

Comments:_____

8270 – Water (Sep. Funnel)

Posted by/Date:	Validated by/Date:

Attachment III: Example 625 Extraction Sheet

Pace Analytical®	Document Name: Extraction Sheet – 625 Water Sep. Funnel	Document Revised: 04 Mar 2014 Page 1 of 1
	Document No.: F-MN-O-113 Rev.04	Issuing Authority: Pace Minnesota Quality Office

SS:		**Amt:** 100 µL	**Analyst:**	**Ext. Date/By:**	
MS:		**Amt:** 500 µL	**Analyst:**	**Batch:**	
BNA SS 750/1500 µg/mL		**BNA MS** 100 µg/mL		**Syringe ID:**	

	Sample ID	I.V. 1000mL	pH	Spike Ver.	pH <2	pH >11	F.V. 1.0mL Lot #	Comments
1	MB-	1000						
2	LCS-	1000						
3	LCSD-	1000						
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample ID Verified by: _____ $MeCl_2$ (Ext): _____ Date Conc. /By: _____

☐ Acid Extraction completed. 1:1 H_2SO_4 Lot: _____ $MeCl_2$ (F.V.): _____

☐ Base/Neutral Extraction completed. 10N NaOH Lot: _____ Bath Temp (90°) Read: _____

Glass Wool Lot: _____ Na_2SO_4 Lot: _____ Bath Temp (90°) Corrected: _____

Pipettor ID: _____ Thermometer ID: _____

Comments: _____

625 – Water
(Sep. Funnel)

Posted by/Date:	Validated by/Date:

Document Information

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Department(s): Wet Chemistry

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Date Information

Effective Date: 04 Oct 2018

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0102 **Revision:** 00

Title: Hexavalent Chromiumin in Water and Wastewater

All dates and times are in Central Time Zone.

Quick Approval

Approve Now

Name/Signature	Title	Date	Meaning/Reason
Jane Schur (JSCHUR)		04 Oct 2018, 01:14:27 PM	Approved

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STANDARD OPERATING PROCEDURE

HEXAVALENT CHROMIUM IN WATER AND WASTEWATER

Reference Methods: SM 3500Cr-B

Local SOP Number:	S-MN-I-358-rev.30
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-358-rev.29

APPROVALS

Laboratory General Manager — Date: 02 May 2018

Laboratory Quality Manager — Date: 20 Mar 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-358-Rev.30

TABLE OF CONTENTS

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. The purpose of this Standard Operating Procedure (SOP) is to determine the concentration of chromium (VI) in water, wastewater, per Standard Method 3500Cr-B.

2. SUMMARY OF METHOD

2.1. Hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color is produced and measured at 540 nm.

3. SCOPE AND APPLICATION

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. Parameters: This method measures hexavalent chromium at concentrations of 0.01-0.50 mg/L. Samples of greater concentration are determined by dilution into this range.

4. APPLICABLE MATRICES

4.1. This SOP is applicable to water and wastewater.

5. LIMITS OF DETECTION AND QUANTITATION

5.1. The applicable reporting limit is 0.01 mg/L. The most current reporting and detection limits can be found in the Laboratory Information Management System (LIMS).

6. INTERFERENCES

6.1. The chromium reaction with diphenylcarbazide is usually free from interferences. Hexavalent molybdenum (Mo) and mercury (Hg) salts will react to form color with the reagent but the intensities are much lower than that of chromium at the specified pH. Concentrations of Mo or Hg as high as 200 mg/L can be tolerated. Vanadium (V) interferes strongly but concentrations up to 10 times that of chromium will not have a significant contribution.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1. Collection, Preservation, Storage and Holding Time Table

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Samples are collected in glass or plastic bottles	N/A	Samples are stored above freezing but below 6°C	The maximum holding time prior to analysis of the samples or extracts is 24 hours

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Equipment and Supplies Table

Supply	Description	Vendor/ Item # / Description
Pipettes	5 mL	Fisher or equivalent
Volumetric Flask	50 mL and 100 mL	Fisher or equivalent
Filter	0.45 µm	Fisher part # SLHV033NK or equivalent
Syringes for Filtering	3 mL or larger	Fisher part # 14-823-436 or equivalent

pH paper	Narrow range, 0.3-2.8 or equivalent	Fisher or equivalent
Syringes	250 µL and 500 µL	Fisher or equivalent
Konelab Discrete Analyzer	Konelab 20	Thermo Fisher Scientific
EPIC Horizon	Data Reporting Software (LIMS)	See master list for current version

10. REAGENTS AND STANDARDS

10.1. Stock standard solutions are obtained from vendors in solution form. These solutions are stored per manufacturer's specifications, and have an expiration date of one year after being opened or the manufacturer's expiration date when specified.

10.2. Reagents and Standards Table

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized (DI) Water	N/A	Verify daily that pH and specific conductivity are within acceptable limits and record in the electronic prep log.
Stock Curve Standard	1000 mg Cr^{6+}/L. Store at room temperature. Expires as specified by manufacturer.	Fisher cat. # Spec-Cr6
Stock QC Standard	1000 mg Cr^{6+}/L. Store at room temperature. Expires as specified by manufacturer.	HACH cat. #14664-42
1,5-Diphenylcarbazide	Powder. Store at room temperature. Expires as specified by manufacturer.	Fisher cat. # D85-25
Sulfuric Acid (H_2SO_4)	Concentrated. Store at room temperature. Expires as specified by manufacturer.	Fisher cat. # A365-1
Acetone	Optima. Store at room temperature. Expires one year after opening.	Fisher cat. # A929-4

10.3. Calibration & QC Standards Table

Standard Name	Solution Used	Solution Volume	Solvent	Final Volume	Final Concentration
Intermediate Curve	Stock Curve Standard	5 mL	DI Water	100 mL	50 mg/L
Intermediate QC	Stock QC Standard	5 mL	DI Water	100 mL	50 mg/L
Initial & Continuing Calibration Verification (ICV/CCV)	Intermediate QC	0.2 mL	DI Water	50 mL	0.20 mg/L
Matrix Spike (MS/MSD)	Intermediate QC	0.04 mL	Sample matrix	10 mL	0.20 mg/L

10.4. Intermediate Curve & QC Solutions – Store in a glass container at a temperature above freezing but lower than 6°C. Expires in six months.

10.5. ICV/CCV and MS/MSD – Store in a glass container at room temperature. Prepare fresh daily.

10.6. Calibration Standards – Using the Intermediate Curve Solution, pipette 0.5 mL into a 50 mL volumetric flask and bring to volume with E-pure water (final concentration is 0.5 mg/L). The Konelab will prepare the remaining calibration standards by serial dilution (see table below).

Calibration Curve Table

Working Standard Concentration (mg/L)	Konelab Dilution	Final Concentration (mg/L)
0	1 + 0	0.0
0.5	1+99	0.005
0.5	1 + 49	0.01
0.5	1 + 9	0.05
0.5	1 + 4	0.10
0.5	1 + 1	0.25
0.5	1 + 0	0.50

10.7. Working Reagents

Standard Name	Reagent Used	Solution Volume	Solvent	Final Volume
Diphenylcarbazide Solution	1,5-Diphenylcarbazide	0.25 g	Acetone	50 mL
10% Sulfuric Acid	Concentrated Sulfuric Acid	10 mL	DI Water	100 mL

10.7.1. Prepare by dissolving 0.25g of 1,5-diphenylcarbazide in 50 mL of acetone. Store above freezing but below 6°C in an amber glass bottle. Discard when solution becomes discolored (pink). Prepare fresh weekly.

10.7.2. Add ~50 mL DI water to 100 mL Class A volumetric flask. Slowly add 10 mL concentrated sulfuric acid and bring to final volume of 100 mL. Expires in six months.

11. CALIBRATION AND STANDARDIZATION

11.1. Calibration Criteria Table

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression	$r \geq 0.995$	If not met, remake standards and recalibrate before sample analysis. Perform and document any maintenance necessary.
Initial & Continuing Calibration Verification (ICV/CCV)	Immediately after each initial calibration.	90-110% recovery of the true value	If the requirements for the ICV are not met, verify standard preparation, remake standards, and reanalyze one additional time. If still not met, the system must be recalibrated. If the requirements for the CCV are not met, stop the system and verify the standard preparation and calculation. Reanalyze the CCV. Only two injections of the same standard are permitted back to back; if failing the system must be stopped and recalibrated. All associated samples must be reanalyzed following passing CCVs.
Initial & Continuing Calibration Blank (ICB/CCB)	Immediately following every ICV and CCV.	The absolute value of the ICB/CCB must be less than the reporting limit. Some client QAPPs may require the absolute value of the ICB/CCB to be less than ½ the reporting limit.	If the absolute value exceeds the criteria, terminate the analysis. Correct the problem, verify the calibration, and re-analyze associated samples. ***Exceptions:*** If sample is ND, report the sample without qualification; If sample result >10x ICB/CCB detects, report sample as not impacted by the blank contamination; If sample result <10x ICB/CCB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

12. PROCEDURE

12.1. Sample Prep

12.1.1. If a sample contains a considerable amount of sediment or other solids, filter the sample through a 0.45 µm membrane filter. Filter the associated method blank and LCS and document which samples were filtered on the raw data run log as well as the lot number of the filter used.

12.2. Sample Analysis

12.2.1. Use Konelab test definition "SM3500Cr-B" to analyze Standard Methods 3500-Cr B samples. The diphenylcarbazide solution is added after the acidification with 10% H_2SO_4. The Konelab will automatically adjust pH of the sample to 2.0±0.5.

12.2.2. The Konelab will automatically correct for sample turbidity by performing a sample blank prior to sample analysis.

12.2.3. Insert the samples into the instrument and analyze as directed by Konelab SOP S-MN-I-507 (or equivalent replacement).

12.2.4. If a sample result is more negative than the PRL, the sample should be diluted to remove interferences. Use Konelab to dilute samples (1x, 2x, 4x, etc) until interferences are no longer present. If the sample has an MS/MSD associated with it, and the spike did not recover, then the MS/MSD should be diluted and post spiked.

12.3. Confirm that the pH range of 2.0 ± 0.5 (for SM3500Cr-B only) is achieved before addition of diphenylcarbazide solution by the Konelab. To perform the confirmation, simulate the chemistries performed by the Konelab in a sample cup. Perform the pH check annually with documentation in the Konelab maintenance logbook.

13. QUALITY CONTROL

13.1. QC Table

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI Water	Once per batch of up to 20 samples	The absolute value of the MB must be less than the reporting limit. Some client QAPPs may require the absolute value of the MB to be less than ½ the reporting limit.	When measurements are above the PRL, reanalyze. If reanalyzed method blank is still above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all associated samples. Sample data may be accepted if the results are non-detect or at least 10x greater than the method blank results.
Laboratory Control Sample (LCS)	The same solution used for the ICV/CCV may be used for the LCS. See table 10.3.	Once per batch of up to 20 samples.	%Rec: 90-110%	Analyze a new LCS. If the second LCS fails, re-analyze all data in the associated batch. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike& Matrix Spike Duplicate (MS/MSD)	See table 10.3.	Once every 10 samples with a minimum of two pairs of MS/MSD per batch of 20 samples.	%Rec: 85-115% %RPD: 20%	If the spike recovery is not 85-115% of the true value and/or the RPD is greater than 20%, **evaluate possible interferences or contamination.** If the spike added is greater than or equal to one-fourth of the sample result, all data with that parent sample is flagged.

				For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Sample Duplicate (DUP)	Client-provided sample.	Per client request.	%RPD: 20%	Report results with an appropriate footnote.

14. DATA ANALYSIS AND CALCULATIONS

14.1. See the most current Quality Manual for calculations

14.2. LCS Recovery

$$\text{LCS Recovery} = \frac{SSR \times 100\%}{SA}$$

Where: SSR = Spike Sample Results
SA = Spike Added from spiking mix

14.3. Relative Percent Differences (RPD)

$$RPD = \frac{|A-B|}{(A+B)/2} x100$$

Where:

RPD = Relative Percent Difference

A = First Sample Value
B = Second Sample Value (duplicate)

14.4. Matrix Spike Recovery

$$\%Recovery = \frac{SSR-SR}{SA} x100$$

Where:
SSR = Spike Sample Results
SR = Sample Result
SA = Spike Added from spiking mix

When the sample concentration is negative, use SR = 0 for the purpose of calculating spike recovery.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. See tables in section 11 & 13.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. See tables in section 11 & 13.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. METHOD PERFORMANCE

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 (or equivalent replacement), Method Detection Limit Studies for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) S-MN-Q-279 (or equivalent replacement), Training and Employee Orientation.

18.4. Periodic **performance evaluation (PE)** samples are analyzed periodically to demonstrate continuing competence per SOP S-MN-Q-258 (or equivalent replacement). Results are stored in the Quality office.

19. METHOD MODIFICATIONS

19.1. 10% H_2SO_4 is used to adjust the pH of each sample instead of 0.2N H_2SO_4 as described in standard method 3500 Cr-B section 4c.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. TROUBLESHOOTING

21.1. Refer to the Konelab Reference Manual – Program version 5.0, Manual version A. 5/6/2001 for troubleshooting techniques.

22. SAFETY

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. WASTE MANAGEMENT

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. POLLUTION PREVENTION

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. REFERENCES

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Standard Methods for the Examination of Water and Wastewater, 3500-Cr B (1997,2009, 2011).

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1. Attachment I – Test Definition for SM 3500 Cr B

27. REVISIONS

Document Number	Reason for Change	Date
S-MN-I-358-Rev.28	Table 10.2: Isopropanol and Phosphoric acid removed. Table 10.3: Volume adjusted Calibration curve information added 10% sulfuric acid added to working reagents.	7/22/16
S-MN-I-358-Rev.29	Updated to LLC throughout document 19.1. – Method modifications updated Attachment I updated	18Nov2016
S-MN-I-358-Rev.30	Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Removed extra empty row in Table 10.2. Updated Table 10.7 volume info for Diphenylcarbazide Solution. Replaced "1.0 g" with "0.25 g" for Solution Volume and "200 mL" with "50 mL" for Final Volume. Updated solution volume in section 10.7.1. to reflect changes made in Table 10.7. Removed "High Standard (HSTD) and Low Standard (LSTD)" reference from Table 11.1. Removed 11.2. Updated Frequency verbiage for MS/MSD in Table 13.1 from "maximum" to "minimum". Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Minor changes to space formatting. Updated reference 25.4. Removed "Online Addition,". Added "2011" to method year references. Updated page number references in Table of Contents.	13Mar2018

ATTACHMENT I – Test Definition for SM 3500 Cr B

Test definition AquaKem 6.5 Page: 1

SM3500CrB
Pace Analytical Services, Inc. MN
Analyst: KEO/JFP
Instrument: 10WET3

Date : 2016-11-17
Time : 11.06

Full name	HEXAVALENT CHROMIUM				
Online Name	SM 3500Cr B	Test In Use	YES		
Test type	Photometric		LOW	HIGH	
		Test limit	*	*	mg/l
Result unit	mg/l	Initial absorbance	*	*	A
Number of Decim.	3	Dilution limit	*	0.5000	mg/l
		Secondary dil 1+	0.0	1.0	
		Critical limit	*	*	mg/l
		Reflex test limit	*	*	mg/l
		Reflex test			

Acceptance	Manual	Reference class	LOW	HIGH	In Use
Dilution 1+	0.0				
Sample type	Water	Correction factor	1.00		
	Raw water	Correction bias	0.00	mg/l	
	Sewage				
Calibration type	Linear				
Repeat time (d)	90	Abs error (mA)		*	
Points/cal.	Single	Rel error (%)		*	
Acceptance	Manual				
Response limit (mA)	MIN	MAX			
	*	*			

ATTACHMENT I – (continued)

Bias correction in use		NO			
Cd reduction		NO			
Type of Calibrators		Series			
Calibrator		Conc.	Dil. ratio		
CR+6-0		0.000	1+0.0		
CR+6-5		0.500	1+99.0		
CR+6-5		0.500	1+49.0		
CR+6-5		0.500	1+9.0		
CR+6-5		0.500	1+4.0		
CR+6-5		0.500	1+1.0		
CR+6-5		0.500	1+0.0		
Manual QC in Use		YES	Routine QC in Use		YES
Acceptance		Manual	Interval	Requests	10
			Additional condition		NO
Control	Mean	SD	Control	Mean	SD
CR+6-CCB	0.00	0.01	CR+6-CCB	0.00	0.01
CR+6-CCV	0.20	0.02	CR+6-CCV	0.20	0.02
Rules in Use		1:1.0*SD	Rules in Use		1:1.0*SD
Blank		YES			
			Normal cuvette		
Sample			Volume (ul)		95
Disp. with		Extra	Add. Volume (ul)		40
Dilution with		Water	Wash reagent		Water

ATTACHMENT I – (continued)

```
==========================================================================
Test definition              AquaKem 6.5                        Page:    2

SM3500CrB                    Pace Analytical Services, Inc.  MN
                             Analyst:  KEO/JFP
                             Instrument: 10WET3
Date :   2016-11-17
Time :   11.06
--------------------------------------------------------------------------

Reagent                      CR+6 ACID    Volume (ul)            2
Disp. with                   Extra        Add. Volume (ul)       20
Wash reagent                 None

Measurement                  End point    Blank
Resp. Min(A)                 *            Resp. Max(A)           *

Reagent                      CR+6 COLOR   Volume (ul)            2
Disp. with                   Water        Add. Volume (ul)       1
Wash reagent                 None

Incubation                                Time (sec)             360

Measurement                  End point
Wavelength (nm)              540 nm       Side wavel. (nm)       None
Meas. type                   Fixed timing
```

Document Information

Document Number: ENV-SOP-MIN4-0129 **Revision:** 00

Document Title: Determination of Inorganic Anions by Ion Chromatography

Department(s): Wet Chemistry

Previous Document Number: S-MN-I-583-rev.08

Date Information

Effective Date: 04 Oct 2018

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Notes

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0129 **Revision:** 00

Title: Determination of Inorganic Anions by Ion Chromatography

All dates and times are in Central Time Zone.

Quick Approval

Approve Now

Name/Signature	Title	Date	Meaning/Reason
Jane Schur (JSCHUR)		04 Oct 2018, 01:37:24 PM	Approved

Pace Analytical Services, LLC
1700 Elm Street SE, Suite 200
Minneapolis, MN 55414

Phone: 612-607-1700
Fax: 612-607-6444

STANDARD OPERATING PROCEDURE

DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPH

Reference Methods: EPA 300.0/SW-846 Method 9056A

Local SOP Number:	S-MN-I-583-rev.08
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-583-rev.07

APPROVALS

Laboratory General Manager — Date: 27 Jun 2018

Laboratory Quality Manager — Date: 15 Jun 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-MN-I-583-Rev.08

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. This method is used to determine the concentration of the following inorganic anions by ion chromatography: Fluoride, Chloride, Bromide, Nitrite, Nitrate, and Sulfate by EPA Method 300.0 and SW-846 Method 9056A.

2. Summary of Method

2.1. Approximately 50 µL of sample is injected into an ion chromatograph system. The anions of interest are separated and measured using a system composed of a guard column, analytical column, suppressor device, and conductivity meter. Anions are identified based on their retention times as compared to known standards. Quantitation is accomplished by measuring the peak area comparing it to a calibration curve generated from known standards.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameters**: This SOP applies to Fluoride, Chloride, Bromide, Nitrite, Nitrate, and Sulfate.

4. Applicable Matrices

4.1. This SOP is applicable to drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, and reagent waters.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) is 0.05 mg/L for Fluoride, 0.08 mg/L for Bromide, 0.10 mg/L for Nitrate and Nitrite, 1.2 mg/L for Chloride and Sulfate. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large concentrations of one anion can interfere with the peak resolution of an adjacent anion. Sample dilution can be used to alleviate most interference problems.

6.2. The negative peak that elutes near the fluoride peak can usually be eliminated by the optional addition of the equivalent of 0.1 mL of concentrated eluent to 5 mL of each standard or sample.

6.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or to elevated baselines in ion chromatograms.

6.4. Samples must be filtered prior to injection to prevent damage to instrument columns and flow systems.

6.5. Known co-elution is caused by other small organic anions eluting in the area of fluoride causing interference.

6.6. The acetate anion elutes early during the chromatographic run, plus the retention times of the anions also seem to differ when large amounts of acetate are present. Avoid analysis of leachates (TCLP extracts), as acetic acid is used for pH adjustment.

6.7. Low molecular weight organic acids (formate, acetate, propionate, etc.) are conductive and co-elute with or near fluoride and can bias the fluoride quantitation in some drinking waters and most wastewaters.

6.8. Residual chlorine dioxide present in the sample will result in the formation of additional chlorite. If chlorine dioxide is suspected (chlorinated drinking water supplies), then purge the sample with nitrogen for 5 minutes.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Glass or plastic. A minimum of 50 mL is required to allow for filtering and duplication.	Unpreserved	Above freezing but below 6°C	Must be analyzed within 28 days from collection. If the analysis is to speculate Nitrate and Nitrite, samples must be analyzed within 48 hours of collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Ion Chromatograph	Metrohm 881 Compact IC Pro, 858 Professional Sample Processor, 800 Dosino for in-line and automatic dilution, A Supp 5 column and Metrohm Suppressor Module (MSM)	Metrohm, or equivalent. See Metrohm Product Catalog for respective part numbers
Sample loop	20 uL	Metrohm part #6.1825.210
H_2O scrubber		Metrohm part # 6.2837.010
CO_2 scrubber		Metrohm part # 6.2837.000
Peristaltic pump tubing (sampler)	Yellow-yellow (1.42 mm I.D.) Black-black (0.76 mm I.D.)	Yellow-yellow: Fisher Part #14-190-509 Black-black: Fisher Part #14-190-504
Peristaltic pump tubing (MSM)	Orange-yellow (0.51 mm I.D.)	Environmental Express Part #PT0020SAN
Ultrafiltration disks	47 mm diameter, 0.2 µm thickness	Metrohm part # 6.2714.020
In-line filtration disks		Metrohm part # 6.2821.130
Column: Metrosep A Supp 5 – 100/4.0		Metrohm part # 6.1006.510
Sample vials	15 mL plastic tubes	Fisher Part #NC1328469
MagIC Net 2.2 software	Used to analyze, integrate and export data from IC	Metrohm
Data transmission software	LIMSLink	See master list for current version
Data reporting software	Horizon	See master list for current version

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
E-pure DI water		Verify daily that pH and conductivity are within acceptance limits
Sodium Carbonate (Na_2CO_3)	Anhydrous. Powder. Store at room temperature. Expires per manufacturer's specifications.	Fisher part # S263-500
Sodium Bicarbonate	Powder. Store at room temperature. Expires per	Fisher part # S233-500

($NaHCO_3$)	manufacturer's specifications.	
Methanol	Store at room temperature. Expires per manufacturer's specifications.	Fisher part # A454-4
Sulfuric Acid, Optima (H_2SO_4)	Concentrated. Store at room temperature. Expires per manufacturer's specifications.	Fisher part # A468-500
Phosphoric Acid (H_3PO_4)	85%. Store at room temperature. Expires per manufacturer's specifications.	Fisher part #A365-1
Oxalic Acid Dihydrate	Store at room temperature. Expires per manufacturer's specifications.	Fisher part # 423152500
Dipicolinic Acid (2,6-Pyridinedicarboxylic Acid)	Store at room temperature. Expires per manufacturer's specifications.	Fisher part # AC131881000
Acetone	Store at room temperature. Expires per manufacturer's specifications.	Fisher part # A929-4
Ascarite	Store at room temperature. Expires per manufacturer's specifications.	Fisher part # AC208091000
Bromide Standard	1000 mg Br/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 046 Inorganic Ventures part # ICBR1-1
Chloride Standard	1000 mg Cl/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 988 Inorganic Ventures part # ICCL1-1
Fluoride Standard	1000 mg F/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 989 Inorganic Ventures part # ICF1-1
Nitrate Standard	1000 mg NO_2-N/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 991 Inorganic Ventures part # ICNNO31-1
Nitrite Standard	1000 mg NO_3-N/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 990 Inorganic Ventures part # ICNNO21-1
Sulfate Standard	1000 mg SO_4/L. Purchase premade. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 995 Inorganic Ventures part # ICSO41-1
A Supp 5 Eluent 20x	64 mM Sodium Carbonate, 20 mM Sodium Bicarbonate. Purchase premade or prepare as outlined in table 10.2. Store at room temperature. Expires per manufacturer's specifications.	Metrohm part # REAIC1101
Suppressor Rinse Solution	DI water with 0.1 % methanol. Purchase premade or prepare as outlined in table 10.2. Store at room temperature. Expires per manufacturer's specifications.	Metrohm part # REAIC1150C
Suppressor Cleaning Solution	1 M Sulfuric Acid & 0.1 Oxalic Acid in 5% Acetone. Purchase premade or prepare as outlined in table 10.2. Store at room temperature. Expires per manufacturer's specifications.	Metrohm part # REAIC1190
Suppressor Regenerant Solution	0.1 M Phosphoric Acid. Purchase premade or prepare as outlined in table 10.2. Store at room temperature. Expires per manufacturer's specifications.	
Calibration Solution (CAL6)	Bromide – 8.0 mg/L Chloride – 100 mg/L Fluoride – 4.0 mg/L Nitrate as N – 8.0 mg/L Nitrite as N – 8.0 mg/L Sulfate – 100 mg/L Purchase premade or prepare as outlined in table	Metrohm part # REAIC1028

	10.2. Store at 4±2°C. Expires per manufacturer's specifications.	
Initial & Continuing Calibration Verification (ICV/CCV) Solution	Bromide – 1.0 mg/L Chloride – 12.5 mg/L Fluoride – 1.0 mg/L Nitrate as N – 1.0 mg/L Nitrite as N – 1.0 mg/L Sulfate – 12.5 mg/L Purchase premade or prepare as outlined in table 10.2. Store at 4±2°C. Expires per manufacturer's specifications.	Metrohm part # REAIC1029
Spiking Solution	Bromide – 10.0 mg/L Chloride – 125 mg/L Fluoride – 10.0 mg/L Nitrate as N – 10.0 mg/L Nitrite as N – 10.0 mg/L Sulfate – 125 mg/L Purchase premade or prepare as outlined in table 10.2. The system automatically prepares the spike at a 10x dilution in the selected parent sample. Store at 4±2°C. Expires per manufacturer's specifications.	ERA part # 092 (custom standard)

10.2. Table 10.2 – Working Standards and Reagents

Note: The system can be programmed to automatically make the necessary dilutions.

Solution	Reagent(s) Used	Reagent(s) Amount	Solvent	Final Solution Volume	Final Concentration
A Supp 5 Eluent 20x	Na_2CO_3	6.78 g	DI Water	1000 mL	64 mM Na_2CO_3 20 mM $NaHCO_3$
	$NaHCO_3$	1.68 g			
Suppressor Rinse Solution	Methanol	1 mL	DI Water	1000 mL	0.1% Methanol
Suppressor Regenerant Solution	H_3PO_4	6.8 mL	DI Water	1000 mL	0.1 M H_3PO_4
	Dipicolinic Acid	0.167 g			
Suppressor Cleaning Solution	H_2SO_4	53 mL	DI Water	1000 mL	1 M H_2SO_4 0.1 M Oxalic Acid 5% Acetone
	Oxalic Acid	12.6 g			
	Acetone	50 mL			
Calibration Solution (CAL6)	Fluoride Standard	0.1 mL	DI Water	50 mL	2.0 mg F/L 100 mg Cl/L 8.0 mg NO_2-N/L 8.0 mg Br/L 8.0 mg NO_3-N/L 100 mg SO_4/L
	Chloride Standard	5 mL			
	Nitrite Standard	0.4 mL			
	Bromide Standard	0.4 mL			
	Nitrate Standard	0.4 mL			
	Sulfate Standard	5 mL			
CAL5	CAL6	5.0 mL	DI Water	10 mL	1.0 mg F/L 50 mg Cl/L 4.0 mg NO_2-N/L 4.0 mg Br/L 4.0 mg NO_3-N/L 50 mg SO_4/L
CAL4	CAL6	2.5 mL	DI Water	10 mL	0.50 mg F/L 25 mg Cl/L 2.0 mg NO_2-N/L 2.0 mg Br/L 2.0 mg NO_3-N/L 25 mg SO_4/L
CAL3	CAL6	0.50 mL	DI Water	10 mL	0.10 mg F/L 5.0 mg Cl/L

					0.40 mg NO_2-N/L 0.40 mg Br/L 0.40 mg NO_3-N/L 5.0 mg SO_4/L
CAL2	CAL6	1.0 mL	DI Water	50 mL	0.040 mg F/L 2.0 mg Cl/L 0.16 mg NO_2-N/L 0.16 mg Br/L 0.16 mg NO_3-N/L 2.0 mg SO_4/L
CAL1	CAL6	0.50 mL	DI Water	50 mL	0.020 mg F/L 1.0 mg Cl/L 0.080 mg NO_2-N/L 0.080 mg Br/L 0.080 mg NO_3-N/L 1.0 mg SO_4/L
ICV/CCV Solution	Fluoride Standard	0.2 mL	DI Water	200 mL	1.0 mg F/L 12.5 mg Cl/L 1.0 mg NO_2-N/L 1.0 mg Br/L 1.0 mg NO_3-N/L 12.5 mg SO_4/L
	Chloride Standard	2.5 mL			
	Nitrite Standard	0.2 mL			
	Bromide Standard	0.2 mL			
	Nitrate Standard	0.2 mL			
	Sulfate Standard	2.5 mL			
Spiking Solution	Fluoride Standard	0.5 mL	DI Water	50 mL	10.0 mg F/L 125 mg Cl/L 10.0 mg NO_2-N/L 10.0 mg Br/L 10.0 mg NO_3-N/L 125 mg SO_4/L
	Chloride Standard	6.25 mL			
	Nitrite Standard	0.5 mL			
	Bromide Standard	0.5 mL			
	Nitrate Standard	0.5 mL			
	Sulfate Standard	6.25mL			
ICV/CCV 2 Solution	Fluoride Standard	0.15 mL	DI Water	100 mL	1.5 mg F/L
	Chloride Standard	5.0 mL	DI Water		50 mg Cl/L
	Nitrite Standard	0.4 mL	DI Water		4.0 mg NO_2-N/L
	Bromide Standard	0.4 mL	DI Water		4.0 mg Br/L
	Nitrate Standard	0.4 mL	DI Water		4.0 mg NO_3-N/L
	Sulfate Standard	5.0 mL	DI Water		50 mg SO_4/L

10.3. A Supp 5 Eluent 20x. Add 6.78 g Na_2CO_3 and 1.68 g $NaHCO_3$ to ~500 mL DI water in a 1 L volumetric flask and dilute to mark. Use a stir bar and a magnetic stirring plate to fully dissolve the solids and degas the water for 20-30 minutes. Store in a glass container at room temperature. Expires in six months.

10.4. Suppressor Rinse Solution & Suppressor Regenerant Solution. Add respective reagents to ~500 mL DI water in a 1 L volumetric flask. Dilute to mark and mix thoroughly. Store in a glass container at room temperature. Expires in six months.

10.5. Supressor Cleaning Solution. Add 53 mL H_2SO_4 to ~500 mL DI water in a 1 L volumetric flask. Add 12.6 g oxalic acid and 50 mL acetone. Dilute to mark and mix thoroughly, making sure the oxalic acid is fully dissolved. Store in a glass container at room temperature. Expires in six months.

10.6. Calibration Solution (CAL6). Spike the respective amount of each anion standard into ~25 mL DI water in a class A 50 mL volumetric flask. Dilute to mark and mix thoroughly. Expires in 24 hours; alternatively, the solution expires in 7 days if nitrite is not included or evaluated. **Note: Be sure to use different lots for each standard than that which is used for the ICV/CCV Solution and the Spiking Solution.**

10.7. ICV/CCV Solution. Spike the respective amount of each anion standard into ~100 mL DI water in a class A 200 mL volumetric flask. Dilute to mark and mix thoroughly. Expires in 24 hours; alternatively, the solution expires in 7 days if nitrite is not included or evaluated. **Note: Be sure to use different lots for each standard than that which is used for the Calibration Solution (CAL6).**

10.8. Spiking Solution. Spike the respective amount of each anion standard into ~25 mL DI water in a class A 50 mL volumetric flask. Dilute to mark and mix thoroughly. Expires in 24 hours; alternatively, the solution expires in 7 days if nitrite is not included or evaluated. **Note: Be sure to use different lots for each standard than that which is used for the Calibration Solution (CAL6).**

11. Calibration and Standardization

11.1. Table 11.1 – Calibration, Verification and Acceptance Criteria

Calibration Metric	Parameter/Frequency	Criteria	Comments
Calibration Curve Fit	Quadratic fit or linear regression. Curve is valid for 90 days but must be confirmed every 30 days with a standard at or below the reporting limit.	$r \geq 0.990$ for each analyte	If criteria are not met, remake the calibration standards and recalibrate the instrument. A quadratic fit is allowed per the Federal Register (see 19.1 and 25.7); a minimum of six points must be included in the calibration. If linear regression is used, a minimum of three calibration points and a blank is required. Manufacturer recommends $r \geq 0.9990$ for each analyte with a relative standard deviation (RSD) of 5% or less. This is not required.
Initial & Continuing Calibration Verification (ICV/CCV)	ICV must be performed immediately after calibration. A CCV must be analyzed every 10 samples and at the end of the analytical sequence. For WI-originating samples, if a quadratic curve is utilized two CCVs have to be analyzed at differing concentrations at the inflection points.	90-110% of the true value	When measurements exceed the acceptance criteria, all analytical samples since the last compliant CCV must be reanalyzed. If the ICV or two consecutive CCVs exceed the control limits, the analysis must be terminated, the problem corrected and the system recalibrated.
Monthly Calibration Verification	Once 30 days after calibration and once 60 days after calibration. The true value must be equal to or less than the reporting limit for each analyte.	60-140% of the true value	If the initial calibration is utilized for greater than 30 days, the reporting limit must be verified every 30 days at the same criteria. If criteria are not met, the reporting limit must be raised to the next standard that meets the criteria until a new calibration has been performed per Minnesota Department of Health (MDH) rules. When necessary, make manual adjustments to the integration in order to obtain the most accurate peak areas. Include all documentation, including a before and after, of any manual integration.
Linear Calibration Range (LCR)	Must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected.	90-110% of the true value	Verify the linear calibration range every six months by analyzing a blank and three standards quantitated against the calibration curve. Following each calibration, analyze a blank and calibration standards 4, 5, and 6. If standards do not meet 90-110% criteria for all analytes, system should be

			recalibrated.
Initial & Continuing Calibration Blank (ICB/CCB)	Analyzed after every ICV/CCV	The absolute value must be less than the reporting limit for each analyte. Per QAPP or client specifications, alternate criteria such as evaluating to ½ RL may apply.	If a CCB exceeds the RL, reanalyze all samples since the last compliant CCB. If the ICB or two consecutive CCBs exceed the control limits, the analysis must be terminated, the problem corrected and the system recalibrated. ***Exception:*** If all the affected samples are non-detect or at least 10x the concentration of the failing result, the sample data may be accepted.
High Check	Required by client-specific tech specs. Must be performed on the same day that client's samples are analyzed. The high check must be above the mid-point of the calibration curve. Utilize CAL6 or ICV/CCV2 Solution.	90-110% of the true value	If either/or check is outside the acceptance criteria, remake the respective check standard and re-analyze. If the reanalysis is outside the acceptance criteria, the instrument must be re-calibrated prior to the analysis of any samples that require this high check.
ICV/CCV 2	Required by WI DNR. If using a quadratic regression for any analytes, must be performed during analysis of any samples of Wisconsin origin for those analytes. Analyze immediately after each ICV/CCV. The analytes in the ICV/CCV 2 should be close to or at the inflection point. Utilize ICV/CCV 2 Solution.	90-110% of the true value	If either/or check is outside the acceptance criteria, remake the respective check standard and re-analyze. If the reanalysis is outside the acceptance criteria, the instrument must be re-calibrated prior to the analysis of any samples of Wisconsin origin.

11.2. Generating a Calibration Curve

11.2.1. Turn the IC on and open the MagIC Net 2.3 software. Click Start HW in the Equilibration tab to allow the system to stabilize and the column heater to come up to temperature (35.0°C). This takes approximately 30 minutes.

11.2.2. Prepare the calibration solution (CAL6) using the volumes found in table 10.2. Alternatively, use the premade calibration stock solution purchased through Metrohm.

11.2.3. Pour an aliquot of calibration solution into a vial and load in the rack. Load five additional empty vials to accommodate dilutions made by the system.

11.2.4. Enter the calibration standards in the determination series of the MagIC software as CAL1, CAL2, CAL3, CAL4, CAL5 and CAL6. The sample type for each standard must be Standard 1, Standard 2, Standard 3, Standard 4, Standard 5 and Standard 6, respectively. CAL6 is the calibration standard made in table 10.1 and CAL1 through CAL5 are dilutions prepared from CAL6 by the system. The dilutions are as follows:

CAL1 = 100x dilution
CAL2 = 50x dilution
CAL3 = 20x dilution
CAL4 = 4x dilution
CAL5 = 2x dilution
CAL6 = 1x dilution

Alternatively, CAL1 through CAL5 may be manually prepared by diluting the CAL6 in class A volumetric flasks. See table 10.1 for instructions for manual dilutions.

11.2.5. After the calibration standards have been analyzed, analyze a CCV and CCB (Note: This will be changed to ICV/ICB during evaluation of the calibration). Once complete, click on Database in the MagIC Net software.

11.2.6. Highlight the six calibration standards and the CCV and CCB, right click and select Reprocess…

11.2.7. In the reprocessing table, change the name of the CCV and CCB to ICV and ICB, respectively.

11.2.8. Click on CAL6 and update the retention time for each anion peak. Click Update once this is complete.

11.2.9. With CAL6 still highlighted, click on Reprocessing. This will open a small window prompting you to reprocess the calibration. Select From standards of reprocessing table and Keep manual integration and click OK.

11.2.10. With CAL6 still highlighted, click on Reprocessing again. This time, select From selected determination and Keep manual integration and click OK.

11.2.11. Next, click Method… and then Save as…

11.2.12. Select the method being used for analysis: EPA 300.0 Anions. Save, noting the day's date and analyst initials.

Note: The method name in the instrument specifies EPA 300.0 Anions although the analysis is also applicable to SW-846 Method 9056A.

11.2.13. Now select OK in the lower right hand corner of the reprocessing table window. When prompted, select Calibration Changed and note the day's date and analyst initials. It may take several minutes for the database to update the calibration information.

11.2.14. To report the calibration curve to a Q batch, follow the sample reporting steps in section 12.2.

12. Procedure

12.1. Sample Prep

12.1.1 If a sample contains a considerable amount of sediment or other solids, filter the sample through a 0.45 µm membrane filter. Filter the associated method blank and LCS and document which samples were filtered on the raw data run log as well as the lot number of the filter used.

12.2. Sample Analysis

12.2.1. Batch up samples in Horizon and allow them to come to room temperature before analysis.

12.2.2. Turn the IC on and open the MagIC Net 2.3 software. Click Start HW in the Equilibration tab to allow the system to stabilize and the column heater to come up to temperature (35.0°C). This takes approximately 30 minutes.

12.2.3. Open LimsLink and click on Get Samples to create a prep run. Pull in the batch information and select Import Instrument and then Export to Metrohm.

12.2.4. In MagIC, go to the Determination series tab and click on Sample table. Select Import Data and then select IC.csv. The run sequence will be generated and fill out all blank cells other than those titled Info.

12.2.5. Begin the sequence by analyzing a CCV and CCB if they have not been performed within the last six hours. The sequence may be edited even after the analysis has begun. Therefore, samples may be added on to the end of a run, the order that samples are run may be altered or dilutions can be added and changed as the run progresses.

12.2.6. Critically evaluate all chromatograms in the Database and determine if any require manual integration or further dilution. Manual integration should be avoided if at all possible. In the event of peak shifts due to sample matrix, retention times for individual analytes may need to be adjusted. If manual integration or retention time adjustment are used, both before and after chromatograms should be included in the data package along with a memo stating that the

chromatogram was manually integrated. If extensive manual integrations or retention time adjustments are being performed, instrument maintenance or recalibration may be necessary.

12.2.7. If doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification must be used.

12.3. Reporting Data

12.3.1. In the Database, highlight all analytical points pertinent to the batch being reported.

12.3.2. Open Determinations in the toolbar at the top of the window and select Export… Makes sure the selection specifies All selected data records and that the export template is set to LimsLink Export. Select OK.

12.3.3. Open the folder C:/Limslink Data/Test Data and find the file named TEST.csv. Rename this to the WETA batch number and close the folder.

12.3.4. Open LimsLink and create a new run under IC_QBATCH.

12.3.5. Open Import Instrument, select 10WT61 and scroll down to find the batch being reported. Select the batch to populate the spreadsheet.

12.3.6. Click on Options and Import / Export Data and click on Set Standard IDs. Once prompted, enter the Q batch number of the most current calibration.

12.3.7. Select the appropriate standard IDs for the CCV and CCBs used in the run.

12.3.7.1. When posting initial calibration data to a Q batch, select the appropriate standard IDs for CAL1 through CAL6 as well as the ICV and ICB.

12.3.8. Click on Options and Import / Export Data again and select Get LIMS Info. Click Yes when prompted to use the entire run.

12.3.9. If any samples are reporting analytes at different dilutions, right click the appropriate sample cell and delete the X from the Use Element column in order to exclude the result from posted.

12.3.10. Highlight all the samples being reported, open Options and Import / Export Data and select Export Run to Epic Pro.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI Water	Analyzed once per batch of up to 20 samples.	The absolute value must be less than the reporting limit for each analyte. Per QAPP or client specifications, alternate criteria such as evaluating to ½ RL may apply.	If outside the acceptance criteria, reanalyze. If reanalysis still does not pass, terminate analysis and reject all batch data associated with the failing MB. ***Exception:*** If all batch samples are non-detect or at least 10x the concentration of the failing result, the sample data may be accepted.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	1 mL of Spiking Solution : 9 mL DI Water	An LCS must be analyzed once per batch of up to 20 samples. An LCSD must be performed quarterly to generate data points to determine a precision limit for the laboratory.	90-110% of the true value Until sufficient data points (>20 points) have been generated, ≤ 20 % RPD will be utilized for criteria.	If LCS recovery is not within the criteria, reanalyze. If reanalysis does not pass, terminate analysis and reject all batch data associated with the failing LCS. The system may need to be recalibrated and/or require maintenance.

Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	1 mL of Spiking Solution : 9 mL of client-provided sample	A pair of MS/MSD must be analyzed once every ten samples or once per batch, whichever is more frequent.	90-110% of the true value RPD ≤ 20%	If the MS recovery is not within the criteria, and the LCS is shown to be in control, the recovery problem is judged to be matrix related and the results may be accepted. If the concentration of matrix spike is less than 25% of the background concentration of the matrix, the matrix spike recovery should not be calculated. For Minnesota Admin Contract Clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.

14. Data Analysis and Calculations

14.1. All calculations are done internally within the MagIC NET 2.3 software.

14.2. Spike sample recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA}$$

Where:
SSR = Spike sample result, mg/L
SR = Sample result, mg/L
SA = Spike added, mg/L

14.3. The relative percent difference (RPD) is calculated as:

$$RPD = \frac{[|S-D| \times 100]}{(S+D)/2}$$

Where:
S = Sample value, mg/L
D = Duplicate sample value, mg/L

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 & 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted every six months or when a new primary operator begins work (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. The calibration curve may be fitted using a quadratic function in lieu of the linear regression model. In such a case, the minimum number of calibration points should be six, and in no case should concentrations be extrapolated for instrument responses that exceed that of the most concentrated calibration point. Ref. 25.7

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.2. Routine Maintenance – Refer to Metrohm USA IC User Guide (25.6.)

20.2.1. Weekly

20.2.2. The ultra-filtration membrane filter disk should be inspected for sediment build-up and algal growth. In this event, it should be changed and the acrylic apparatus holding it together be thoroughly rinsed and cleaned with E-pure DI water.

20.2.3. The water scrubber should be changed (beads changed and baked out) and the carbon dioxide scrubber should be checked and changed as needed.

20.2.4. On a bi-weekly schedule, flows should be checked through the autosampler pump tubes and through the suppressor acid and water pump tubes. If not to the manufacturer's specification, replace any pump tubing as needed.

20.2.5. The remainder of the maintenance is to be performed on either a monthly, quarterly or annual basis. The annual maintenance is covered by a maintenance contract and performed as part of the annual preventative maintenance. All other maintenance is performed by the analyst. The frequency of maintenance is summarized below:

20.3. Table 20.1 Instrument Maintenance and Frequency

Maintenance	Frequency
Change suppressor rinse solution	As needed
Change suppressor regenerant solution	As needed
Triple rinse the dilution water reservoir and change water	As needed
Change A Supp 5 Eluent 20x	As needed
Replace the ultra-filtration disk, cleaned disk apparatus	Weekly or as needed
Replace the water scrubber and/or CO2 scrubber	As needed
Change sampler and MSM pump tubing	As needed
Replace the RP guard disk and filter	Quarterly or as needed
Triple rinse the eluent water reservoir and change water	Weekly or as needed
Replace the 3 inline filters	Quarterly or as needed
Change the trap on top of the eluent (Replace ascarite)	Semi-Annually
Change the eluent aspirating filter	As needed
Replace check valves	Annually
Replace piston seals	Annually
Replace sapphire support rings	Annually
Replace sample waste lines	Annually
Clean pump rollers with deionized water	Annually

21. Troubleshooting

21.1. Refer to the Metrohm operators' manual for troubleshooting techniques.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, Method 300.0, Revision 2.1, August 1993.

25.5. Metrohm USA Operators Manual - stored on desktop of instrument computer

25.6. Federal Register/Vol. 77, No. 96/Friday, May 18, 2012/Rules and Regulations – Page 54, section (x.)

25.7. U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Anions by Ion Chromatography, Revision 1, February 2007, SW-846 Method 9056A.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-583-rev.07	Removed uncontrolled. Added "Copies without a distribution number below are considered uncontrolled." to the statement of copyright. Added Phosphoric Acid and Dipicolinic Acid rows to Table 10.1. Edited Suppressor Regenerant Solution row in Table 10.1 to Phosphoric acid from Sulfuric acid. Edited Suppressor Regenerant Solution row in Table 10.2 to the new reagents in Table 10.1. Edited the Comments in Table 11.1 Linear Calibration Range (LCR) row, adding "Following each calibration, analyze a blank…" Added the following to Section 12.2.6 "In the event of peak shifts due to sample matrix, retention times for individual analytes may need to be adjusted," "or retention time adjustment," and "If extensive manual integrations or retention time adjustments are being performed, instrument maintenance or recalibration may be necessary." Added "or when a new primary operator begins work" to Section 18.2	07Aug2017
S-MN-I-583-rev.08	Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Table 9.1 – Updated Vendor/Item # column for Peristaltic pump tubing (sampler) and (MSM) and Sample vials, updated Description from 11 to 15 mL plastic tubes for sample vials Table 11.1 – added "or below" to Calibration Curve Fit row, Parameter column	11Jun2018

CHLORINATED ACID HERBICIDES BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

BASED ON SW846 METHOD 8151

1 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is a capillary GC/MS method used to determine the concentration of various halogenated acid herbicides and related compounds in aqueous and soil matrices. Specifically, this method has been validated to determine the following compounds:

Compound	CAS Number
2,4-D[1]	94-75-7
2,4-DB[1]	94-82-6
2,4,5-T[1]	93-76-5
2,4,5-TP (Silvex)[1]	93-72-1
Acifluorfen	50594-66-6
Bentazon[1]	25057-89-0
Bromoxynil	1689-84-5
Chloramben	133-90-4
Clopyralid	1702-17-6
Dacthal	2136-79-0
Dalapon	75-99-0
Dicamba[1]	1918-00-9
Dichlorprop	120-36-5
Diclofop Acid	40843-25-2
Dinoseb	88-85-7
MCPA[1]	94-74-6
MCPB	94-81-5
MCPP	7085-19-0
4-Nitrophenol	100-02-07
Pentachlorophenol	87-86-5
Picloram[1]	1918-02-01
Triclopyr[1]	55335-06-3

[1] Minnesota Department of Agriculture target compound

1.2 The analytes include several classes of compounds with phenoxy acid or phenol backbones used primarily as agricultural herbicides. Most compounds are applied in many forms (i.e. free acid, phenol, ester or salts.) These forms are converted to and reported as the total of the free acid or phenol.

1.3 Compound identification is done by retention time and mass spectra of the methyl ester derivative of each individual reference compound compared to each derivative compound in unknown samples. Each compound has a primary quantitation ion with one or more secondary identification ions.

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1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of HP GC/MS systems. The analyst must understand spectra generated by the MS and how to use the spectra for identification and quantitation.

2 SUMMARY OF METHOD

2.1 This method provides hydrolysis, extraction, derivatization and GC/MS conditions for the analysis of halogenated/nitro acid herbicides/phenols in water and soil samples.

2.2 Aqueous samples are hydrolyzed at pH ≥ 13, made acidic to pH ≤ 2 and the herbicide acids/phenols are extracted with ethyl ether. Soil samples are extracted using methanol, water, and sodium hydroxide on a shaker table extraction apparatus. The soil extract is transferred to a separatory funnel. The pH is adjusted to pH ≤ 3, and extracted with ethyl ether. The resulting water and soil extracts are concentrated, derivatized with diazomethane, and made to volume with hexane.

2.3 A measured amount of the extract is transferred to an auto-sampler vial containing internal standard and analyzed by GC/MS. Calibration is accomplished by using an internal standard method, comparing the response of a primary characteristic (quantitation) ion relative to an internal standard using a multi-point standard curve.

2.4 Quantitation is accomplished by reverse extrapolation obtaining an equivalent µg/mL or ng/mL concentration of the free acid form in the extract.

2.5 Final reported results are calculated by the ECCS LIMS system.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

3.2 The term lab pure is defined as a method specific control standard, consisting of an aliquot of the parent acid forms, that is not extracted but is derivatized. This control standard is used to monitor the effectiveness of the derivatization process only.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in total ion chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by analyzing reagent blanks.

4.1.1 Glassware must be scrupulously cleaned. Refer to the GEN-006, Glassware Washing SOP for additional information.

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4.1.2 Glassware used to make final volume or a lab pure is additionally acid washed to remove active sites from the glassware that may trap target analytes before their final transfer to a 12 mL amber glass storage vial.

4.1.3 High purity solvents and reagents help to minimize interference problems.

4.2 Non-target organic acids/phenols cause the most direct interference with the methylation process and quantitation by the GC/MS.

4.3 The herbicides, being strong organic acids, are readily adsorbed to alkaline substances and may be lost during analysis. Therefore, sodium sulfate must be acidified prior to use.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure. Lab coats are recommended.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

5.5 Diazomethane is a carcinogen and an explosive. Once made the reagent is stored in a freezer minimizing volatility. Since diazomethane is volatile, always work in a hood with the reagent. To be considered explosive, the solution in ethyl ether would have to be concentrated at least 10 fold.

6 APPARATUS AND MATERIALS

6.1 Instrumentation

6.1.1 HP 5890 Gas Chromatograph with split/splitless injector or equivalent

6.1.2 HP 5972 Mass Selective Detector or equivalent

6.1.3 Leap Technologies CTC AS-200 Auto-sampler or equivalent

6.2 Computer Hardware

6.2.1 HP Compaq DC7600 or equivalent

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6.3 Computer Software

6.3.1 Microsoft Windows 2000 operating system or equivalent current version

6.3.2 Microsoft Office 2007 or equivalent current version

6.3.3 Agilent MSD Productivity Chemstation with EnviroQuant

6.3.4 Promium Element Data System

6.4 GC Supplies List:

6.4.1 Restek RTX-200MS, 30 m x 0.32 mm ID, 0.25 um film thickness capillary column or equivalent. Restek Cat # 15624

6.4.2 Restek Rxi®Gaurd, 5 m x 0.32 mm ID guard column or equivalent. Restek Cat # 10039

6.4.3 Dual Vespel ring inlet seals, stainless steel or gold plated. Restek Cat # 21239

6.4.4 LB-2, 11 mm septa. Supelco Cat # 164742

6.4.5 1/16 x 0.5 mm Vespel/graphite ferrules. Restek Cat #'s 20231 & 20249

6.4.6 Single gooseneck splitless inlet liners, 4 x 6.5 x 78.5 mm. Restek Cat # 20799

6.5 Zymark Turbovap Evaporation System

6.5.1 Concentrator tubes - 200 mL capacity

6.6 Balances

6.6.1 Top loader - capable of weighing to 0.01 g

6.6.2 Analytical - capable of weighing to 0.0001 g

6.7 Vials

6.7.1 12 mL amber storage vials with Teflon® lined screw cap

6.7.2 2 mL amber auto-sampler vials and PTFE/silicone aluminum seals

6.7.3 20 mL glass scintillation vials with polypropylene caps

6.7.4 40 and/or 60 mL amber or clear VOA vials with Teflon® lined screw caps

6.8 Syringes: Various sizes, 10-1000 µL, Gastight

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6.9 Air displacement pipette, 200-1000 µL variable, and 1 mL tips (Eppendorf or equivalent)

6.10 Repeater Plus with Combitips of various sizes (Eppendorf or equivalent)

6.11 Glass funnels: 40 - 60 mL capacity

6.12 Glass disposable transfer pipettes - 5 3/4" and 9", with pipette bulbs

6.13 Assorted laboratory glassware - beakers, volumetric flasks, pipettes and graduated cylinders

6.14 Stainless steel spatulas

6.15 Separatory funnels: 2 L, 1 L, 500 mL and 250 mL with Teflon® stopcock

6.16 Optifix solvent dispensers capable of 10 mL and 50 mL volume (EM Science or equivalent)

6.17 Wide range pH Indicator paper, 1 to 14

6.18 Ovens

6.18.1 Drying oven capable of maintaining 105 °C

6.18.2 Muffle oven capable of maintaining 400 °C

6.19 Compressed gas - helium

6.20 Refrigerator capable of maintaining 4 °C

6.21 Freezer capable of maintaining temperatures below -15 °C

6.22 Erlenmeyer flasks: assorted sizes

6.23 Diazomethane generator kit, Aldrich: See Figure 2.

6.24 Shaker table

6.25 Centrifuge capable of 2000 RPM and capacity to hold 40 mL VOA vials

7 REAGENTS AND STANDARDS

7.1 Solvents

7.1.1 Dichloromethane, CH_2Cl_2 - pesticide quality or equivalent

7.1.2 Acetone, CH_3COCH_3 - pesticide quality or equivalent

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7.1.3 Iso-octane, C_8H_{18} – pesticide quality or equivalent

7.1.4 Soil extraction solvent – 500:450:50; Methanol:H_2O:10N NaOH,

7.1.4.1 Combine the following in a 1 L reagent bottle: 500 mL of methanol, 450 mL of DI water and 50 mL of 10N NaOH, mix well and assign an expiration date of 1 month. Store at room temperature.

7.1.5 DI water or finished treated DI water

7.1.6 Hexane, C_6H_{14} – pesticide quality or equivalent

7.1.7 Diethyl ether, $C_2H_5OC_2H_5$- Mallinckrodt Nanograde 3434 or equivalent, no preservatives added

7.1.8 Methanol, CH_3OH – pesticide quality or equivalent

7.1.9 Methyl-tert-butyl ether (MTBE) – pesticide quality or equivalent

7.2 Solid Reagents

7.2.1 Sodium chloride (NaCl) - reagent grade

7.2.1.1 Sodium chloride is baked in a muffle furnace at 400 °C overnight prior to use. Baked sodium chloride is stored in sealed glass containers prior to use.

7.2.2 Sodium sulfate (Na_2SO_4) – reagent grade

7.2.2.1 Sodium sulfate is baked in a muffle furnace at 400 °C overnight prior to use. Baked sodium sulfate is stored in sealed glass containers prior to use.

7.2.3 Sand

7.2.3.1 Sand is baked in a muffle furnace at 400 °C overnight prior to use. Baked sand is stored in sealed glass containers prior to use.

7.2.4 Acidified sodium sulfate, Na_2SO_4

7.2.4.1 Standard method

7.2.4.1.1 Prepare by adding enough diethyl ether to just cover baked sodium sulfate in a sealable container. Then add a minimum of 0.1 mL concentrated sulfuric acid per 100 g of sodium sulfate. Mix well then transfer the slurry to a shallow Pyrex dish and evaporate the diethyl ether in a hood overnight.

7.2.4.1.2 Mix 1 g acidified sodium sulfate with 5 mL water and measure the pH of the mixture. The pH must be below 4.

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7.2.4.1.3 If the pH is greater than 4, re-slurry and add another 0.1 mL of sulfuric acid per 100 g of sodium sulfate. Mix well, then repeat 7.2.4.1.1 and 7.2.4.1.2.

7.2.4.1.4 Activate the acidified sodium sulfate at 130 °C overnight prior to use.

CAUTION: Do not place the acidified sodium sulfate into the oven until the ether has evaporated in the hood and the acidified sodium sulfate is a free flowing powder.

7.2.4.1.5 Transfer to sealed labeled glass jars for use. No expiration date if stored in a sealed jar.

NOTE: Exposure to atmospheric moisture will eventually reduce the effectiveness of the acidified sodium sulfate. If this is suspected, re-activate at 130 °C overnight.

7.2.4.2 Alternate method

7.2.4.2.1 In a clean 7" x 11" Pyrex dish cover the base with approximately 1" of sodium sulfate.

7.2.4.2.2 Cover the sodium sulfate with ethyl ether and add about 2 mL concentrated sulfuric acid.

7.2.4.2.3 Mix thoroughly with a spatula or other mechanical means.

7.2.4.2.4 Leave in hood overnight to allow ethyl ether to evaporate.

7.2.4.2.5 Test acidified sodium sulfate by mixing 1 g in 5 mL of water.

7.2.4.2.6 Measure the pH with wide range pH paper. If the pH is not 4 or below, repeat steps 7.2.4.2.2 to 7.2.4.2.5.

7.2.4.2.7 Once the pH is 4 or less and solvent has been evaporated, place the dish in the oven at about 130 °C overnight to activate.

CAUTION: Do not place the tray into the oven until the ether has evaporated in the hood and the acidified sodium sulfate is a free flowing powder.

7.2.4.2.8 Transfer to sealed labeled glass jars for use. No expiration date if stored in a sealed jar.

7.2.5 Diazomethane reagents

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7.2.5.1 N-methyl-N-nitroso-p-toluenesulfonamide (Diazald), $CH_3C_6H_4SO_2N(CH_3)NO$ – Available from Aldrich Chemical Co.

7.2.5.2 Absolute alcohol, 95% ethanol available at the local liquor store

7.2.5.3 Potassium hydroxide pellets (KOH), AR reagent

7.2.5.4 Ethyl ether, Mallinckrodt nano grade 3434 or equivalent, no preservatives added

7.2.5.5 Filter paper, Whatman #40 or equivalent

7.3 Acids and Bases

7.3.1 10 N NaOH solution – VWR Scientific

7.3.2 Concentrated sulfuric acid, H_2SO_4, EM GR grade

7.3.3 12 N H_2SO_4

7.3.3.1 Slowly add 333 mL of concentrated H2SO4 to 400 mL of DI water in a 1 L volumetric flask placed in an ice bath, swirling occasionally to dissipate heat. Once cool, fill to the mark with DI water and invert. Allow to cool and fill to the mark with DI water again. Transfer to a 1-L glass bottle with a Teflon lined cap. Assign a one year expiration date, label with a LIMS number, and store in a refrigerator.

7.3.4 Glassware Washing Solution (10% (v/v) HCl)

7.3.4.1 In a 2.5 L reagent bottle, dilute 200 mL of concentrated hydrochloric acid (HCl) in 1800 mL of DI water and mix well. Assign a one year expiration date, label with a LIMS number, and store at room temperature.

CAUTION: Care should be taken when combining any strong acid with water. Acid should always be added to water and not *vice versa*. The addition of water to acid can result in a highly rigorous and exothermic reaction that has the potential to be explosive and release noxious gasses.

7.4 Stock Standards

7.4.1 The acid form spiking solution is purchased from Absolute Standards at varied concentrations of 10 to 100 µg/mL (Part # 94540). See Table 4A for a list of the components. Label with a LIMS number and a 2-yr expiration date from the vendor preparation date and store in freezer.

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7.4.2 The methyl ester form initial calibration (ICAL) solution is purchased from Absolute Standards at a concentration of 20-200 µg/mL (Part # 95027). See Table 4B for a list of the components. Label with a LIMS number and a 2 year expiration date from the vendor preparation date and store in freezer.

7.4.3 Internal standard 2,4,5-T-d_4, CDN Isotopes # D-5553, CAS# 358731-37-01

7.4.4 Surrogate, 2,4-D-d_5, CDN Isotopes # D-5121, CAS# 352438-69-8

7.4.5 Surrogate, 2,4-dichlorophenyl acetic acid (DCAA), Chem. Service, F2035 CAS#19719-28-9

7.4.6 Second source stock standard (methyl ester) solution purchased from a different vendor than listed in Section 7.4.2, typically Restek Part #562838. See Table 4C for list of components. When entering the second source information into LIMS, enter the concentrations as acid equivalents.

7.4.7 CLP semi-volatile tuning standard: benzidine, 4,4'-DDT, decafluorotriphenylphosphine (DFTPP) and pentachlorophenol (500 µg/mL each) purchased from Absolute Standards (Part #43030)

7.5 Intermediate ICAL Standards – Not applicable to this method

7.6 Working Calibration Standards

7.6.1 ICAL Levels 4-7 are prepared by dilution of the stock standard (See Section 7.4.2) in hexane and addition of the intermediate ICAL surrogate solution (See Section 7.7.4). See Table 6A for dilution scheme and resulting target analyte ICAL and surrogate concentrations.

7.6.2 ICAL Levels 1-3 are prepared by dilution of the ICAL Levels 4-6 in hexane. See Table 6A for dilution scheme and target analyte ICAL concentrations.

7.6.3 Refer to ECCS SOP GEN-004, Analytical Standards, for proper LIMS entry and labeling requirements.

7.6.4 Transfer standards to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 1 year expiration date.

7.7 Surrogate Standards Solutions

7.7.1 Stock 2,4-D-d_5 (1000 µg/mL) – free acid

7.7.1.1 Using an analytical balance, weigh 0.025 g neat material (See Section 7.4.4) into a 25 mL volumetric flask. Dilute to volume with acetone. Transfer contents to a LIMS labeled 40 mL amber VOA vial, store frozen, and assign a 2 year expiration date.

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7.7.2 Stock DCAA (1000 µg/mL) – free acid

7.7.2.1 Using an analytical balance, weigh 0.10 g neat material (See Section 7.4.5) into a 100 mL volumetric flask. Dilute to volume with acetone or methanol. Transfer contents to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 2 year expiration date.

7.7.3 Stock ICAL surrogate standard: 2,4-D-d_5 methyl ester (1000 µg/mL)

7.7.3.1 Weigh 0.025 g of neat acid form (See Section 7.4.4) using an analytical balance into a 25 mL volumetric flask.

7.7.3.2 Dissolve standard in about 8 mL of ethyl ether.

7.7.3.3 Add diazomethane in 100 µL increments until yellow tint persists for a minimum of 15 minutes.

7.7.3.4 Removal of excess diazomethane is not necessary but can be done using a gentle stream of nitrogen for no longer than 2 minutes.

7.7.3.5 Make to volume with MTBE and transfer to a LIMS labeled 40 mL amber VOA vial, store frozen, and assign a 2 year expiration date.

7.7.4 Intermediate ICAL surrogate standard: 2,4-D-d_5 methyl ester (25 µg/mL)

7.7.4.1 Using a Gastight syringe, aliquot 2.5 mL of the 1000 µg/mL stock (See Section 7.7.3) into a 100 mL volumetric flask.

7.7.4.2 Make to volume with MTBE and transfer to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 1 year expiration date.

7.7.5 Surrogate Spiking Solution (25 µg/mL)

7.7.5.1 Using a Gastight syringe, aliquot 2.5 mL of 2,4-D-d_5 1000 µg/mL (See Section 7.7.1) and 2.5 mL of DCAA 1000 µg/mL (See Section 7.7.2) into a 100 mL volumetric flask.

7.7.5.2 Make to volume with MTBE and transfer to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a one year expiration date. See Table 6C for the dilution scheme.

7.8 Laboratory Control Sample (LCS) Spike Solutions

7.8.1 The performance of the extraction, derivitization, cleanup (when used), and analytical system is monitored by spiking reagent water or silica sand with the acid form of the compounds of interest.

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7.8.2 The LCS spiking solution is the commercial acid form stock standard solution (See Section 7.4.1).

7.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Spike Solutions

7.9.1 Same as LCS (See Section 7.8)

7.10 LOD Spike Solution

7.10.1 Dilute 5 mL of the acid form spiking solution (See Section 7.4.1) and 4 mL of the surrogate spiking solution (See Section 7.7.5) in a 50 mL volumetric flask and make to volume with acetone. Transfer to a LIMS labeled 40 mL amber VOA vial, store frozen, and assign a 1 year expiration date.

7.11 Second Source Calibration Verification Standard – Methyl Ester (200, 400, 800,1000 ng/mL)

7.11.1 Using a Gastight syringe, aliquot 1 mL of the second source calibration verification standard (See Section 7.4.6) and 0.8 mL of 2,4-D-d_5 surrogate solution into a 100 mL volumetric flask. See Table 6B for list of compounds included and respective concentrations and Table 6D for methyl ester equivalents.

7.11.2 Make to volume with hexane, transfer to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 1 year expiration date.

7.12 Internal Standard Solutions

7.12.1 Stock internal standard: 2,4,5-T-d_4 methyl ester (1000 µg/mL)

7.12.1.1 Weigh 0.025 g of neat acid form (See Section 7.4.3) using an analytical balance into a 25 mL volumetric flask.

7.12.1.2 Dissolve standard in about 8 mL of ethyl ether.

7.12.1.3 Add diazomethane in 100 µL increments until a yellow tint persists for a minimum of 15 minutes.

7.12.1.4 Removal of excess diazomethane is not necessary, but can be done using a gentle stream of nitrogen for no more than 2 minutes.

7.12.1.5 Make to volume with hexane, transfer to a LIMS labeled 40 mL amber VOA vial, store frozen, and assign a 2 year expiration date.

7.12.2 Working internal standard, 2,4,5-T-d_4 (5 µg/mL)

7.12.2.1 Using a Gastight syringe, transfer 500 µL of the stock internal standard (See Section 7.11.1) to a 100 mL volumetric flask.

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7.12.2.2 Make to volume with acetone, transfer to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 1 year expiration date.

7.13 Working Tune Performance Check Solution (2000 ng/mL)

7.13.1 Using a Gastight syringe, transfer 400 µL of the stock CLP semi-volatile tuning standard (See Section 7.4.7) to a 100 mL volumetric flask.

7.13.2 Make to volume with hexane, transfer to LIMS labeled 40 mL amber VOA vials, store frozen, and assign a 1 year expiration date.

7.14 Diazomethane Generation

7.14.1 The apparatus is shown in Figure 2. The procedure outlined will generate approximately 160 mL diazomethane solution. **CAUTION: Diazomethane is a carcinogen and an explosive reagent.** The kit is produced by Aldrich.

7.14.2 Pre-rinse the glassware with diethyl ether. Set up the ice bath and do not turn on the re-circulating pump until you are ready to start the distillation. Set up the hot water bath and maintain at 65 °C.

7.14.3 Add 20 g potassium hydroxide and 32 mL DI water to a 250 mL round bottom flask. Swirl the flask to dissolve the potassium hydroxide and allow to cool. Add 40 mL absolute alcohol (alcohol should be 95% or 190 proof) to this solution.

7.14.4 In a 250 mL Erlenmeyer flask, dissolve 20 g diazald (See Section 7.2.5.1) in 180 mL diethyl ether. Filter the solution into a 125 mL separatory funnel. Rinse the sidewalls with diethyl ether.

7.14.5 Once the water bath temperature is controlled at 65 °C, assemble the 250 mL round bottom reaction flask and attach the separatory funnel. Add 10-15 mL diethyl ether to the 500 mL collection flask prior to starting distillation.

7.14.6 Distillation

7.14.6.1 Once all the fittings are secure and the ice and hot water baths are at temperature, slowly raise the hot water bath to cover the bottom of the reaction flask and solution in the 250 mL round bottom flask.

7.14.6.2 Slowly add the diazald solution to the reaction flask. A yellow colored distillate should form within 1-2 minutes.

7.14.6.3 Continue to add diazald drop wise (1-2 drops per second). The rate of addition should equal the rate of distillation.

7.14.6.4 Monitor the water bath temperature throughout the distillation at 65 ± 2 °C.

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7.14.6.5 Distillation is complete once all of the diazald has been added. Rinse the separatory funnel with 10 mL diethyl ether. The distillate should become colorless. If not, add more diethyl ether. Remove the reaction flask from the hot water bath and allow it to cool to room temperature.

7.14.7 Transfer the diazomethane solution to LIMS labeled 40 mL amber VOA vials and store frozen.

NOTE: First time preparers should be under the supervision of an experienced preparer.

7.14.8 Record information for each batch that is prepared in the diazomethane logbook and in LIMS. There is no assigned expiration date.

NOTE: The solution is usable as long as there is a bright yellow color present. When the solution fades to a pale yellow color the reagent should be discarded and a new solution prepared.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples should be collected in 1-L amber glass bottles with Teflon-lined caps and refrigerated at 4 °C after collection. Aqueous samples should be extracted within 7 days of collection.

8.2 Soil samples should be collected in amber glass jars (usually 4 oz.) with Teflon-lined caps and refrigerated at 4 °C after collection. Soil samples should be extracted within 14 days of collection.

8.3 Store extracts in a freezer at -15 °C or below and analyze within 40 days of extraction.

9 PROCEDURE

9.1 Preparation Methods

9.1.1 Extraction of aqueous samples

9.1.1.1 Prepare batch QC samples in accordance with Section 10: Quality Control.

9.1.1.2 An additional QC requirement for this method is a lab pure. The lab pure consists of the acid form of the targets that is derivatized (but not extracted) and analyzed. Follow the preparation instructions below.

9.1.1.2.1 In a 10 mL volumetric, add about 4 mL of hexane.

9.1.1.2.2 Add 80 µL of surrogate and 100 µL of spike solution to the 10 mL volumetric as in 9.1.4 and 9.1.5.

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9.1.1.2.3 Add diazomethane in 100 µL increments until a yellow tint persists for a minimum of 15 minutes. Typically 300-400 µL of diazomethane is needed to complete derivitization. Keep the volumetric covered during the wait time.

9.1.1.2.4 Make to 10 mL with hexane, and transfer to a LIMS labeled 12 mL vial, and store in a freezer with the extraction batch.

9.1.1.2.5 The lab pure must be injected as part of the batch QC to demonstrate the efficiency of the derivatization, and is designated in LIMS as an SRM sample.

9.1.1.3 All glassware used to extract water samples is pre-rinsed with acetone and then dichloromethane or ethyl ether prior to use (i.e. separatory funnels, Zymark tubes, transfer funnels, and stopcocks).

9.1.1.4 Transfer the contents of the 1 L bottle as received (mark the meniscus of the water on the bottle with black marker prior to transferring) into a 2 L separatory funnel. Avoid transferring any sediment if possible. Fill the container with tap water to the black mark and pour into a 1 L graduate. Record the volume of water in LIMS.

Or

Using a 1 L graduated cylinder, measure 1 L (nominal) of sample and transfer quantitatively to the 2 L separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 L. Record the volume of water in LIMS.

NOTE: If a sample contains a sediment layer at the bottom of the sample bottle, decant, leaving the sediment layer in the sample container. Remember to determine the volume of water used during the extraction and record the volume of water in LIMS.

NOTE: When water samples have sample descriptions of floor waste/tank rinsing, sump water or some unusual description, consult senior staff/ops manager before proceeding.

9.1.1.5 Add 80 µL of surrogate (25 µg/mL, See Section 7.7.5) to all samples including the blank, LCS, and MS/MSD.

9.1.1.6 Add 100 µL of the LCS/MS/MSD spike solution (See Sections 7.8 and 7.9) to the LCS, MS/MSD.

9.1.1.7 Sample hydrolysis

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9.1.1.7.1 Add 200 g of baked sodium chloride (See Section 7.2.1) to the sample, seal, and shake to dissolve the salt.

9.1.1.7.2 Add 10 mL of 10 N sodium hydroxide to all the samples, seal, and shake to mix.

9.1.1.7.3 Check pH with wide range pH paper and verify each sample is at pH ≥ 13. If not, add additional 10 N sodium hydroxide to increase the pH ≥ 13.

9.1.1.7.4 Allow to sit at room temperature for a minimum of one hour.

9.1.1.7.5 Shake the separatory funnels a minimum of three times at 15, 30, and 45 minutes.

9.1.1.7.6 This will complete the hydrolysis and convert the various forms to the free acid form.

9.1.1.8 Sample clean-up

NOTE: Since samples are analyzed by mass spectroscopy, the sample clean-up step may not be required for known sample matrices.

9.1.1.8.1 Once the hydrolysis step is complete, add 100 mL of dichloromethane to each separatory funnel.

9.1.1.8.2 Cap and invert the separatory funnel and vent prior to shaking. Shake sample vigorously for a minimum of two minutes while venting frequently.

NOTE: Vent in to the hood to minimize dichloromethane exposure.

NOTE: If the sample appears to form an emulsion with the addition of the dichloromethane, use an alternative shaking technique by employing end to end tilting of the separatory funnel rather than vigorous shaking for two minutes.

9.1.1.8.3 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, employ mechanical techniques to help complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods.

9.1.1.8.4 Discard the dichloromethane phase.

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9.1.1.9 Sample extraction

9.1.1.9.1 Add 15 mL of cold (4 °C) 12 N sulfuric acid to each separatory funnel, seal, and shake to mix. Check the pH of the sample with wide range pH paper. If the sample does not have a pH ≤ 2, adjust the pH by adding more 12 N sulfuric acid.

9.1.1.9.2 Add 60 mL diethyl ether to each separatory funnel. Cap and invert the separatory funnel and vent prior to shaking. Shake sample vigorously for a minimum of two minutes while venting frequently.

NOTE: Vent quickly after capping as pressure builds up quickly.

9.1.1.9.3 Allow the ether layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one third the volume of the solvent layer, employ mechanical techniques to complete the phase separation.

9.1.1.9.4 Transfer the aqueous layer into a 1000 and 250 mL Erlenmeyer flask. Both flasks will be necessary to drain the aqueous layer.

9.1.1.9.5 Transfer approximately 10 g of acidified sodium sulfate to a solvent rinsed 250 mL Erlenmeyer flask. An adequate amount of sodium sulfate just covers the bottom of the Erlenmeyer flask.

9.1.1.9.6 Transfer the ether extract in the separatory funnel to the 250 mL Erlenmeyer by pouring out of the top of the separatory funnel. Be careful not to transfer any remaining water.

NOTE: If water is transferred, the sodium sulfate will clump up. This will cause problems later in the method and could result in low recoveries.

9.1.1.9.7 Swirl the ether in the Erlenmeyer flask to allow adequate drying of the ether by the sodium sulfate. Swirl the Erlenmeyer flask and make sure the sodium sulfate is free flowing. If not, add 1 to 2 g more acidified sodium sulfate until the sodium sulfate is free flowing.

9.1.1.9.8 Return the aqueous phase that is in the 1000 and 250 mL Erlenmeyer flasks to the separatory funnel.

9.1.1.9.9 Add 30 mL diethyl ether to each of the 1000 and 250mL Erlenmeyer flasks, swirl to rinse and transfer the ether to the separatory funnel. Cap the separatory funnel and extract by shaking vigorously for a minimum of 2 minutes with periodic venting.

NOTE: Vent quickly after capping as pressure builds up very fast.

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9.1.1.9.10 Allow the layers to separate for a minimum of 10 minutes.

9.1.1.9.11 Drain the aqueous layer into the 1000 and 250 mL Erlenmeyer flasks.

9.1.1.9.12 Transfer the ether layer by carefully pouring the ether out the top of the separatory funnel into the 250 mL Erlenmeyer flask containing the previous extract, making sure not to transfer any remaining water. Swirl the Erlenmeyer flask and make sure the sodium sulfate is free flowing. If not, add 1 to 2 g more acidified sodium sulfate until the sodium sulfate is free flowing.

9.1.1.9.13 Repeat steps 9.1.1.8.8 to 9.1.1.8.12 one additional time using 60 mL of ethyl ether. At the completion of the third extraction discard the aqueous layer.

9.1.1.9.14 Allow the extract to remain in contact with the sodium sulfate for 30 minutes. The extracts can also be held in contact with the sodium sulfate overnight in the hood. However, cover the 250 mL Erlenmeyer flask with aluminum foil or ground glass stopper.

NOTE: The drying step is very critical. Any moisture remaining in the ether could cause incomplete derivatization resulting in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling.

NOTE: The longer the extract is in contact with the sodium sulfate the less water will be left in the ethyl ether, reducing the chances of water in the bottom of the Zymark tube prior to derivatization.

9.1.1.9.15 Transfer the dried extract into a 200 mL Zymark collection tube. Rinse the Erlenmeyer flask and funnel with three 10 mL portions of diethyl ether to complete the quantitative transfer.

NOTE: Do not transfer any of the sodium sulfate.

9.1.1.9.16 Samples are now ready for concentration (See Step 9.1.3).

9.1.2 Extraction of soil samples

9.1.2.1 Soil samples are typically not homogenous. Eliminate stones and other debris from the sample.

9.1.2.2 Prepare batch QC samples in accordance with Section 10: Quality Control.

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9.1.2.2.1 An additional QC requirement for this method is a lab pure. The lab pure consists of the acid form of the targets that is derivatized (but not extracted) and analyzed. Follow preparation instructions below.

9.1.2.2.1.1 In a 25 mL volumetric, add about 4 mL of hexane.

9.1.2.2.1.2 Add 200 µL of surrogate and 250 µL of spike solution to the 25 mL volumetric as in 9.2.4 and 9.2.5.

9.1.2.2.1.3 Add diazomethane in 100 µL increments until a yellow tint persists for a minimum of 15 minutes. Typically 300-400 µL of diazomethane is needed to complete derivitization. Keep the volumetric covered during the wait time.

9.1.2.2.1.4 Make to 25 mL with hexane, and transfer to a LIMS labeled 12 mL vial, and store in a freezer with the extraction batch.

9.1.2.2.1.5 The lab pure must be injected as part of the batch QC to demonstrate the efficiency of the derivatization, and is designated in LIMS as an SRM sample.

9.1.2.3 Weigh 10 g of each unknown sample into a LIMS labeled and tared 40 mL clear VOA vial.

9.1.2.4 Add 200 µL of surrogate spiking solution (See Section 7.7.5).

9.1.2.5 Add 250 µL of the LCS/MS/MSD spike solution (See Section 7.8) to the LCS, MS/MSD.

9.1.2.6 Add 25 mL of soil extraction solvent (See Section 7.1.4) to all samples.

9.1.2.7 Cap and place horizontally on a shaker table for a minimum of one hour. Set the speed to ensure adequate mixing and to ensure clay samples will break up.

NOTE: If necessary, clayey soils should be manually broken up with a spatula.

9.1.2.8 Centrifuge samples in VOA vials at approximately 2000 RPM for about 15 minutes or until the upper aqueous layer is clear.

9.1.2.9 Set up a 500 mL or 1 L separatory funnel and add 350 mL of DI water and 100 g of baked sodium chloride (See Section 7.2.1.1).

9.1.2.10 Shake to dissolve the sodium chloride prior to addition of the initial extract to the separatory funnel.

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9.1.2.11 Decant the clear extract to the 500 mL or 1 L separatory funnel.

NOTE: Do not transfer any of the soil particles.

NOTE: Transfer may be done using a disposable pipette.

9.1.2.12 Repeat steps 9.1.2.7 to 9.1.2.8 and 9.1.2.11 two additional times with approximately 15 mL of extraction solvent. The shaking time is reduced from 1 hour to 10 minutes.

NOTE: Sample extracts may be combined in a 60 mL vial before transferring to the 500 mL or 1 L separatory funnel and stored in a refrigerator overnight as long as 4-nitrophenol is not a target analyte.

9.1.2.13 Check pH of separatory funnel to make sure the pH ≥ 12. If not, add 10 N sodium hydroxide until it reaches pH ≥ 12. Allow an hour after adding the sodium hydroxide for the saponification process to free all forms of the herbicides in the samples.

9.1.2.14 Set up a 500 mL Erlenmeyer flask to collect the aqueous layer between each of the ethyl ether extracts, and a second 250 mL flask, to collect the ethyl ether extracts. Transfer approximately 10 g of acidified sodium sulfate in a solvent rinsed 250 mL Erlenmeyer flask. An adequate amount of sodium sulfate just covers the bottom of the Erlenmeyer flask.

9.1.2.15 Add 15 mL of cold 12 N (4 °C) sulfuric acid to the separatory funnel. Shake to ensure mixing. Check pH and make sure the pH < 3. If not, add additional sulfuric acid until the pH < 3.

9.1.2.16 Add 60 mL ethyl ether. Cap and invert the separatory funnel and vent prior to shaking. Shake the sample vigorously for a minimum of two minutes while venting frequently.

NOTE: Vent quickly after capping as pressure builds up quickly.

9.1.2.17 Allow the layers to separate completely for at least 10 minutes.

9.1.2.18 Transfer the aqueous portion to the 500 mL Erlenmeyer flask. Leave the emulsion in the separatory funnel and break up mechanically. Adding additional ether will help the process.

9.1.2.19 Transfer the ether layer by carefully pouring the ether out the top of the separatory funnel into the 250 mL Erlynmeyer flask, making sure not to transfer any remaining water. Swirl the Erlenmeyer flask and make sure the sodium sulfate is free flowing. If not, add 1 to 2 g more acidified sodium sulfate until the sodium sulfate is free flowing.

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9.1.2.20 Transfer the aqueous portion in the Erlenmeyer flasks back to the separatory funnels.

9.1.2.21 Repeat steps 9.1.2.16 to 9.1.2.20 two more times with 30 mL of ethyl ether combining all the extracts in the same Erlenmeyer flask.

9.1.2.22 Allow the extract to remain in contact with the sodium sulfate for 30 minutes. The extracts can also be held in contact with the sodium sulfate overnight in the hood. However, cover the 250 mL Erlenmeyer flask with aluminum foil or ground glass stopper.

NOTE: The drying step is very critical. Any moisture remaining in the ether could cause incomplete derivatization resulting in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling.

NOTE: The longer the extract is in contact with the sodium sulfate the less water will be left in the ethyl ether, reducing the chances of water in the bottom of the Zymark tube prior to derivatization.

9.1.2.23 Transfer the dried extract into a 200 mL Zymark collection tube. Rinse the Erlenmeyer flask and funnel with three 10 mL portions of diethyl ether to complete the quantitative transfer.

NOTE: Do not transfer any of the sodium sulfate.

9.1.2.24 Samples are now ready for concentration (See Step 9.1.3).

9.1.3 Extract concentration

9.1.3.1 Place the tubes in the Turbovap with the water bath set at 30 °C. Adjust the Turbovap pressure control to approximately 10 PSI. Set the timer for 10 minutes and begin the evaporation.

9.1.3.2 Evaporate the extract to approximately 20 mL. Do not try and reduce the volume below 20 mL as light target analytes like dalapon will volatilize and will result in low recoveries.

NOTE: If water separates in the bottom of the Zymark tube following evaporation to 20 mL, a small amount of acidified sodium sulfate may be added to fill the tip of the Zymark tube.

9.1.3.3 Add 30 mL of hexane.

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9.1.3.4 Evaporate extracts for waters to a volume of 7-8 mL and soil extracts to a volume of 10-11 mL.

9.1.3.5 Proceed to step 9.1.4 derivatization.

9.1.4 Derivatization

9.1.4.1 Add approximately 1 mL of diazomethane solution to each Zymark tube or until a yellow tint persists, swirl gently and let stand for at least 15 minutes. If the yellow color dissipates, add more diazomethane solution in 200 µL increments, swirl gently, and allow to stand until the yellow tint persists for 15 minutes.

9.1.5 Final quantitative transfer

9.1.5.1 Waters: Quantitatively transfer the extract to a 10 mL volumetric using hexane.

9.1.5.2 Soils: Quantitatively transfer the extract to a 25 mL volumetric using hexane.

NOTE: If the hexane is cloudy, probably due to residual water, add a small amount of sodium sulfate to the extract after it is brought to volume in the volumetric flask. Cap and shake the volumetric to clear the hexane.

9.1.5.3 Transfer extracts to LIMS labeled 12 mL amber vials, store frozen, and analyze within 40 days of extraction.

NOTE: Do not transfer any sodium sulfate to the extract storage vial.

9.2 Clean-Up Methods

9.2.1 A cleanup is not performed with this method.

9.3 Instrument Conditions

9.3.1 GC conditions

Head pressure:	Constant Flow Off, 5 PSI at 80 °C
Carrier gas:	Helium
Column Flow:	1.5 mL/minute
Split Flow:	35 mL/minute
Purge Valve:	Initial Off
On Time:	3.0 minutes
Transfer Line Temperature:	320 °C
Injector Temperature:	200 °C
Injection Volume	6 µL

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GC Temperature Program

Initial Temperature:	45 °C
Initial Hold:	5.0 minutes
1st Ramp:	22 °C/minute
1st Final Temperature:	150 °C
1st Hold Time:	0 minutes
2nd Ramp:	12 °C/minute
2nd Final Temperature:	250 °C
2nd Hold Time:	0 minutes
3rd Ramp:	22 °C/minute
3rd Final Temperature	320 °C
3rd Final Hold Time:	3.0 minutes

9.3.2 MS conditions

Tune:	Maximum Sensitivity Autotune
EM Voltage:	Adjusted to 4 million counts for m/z 69
Scan Range:	45-450 amu
Scan Time:	0.83 scans/sec.
Solvent Delay:	4.5 minutes

9.3.3 The above parameters may be modified based on specific objectives of a non-routine project or to solve a chromatography problem with a particular batch of samples.

9.4 Preventive Maintenance/Troubleshooting

9.4.1 Preventative maintenance should be performed prior to the start of each analytical sequence. This includes clipping the front end of the guard column and replacing the septa, inlet liner, inlet seal and their associated o-rings and ferrules.

9.4.2 System performance can be evaluated by running low level and mid-level ICAL standards known as CRLs. If general guidelines are not met for CRL standards refer to the subsequent topics in this section for further troubleshooting.

9.4.2.1 Low level CRL: L-1 of the ICAL. Evaluate this low level standard for acceptable peak shape and signal to noise ratio for target compounds.

9.4.2.2 Mid-level CRL: L-5 of the ICAL. Evaluate this mid-level standard for acceptable minimum areas for target compounds, surrogates internal standards. This CRL is also used to update retentions times of analytes prior to the start of the ICAL.

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9.4.2.3 Tune performance check: Evaluate this tune standard for peak tailing, compound degradation, and MS tune performance. Refer to section 9.6.1.2 for specific acceptance criteria regarding the tune performance check.

9.4.3 If the guidelines established above have not been met, further instrument maintenance may be required to optimize system performance. The following sections outline potential steps in optimizing the GC/MS system.

9.4.3.1 Evaluate the guard column for sufficient length. If less than one meter of the guard column is left, it is recommended that it be replaced. Moreover, prior to installing a new guard column, one meter should be clipped from the analytical column.

9.4.3.2 If replacing the guard column does not correct the problem, the analytical column may then need to be replaced.

9.4.3.3 If problems still persist in the GC/MS system, refer to the next section regarding MS preventative maintenance.

9.4.4 Less routinely, the MS source should be cleaned, filaments examined/changed, and electron multipliers replaced. This may aid in the optimization of the overall GC/MS system.

9.4.5 Advanced MS troubleshooting and other topics.

9.4.5.1 Detection of leaks in the GC/MS system: the presence of a base peak at m/z 28 (N_2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of a leak is a loose transfer line nut sealing the GC capillary column to the MS transfer line. Less commonly, a break or improper alignment of the gasket between the MS source and the vacuum manifold may be the source of the leak.

9.4.5.2 Leak checking: Leak checking may be done by lightly spraying an air duster around areas of the MS that pose the most chance for a leak. These areas include around the gasket of the vacuum manifold, the seal between the MS and the MS interface, and the transfer line nut. Most commercially available air dusters (air in a can) contain difluoroethane, which will be quickly sucked into the MS system at any potential leak point. Actively scanning the MS while spraying the air duster will yield base peaks at m/z 51 and 65 at the point of any potential leak.

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9.4.5.3 Water in the GC/MS system: The presence of a base peak at m/z 18 (H_2O) that is higher than m/z 69 in the tune report indicates excessive water remains in the MS manifold. The MS vacuum system does not efficiently remove water and this condition indicates that a long equilibration time is needed prior to the initial calibration. To aid in accelerating the equilibration process, the GC oven can be ramped to 115 °C during the pump down process. Injecting methanol or acetone can also aid in removing water.

9.4.5.4 Electron multiplier voltage: Raising the electron multiplier voltage by 200 V should approximately double the abundance of the m/z 69 ion in the tune report for a properly functioning electron multiplier. Over time this ratio will decrease. Nominal increase of the m/z 69 ion abundance as the voltage is increased in the MS is an indicator that the electron multiplier is damaged or worn out and requires replacement. An electron multiplier voltage higher than 2700 V in the tune report indicates that the multiplier needs replacement and/or the MS source needs cleaning.

9.4.5.5 Peak shape/resolution of PFTBA (perfluorotributylamine) calibration peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MS should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the tune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MS source needs to be cleaned. Refer to section 9.6.1.1 for more specific criteria when evaluating the PFTBA spectrum.

9.4.5.6 Standard spectra autotune: In the event that a maximum sensitivity autotune program does not yield acceptable spectra a standard spectra autotune program may be employed to tune the MS. This autotune program is designed to adjust ion ratios to match those found in mass spectral library reference spectra, but does typically provide high MS sensitivity. As a consequence, the MS lenses (i.e. the entrance lens offset, entrance lens, ion focus repeller) may need adjustment to provide high MS sensitivity while also keeping the relative intensities of m/z 69, 219, & 502 within acceptable limits.

9.4.6 All maintenance performed on the GC/MS system should be performed by a chemist skilled in GC/MS maintenance and recorded in the GC and/or MS maintenance logs.

9.5 Retention Time Windows

9.5.1 Relative retention time of the sample component must be within $\pm$ 0.06 minutes of the standard component. The search window that the integration algorithm uses to identify peaks as a target analyte is set at ± 0.3 minutes.

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9.5.2 If retention times vary or are increasing from injection to injection, the analyst must assure all target analytes are being properly identified. System maintenance may be required.

9.6 Instrumental Analysis

9.6.1 Tuning

9.6.1.1 Maximum sensitivity autotune: The mass spectrometer is tuned prior to calibration using the maximum sensitivity autotune program. This autotune program is designed to adjust ion ratios to match those found in mass spectral library reference spectra while also providing high MS sensitivity. A valid autotune should provide peak widths < 0.60 amu with mass assignments not differing by more than 0.20 amu for masses 69, 219, and 502 from the PFTBA tuning compound. The relative intensities for m/z 69, 219, and 502 should be 100%, 25-45%, and 0.5-1.5%, respectively. Isotope masses should be present for m/z 70, 220, and 503. Figure 3 shows a typical report for a successful maximum sensitivity autotune.

NOTE: If a maximum sensitivity autotune program does not yield an acceptable tune, a standard spectra autotune program may be used to tune the MS. Refer to the Preventative Maintenance/Troubleshooting section of this SOP for further details regarding this tune program.

9.6.1.2 After running the maximum sensitivity autotune program, the tuning parameters must be verified by injecting 6 µL of a 2000 ng/ml solution (12 ng) of DFTPP (Section 5.12). This must occur before initial calibration and every twelve hours thereafter.

9.6.1.3 Using the exact run parameters when analyzing samples, inject the DFTPP and acquire data. Once the DFTPP elutes use any combination of the top 3 scans to determine the fragment ratios for the ions listed in Table 9. The spectra must meet the ranges listed in Table 9 for Method 8270D before analysis of samples or standards continues. DFTPP must be injected at a minimum of the beginning of every twelve hours of instrument operation.

9.6.2 Tune performance check: The tune performance check is used to evaluate GC/MS performance. DFTPP (decafluorotriphenylphosphine) is used to validate MS performance by evaluating relative mass intensities of DFTPP ions. GC column performance and injection port inertness is also validated by calculating the tailing factors of benzidine and pentachlorophenol as well as the breakdown of DDT to DDE and DDD.

9.6.2.1 Inject 6 µL of a 2000 ng/mL GC/MS tuning standard.

9.6.2.2 Refer to Table 9 for required DFTPP acceptance criteria.

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9.6.2.3 Benzidine and pentachlorophenol should not exceed a tailing factor of 2 as given by the following equation:

9.6.2.4 $TailingFactor = \frac{BC}{AB}$

9.6.2.5 The equation compares the width of the back half of the peak to the width of the front half of the peak at 10% height of the peak. See Figure 4 for an example tailing factor calculation.

9.6.2.6 Degradation of DDT to DDE and DDD should not exceed 20%.

9.6.2.7 If the acceptance criteria are not met, corrective action should be taken. This may include system maintenance or retuning the instrument prior to continuation of analysis.

9.6.3 Calibration

9.6.3.1 The ICAL is performed using the internal standard technique. Typically seven different levels of calibration standards are used to plot calibration curves using a quadratic regression fit with the weight on the inverse of the concentration. An acceptable calibration curve has a correlation coefficient (R) of 0.995 or greater.

NOTE: If evaluating the calibration curve based on the coefficient of determination (R^2), the R^2 value must be 0.990 or greater.

9.6.3.2 Table 3 provides a list of characteristic primary and secondary ions for each of the target compounds, internal standards and surrogates.

9.6.3.3 The concentrations of the ICAL standards are corrected by mass ratio (See Section 9.7.6.3) to the acid form prior to entering them into the MS EnviroQuant® software calibration file. See Table 6D for the corrected ICAL concentrations.

9.6.3.4 To each 2 mL auto-sampler vial, add 40 µL of the internal standard solution (See Section 7.12.2) with a syringe. Transfer 800 µL of each calibration standard to the vial. Make sure all vials to be analyzed are prepared the same and properly labeled. Cap and invert to mix.

NOTE: Once prepared as in 9.6.2.4, ICAL standards and CCV vials can be subdivided into micro-vials inserted into 2 mL auto-sampler vials.

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9.6.3.5 Inject each calibration standard, collect the data and tabulate the area response of the characteristic ions against the concentration for each target analyte and each internal standard. Select the quadratic curve fit analysis from the calibration options for each compound with the weight on the inverse of the concentrations.

Quadratic regression with inverse of concentration weighting

$$Y = AX^2 + BX + C$$

Where: Y = Peak area ratio of each analyte

$$\text{Peak Area Ratio} = \frac{\text{Peak Area of Target Analyte}}{\text{Peak Area of Associated Internal Standard}}$$

A = Second order constant term (quadratic term)
B = First order constant term (linear term)
C = Constant term
X = Concentration Ratio of each target analyte

$$\text{Concentration Ratio} = \frac{\text{Concentration of Target Analyte in ng/ml}}{\text{Concentration of Internal Standard in ng/ml}}$$

$$\text{Weighting Factor} = \frac{1}{\text{X}}$$

9.6.4 Sample Analysis

9.6.4.1 Samples are analyzed in a group referred to as a GC sequence, which is designated by the sequence number given by LIMS. A typical GC sequence begins with CRL samples and a tune performance check. If initial instrument checks are acceptable, the sequence is followed by an instrument blank (IBL), the ICAL, second source (SCV) standards, and initial calibration verification (ICV) standards. Samples extracts interspersed with CCV and tune performance checks, on a one per 10 basis, follow. The sequence ends with a CCV when the entire sequence has been injected, or when quantitative and/or qualitative QC criteria are no longer acceptable.

9.6.4.2 Add 40 µL of the internal standard solution (See 7.12.2) to a properly labeled 2 mL injection vial. Transfer 800 µL of each sample to the vial. Preparation of vials in sets of five at a time is recommended. Prepare each vial (standard, sample or QC sample) the same. Inject the samples and collect and process the data using a chromatography workstation. Internal standard limits are -50% to 200% from the first injected ICV or CCV standard. If recoveries are outside of that range, samples must to be re-aliquoted and re-injected.

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9.6.4.3 If a response exceeds the theoretical concentration of the highest standard, dilute the sample extract and reanalyze. Dilute such that the concentration is in the upper half of the standard curve if possible.

9.6.4.4 A calibrated system requires analysis of CCVs to remain valid. CCV standards must be injected after every ten samples or less and at the end of the analysis sequence. Two CCVs concentrations are used (100 and 500 ng/mL) because quadratic curves are employed. The response for each compound must not exceed a 20% difference when compared to the theoretical value of the calibration standard, except for dalapon and acifluorfen which have ± 30% limits. When the limit is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary before re-calibrating and proceeding with sample analysis. All samples that were injected after the last acceptable CCV should be re-injected, or appropriately qualified in accordance with GEN-015, Qualification of Data.

9.6.4.5 $Percent\ Difference = \frac{R2-R1}{R1} \times 100$

9.6.4.6 Where: R1 = Theoretical Concentration.
R2 = Calculated Concentration from CCV analyses.

9.6.4.7 Qualitative analysis

9.6.4.8 The qualitative identification of each target analyte is based on retention time, and on comparison of the extracted ion chromatograms of the sample with the characteristic extracted ion chromatograms from reference spectra in the current method. The current reference spectra should be generated from the current ICAL using the conditions of the method and/or by using library reference spectra. The characteristic ions for this method are listed in Table 3. Compounds are identified as present when the following criteria are met.

9.6.4.8.1.1 The intensities of the characteristic ions of a target analyte maximize in the same scan or within one scan of each other.

9.6.4.8.1.2 Selection of a peak as a target analyte by the data system occurs where the identification is based on the presence of the target ions specific for the target analyte at the target analyte-specific retention time.

9.6.4.8.1.3 The relative intensities of the characteristic ions agree within 30% of when compared to the reference spectrum. As an example, for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

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9.6.4.8.2 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When peaks obviously represent more than one component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte background spectra is important. If two components co-elute directly over one another it becomes impossible to collect a pure spectra of either component. If one component is a shoulder peak of another component, the first spectra is taken from the far side of the sample component peak of interest and the second spectra is taken from the far side of shoulder peak. Background subtraction then yields relatively pure spectra of the sample target analyte peak of interest.

9.6.4.8.3 Examination of extracted ion chromatograms can aid in the selection of spectra and in qualitative identification of target analytes when co-elution occurs. The identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound(s).

9.6.4.8.4 In rare cases where the primary characteristic ion experiences interference from the chromatographic background, a secondary ion may be used to quantitate a target analyte.

9.6.4.9 Quantitation

9.6.4.9.1 Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the extracted ion chromatogram of the primary characteristic ion. The concentration in ng/mL of the compounds (acid form) found in the sample extract is calculated by the chromatography workstation using reverse extrapolation from the standard curve generated in Section 9.7.2.

9.6.4.9.2 The concentration of water samples in µg/L, or soil samples in µg/kg is then calculated as follows:

$$Concentration\ (\mu g/L) = \frac{A_x \times D \times V_e \times \frac{1\ \mu g}{1000\ ng}}{V_s}$$

Where: A_x = Concentration of compound (acid form) determined in ng/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
V_s = Volume of sample extracted in Liters

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$$Concentration\ (\mu g/kg) = \frac{A_x \times D \times V_e \times \frac{1\ \mu g}{1000\ ng}}{W_s}$$

Where: A_x = Concentration of compound (acid form) determined in ng/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
W_s = Weight of sample extracted in kg

9.6.4.9.3 Methyl ester correction factors

NOTE: These correction factors are included as a reference. All initial calibrations will have the standard concentrations corrected to the acid form at the time they are entered into the data system. See Table 6D for corrected standard concentrations.

Compound	Correction Factor
2,4-D	1.0633
2,4-DB	1.0564
2,4,5-T	1.0548
2,4,5-TP (Silvex)	1.0519
Acifluorfen	1.0387
Bentazon	1.0582
Bromoxynil	1.0506
Chloramben	1.0680
Clopyralid	1.0729
Dacthal	1.0921
Dalapon	1.0979
Dicamba	1.0633
Dichlorprop	1.0596
Diclofop	1.0428
Dinoseb	1.0525
MCPA	1.0700
MCPB	1.0612
MCPP	1.0654
4-Nitrophenol	1.1007
Pentachlorophenol	1.0525
Picloram	1.0580
Triclopyr	1.0546
DCAA (Surrogate)	1.0686
2,4-D-d_5 (Surrogate)	None[1]

[1]No correction. Methyl ester made from acid form.

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9.6.5 Data Package and Review

9.6.5.1 Refer to GEN-016, Data Review Procedures for preparation and review of each sequence data package.

9.6.5.2 Manual integrations must be properly documented in accordance with GEN-018.

9.6.5.3 Prior to reporting results via LIMS, false positives must be removed by Q-deletion.

10 QUALITY CONTROL

10.1 Refer to Method 8000B for general GC quality control procedures for chromatography methods.

10.2 Procedures to check the GC system operation are found in Method 8000B and in manufacturers' manuals.

10.3 An analytical batch consists of 20 or fewer unknown samples. Quality control samples must be analyzed with each batch at the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

Note: If an MS/MSD cannot be prepared because of limited sample volume, a duplicate LCS must be prepared.

10.4 Method blanks consist of an aliquot of laboratory reagent water or silica sand prepared and processed through every step of the extraction process. If target analytes or interfering contamination is found, the samples (including quality control samples) should be re-extracted or reported with appropriate qualifiers.

10.5 LCSs consist of an aliquot of laboratory reagent water or silica sand fortified with the target compounds, prepared and processed through every step of the extraction process. LCS recovery control limits are generated through LIMS on at least a yearly basis. If the recovery of any of the target compounds is outside control limits, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data. If an MS/MSD cannot be analyzed with a batch, a duplicate LCS must be prepared with the batch. If an LCS duplicate is analyzed, the precision acceptance limit is an RPD of $\leq$ 20%.

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10.6 A lab pure sample (Sections 9.1.1.2 and 9.1.2.2) is prepared with each batch of 20 or fewer samples, minimum one per day. Lab pure control limits are the same as CCV limits. If the response of a lab pure differs by more than ± 20% (except for dalapon and acifluorfen which have a ± 30% limit), check the batch LCS results. If the LCS recoveries are acceptable and higher than the lab pure, no action is required. If the LCS results are acceptable, but low in the range of the lab pure results, qualify the data in accordance with GEN-015, Qualification of Data. If the lab pure and LCS results are both outside control limits, corrective action must be taken, and the samples (including quality control samples) should be re-extracted, if possible, or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.7 MS/MSD samples consist of duplicate aliquots of sample fortified with the target compounds, prepared and processed through every step of the process. MS/MSD recovery control limits are generated through LIMS on at least a yearly basis but are considered advisory in nature. The precision acceptance limit is an RPD of $\leq$ 20%. If an MS/MSD fails a control limit, the MS/MSD results are qualified in accordance with GEN-015, Qualification of Data.

10.8 An ICV is analyzed at the beginning of an analysis sequence, and CCVs after every ten or fewer injections and at the end of the run. Two ICV/CCV concentrations are used (100 and 500 ng/mL) because quadratic curves are employed. If the response of any compound in either ICV/CCV differs by more than ± 20% (except for dalapon and acifluorfen which have a ± 30% limit), the data is evaluated for corrective action back to the last acceptable set of CCVs and forward to the next set of acceptable CCVs. When a CCV fails, the instrument should be recalibrated or the data properly qualified in accordance with GEN-015, Qualification of Data.

10.9 A tune performance check is injected and required to pass DFTPP, tailing, and degradation criteria before initial analysis proceeds. Refer to section 9.7.1 for specific tune performance check criteria. Subsequent tune performance checks are analyzed every 12 hours or less, typically alongside CCV standards, and are required to pass DFTPP criteria and should pass tailing and degradation criteria.

10.10 A second source calibration verification (SCV) standard is analyzed with every analysis sequence immediately after the ICAL. Two SCV concentrations are used (200 and 1000 ng/mL) because quadratic curves are employed. Acceptance limits are the same as those used for ICVs/CCVs. .

10.11 Surrogates are added to every sample. Surrogate control limits are generated from LIMS on at least a yearly basis. If a surrogate recovery falls outside of the control limits, the sample is re-extracted and reanalyzed, or the data is qualified in accordance with GEN-015, Qualification of Data.

10.12 Internal standards are added to every injection vial. Internal standard limits are -50% to 200% from the first injected ICV or CCV of the sequence (typically the 500 ng/mL CCV). If a limit is exceeded, the sample must to be re-aliquoted and re-injected.

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11 METHOD PERFORMANCE

11.1 The LOQ for this methods is the level 2 standard of the calibration curve.

11.2 LODs for water and soil samples are listed in Table 1A and Table 1B, respectively. LODs are either run or verified on an annual basis as described by GEN-019.

11.3 Demonstration of capability (DOC) data for water and soil are provided in Table 2.

12 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

12.1 Out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and included as part of the raw data package.

12.2 The client must be notified when it becomes apparent that an out-of-control situation may lead to unacceptable data.

12.3 Analytical results are qualified according to ECCS SOP GEN-015.

13 WASTE MANAGEMENT / POLLUTION PREVENTION

13.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment.

13.2 All ECCS employees are required to abide by the ECCS Chemical Hygiene Plan document.

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14 REFERENCES

14.1 Method 8151A; SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update II; U.S. EPA, OSWER; December 1996.

14.2 Method 8321B; SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update II; U.S. EPA, OSWER; November 1999.

14.3 Taylor, V.; Hickey, D. M.; Marsden, P.J. "Single Laboratory Validation of EPA Method 8140"; U.S. EPA, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, 1987; EPA-600/4-87-009.

14.4 Method 8270C; SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update II; U.S. EPA, OSWER; December 1996.

14.5 Method 8270D; SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update II; U.S. EPA, OSWER; February 2007.

14.6 Method 3510C; SW-846, Separatory funnel extraction.

14.7 Method 3511; Organic Compounds in Water by Micro-extraction; SW846; November 2002.

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TABLE 1A

LIMIT OF DETECTION

WATER SAMPLES

Compound	Spike Conc. (µg/L)	Average Rec (%)	Standard Deviation	MDL (µg/L)	RSD (%)	Compared To Spike
2,4-D	0.20	126	0.014	0.050	5.6	4.7
2,4-DB	0.20	112	0.009	0.026	3.9	7.6
2,4,5-T	0.20	119	0.023	0.068	9.5	2.9
2,4,5-TP (Silvex)	0.20	101	0.008	0.025	4.2	7.9
Acifluorfen	1.0	90	0.124	0.37	14	2.7
Bentazon	0.10	102	0.010	0.029	9.5	3.4
Bromoxynil	0.10	109	0.007	0.021	6.3	4.9
Chloramben				NA		
Clopyralid				NA		
Dacthal (DCPA)	0.20	92	0.008	0.023	4.2	8.7
Dalapon	0.80	65	0.056	0.17	11	4.8
Dicamba	0.20	91	0.009	0.026	4.7	7.8
Dichlorprop	0.20	101	0.008	0.023	3.8	8.6
Diclofop				NA		
Dinoseb	0.20	79	0.016	0.049	10	4.1
MCPA	0.20	100	0.010	0.029	4.8	6.9
MCPP	0.20	95	0.008	0.025	4.4	8.1
4-Nitrophenol	0.20	139	0.017	0.051	12	2.0
Pentachlorophenol	0.20	88	0.017	0.050	9.5	4.0
Picloram	0.10	120	0.009	0.028	7.9	3.5
Triclopyr	0.20	106	0.012	0.037	5.8	4.7
2,4-D-d_5 (Surr)		80	0.024	3.1		
DCAA (Surr)		71	0.062	8.8		

Data Source: A9B1904 – 02/19/09

NA = Not available.

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TABLE 1B

LIMIT OF DETECTION

SOIL SAMPLES

Compound	Spike (µg/kg)	Average Recovery (%)	Standard Deviation	RSD (%)	MDL (µg/kg)	Compared To Spike
2,4,5-T	40	115	7.09	15	21	1.9
2,4,5-TP (Silvex)	40	107	2.14	5.0	6.4	6.2
2,4-D	40	122	4.36	8.9	13	3.1
2,4-DB	40	97.6	2.19	5.6	6.6	6.1
Acifluorfen	200	84.8	5.75	3.4	17	11.6
Bentazon	20	104	2.02	9.7	6.0	3.3
Bromoxynil	20	84.9	1.87	11	5.6	3.6
Chloramben					NA	
Clopyralid	40	88.5	3.70	10	11	3.6
Dacthal	40	90.6	2.21	6.1	6.6	6.0
Dalapon	40	82.1	8.13	6.2	24	6.6
Dicamba	40	97.1	3.17	8.2	9.5	4.2
Dichlorprop	40	96.9	2.54	6.5	7.6	5.3
Diclofop Acid	40	93.8	2.48	6.6	7.4	5.4
Dinoseb	40	85.8	3.30	9.6	9.9	4.0
MCPA	40	86.9	3.72	11	11	3.6
MCPP	40	102	2.70	6.6	8.1	4.9
Pentachlorophenol	40	78.8	2.64	8.4	7.9	5.1
Picloram	20	85.4	1.24	7.3	3.7	5.4
Trichlopyr	40	99.3	2.12	5.4	6.4	6.3
2,4-D-d_5 (Surr)		77.9	0.05	5.9		
DCAA (Surr)		79.2	0.04	5.6		

Data Source: A9G2001 – 07/20/09

NA = Not Available

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TABLE 2

TYPICAL DOCS

	SILICA SAND[1]			WATER[2]		
Compound	Spike Conc. (µg/kg)	Average Rec (%)	RSD (%)	Spike Conc. (µg/L)	Average Rec (%)	RSD (%)
2,4,5-T	500	100	14	5.0	106	6.2
2,4,5-TP	500	100	8.9	5.0	112	4.3
2,4-D	500	102	4.7	5.0	106	7.3
2,4-DB	500	107	8.6	5.0	105	4.9
4-Nitrophenol	250	100	10	2.5	118	4.8
Acifluorfen	2500	89.9	5.8	20	98.5	9.4
Bentazon	250	110	12	2.5	120	6.5
Bromoxynil	250	92.5	8.1	2.5	109	6.5
Dalapon	2000	86.2	10	25	102	3.9
DCPA	500	91.6	9.1	5.0	105	5.8
Dicamba	500	102	12	5.0	106	5.4
Dichlorprop	500	108	9.1	5.0	114	5.7
Dinoseb	500	92.8	10	5.0	109	6.9
MCPA	500	104	11	5.0	104	6.4
MCPP	500	95.6	8.1	5.0	94.5	9.6
Pentachlorophenol	500	96.6	9.9	5.0	101	7.2
Picloram	250	83.2	11	2.5	92.4	13
Trichlopyr	500	106	11	5.0	121	3.3

[1] Data Source: GC-1968; 03/28/08

[2] Data Source: GC-1964; 03/28/08

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TABLE 3

CHARACTERISTIC IONS FOR ACID HERBICIDES

Compound	Quantitation Ion (m/z)	Qualifier Ions (m/z)
2,4-D	234	199, 175, 111
2,4-DB	101	162, 231, 59
2,4,5-T	235	233, 268, 209
2,4,5-TP (Silvex)	196	198, 282, 223
Acifluorfen	344	223, 207, 75
Bentazon	212	254, 105, 175
Bromoxynil	291	293, 276, 88
Chloramben	188	219, 190, 221
Clopyralid	147	174, 110, 205
Dacthal	301	303, 332, 221
Dalapon	97	99, 121, 59
Dicamba	205	201, 234, 236
Dichlorprop	162	164, 189, 248
Diclofop	253	340, 281, 120
Dinoseb	225	254, 195, 77
MCPA	214	155, 141, 77
MCPP	169	228, 142, 77
4-Nitrophenol	153	123, 77, 92
Pentachlorophenol	265	267, 280, 237
Picloram	196	198, 223, 254
Triclopyr	210	212, 269, 146
<u>Surrogate</u>		
DCAA	159	161, 183, 218
2,4-D-d_5	204	206, 239, 241
<u>Internal Standard</u>		
2,4,5-T-d_4	276	237, 217, 239

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TABLE 4A

SPIKING STOCK STANDARD
ACID FORM

Compound	Concentration (µg/mL)
2,4-D	20
2,4-DB	20
2,4,5-T	20
2,4,5-TP (Silvex)	20
Acifluorfen	100
Bentazon	10
Bromoxynil	10
Chloramben	10
Clopyralid	20
Dacthal	20
Dalapon	80
Dicamba	20
Dichlorprop	20
Diclofop	20
Dinoseb	20
MCPA	20
MCPB	20
MCPP	20
4-Nitrophenol	10
Pentachlorophenol	20
Picloram	10
Triclopyr	20

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TABLE 4B

PRIMARY STOCK STANDARD

ICAL METHYL ESTER

Compound	Concentration (µg/mL)
2,4-D	20
2,4-DB	20
2,4,5-T	40
2,4,5-TP (Silvex)	20
Acifluorfen	80
Bentazon	20
Bromoxynil	20
Chloramben	20
Clopyralid	20
Dacthal	20
Dalapon	200
Dicamba	20
Dichlorprop	20
Diclofop	20
Dinoseb	20
MCPA	20
MCPP	20
4-Nitrophenol	20
Pentachlorophenol	20
Picloram	20
Triclopyr	20
DCAA (Surr)	80

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TABLE 4C

SECOND SOURCE STOCK STANDARD

METHYL ESTER

Compound	Concentration (µg/mL)
2,4-D	20
2,4-DB	20
2,4,5-T	40
2,4,5-TP (Silvex)	20
Acifluorfen	80
Bentazon	20
Bromoxynil	20
Chloramben	20
Clopyralid	Not Included
Dacthal	20
Dalapon	200
Dicamba	20
Dichlorprop	20
Diclofop	20
Dinoseb	20
MCPA	20
MCPP	20
4-Nitrophenol	20
Pentachlorophenol	20
Picloram	20
Triclopyr	20
DCAA (Surr)	80

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TABLE 5

NO INTERMDEDIATE CONCENTRATIONS FOR THIS METHOD.

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TABLE 6A

ICAL STANDARD CONCENTRATIONS

METHYL ESTER

	Concentration (ng/mL)						
Compound	L-1	L-2	L-3	L-4	L-5	L-6	L-7
2,4-D	10	20	50	100	200	500	1000
2,4-DB	10	20	50	100	200	500	1000
2,4,5-T	20	40	100	200	400	1000	2000
2,4,5-TP (Silvex)	10	20	50	100	200	500	1000
Acifluorfen	40	80	200	400	800	2000	4000
Bentazon	10	20	50	100	200	500	1000
Bromoxynil	10	20	50	100	200	500	1000
Chloramben	10	20	50	100	200	500	1000
Clopyralid	10	20	50	100	200	500	1000
Dacthal	10	20	50	100	200	500	1000
Dalapon	100	200	500	1000	2000	5000	10000
Dicamba	10	20	50	100	200	500	1000
Dichlorprop	10	20	50	100	200	500	1000
Diclofop	10	20	50	100	200	500	1000
Dinoseb	10	20	50	100	200	500	1000
MCPA	10	20	50	100	200	500	1000
MCPP	10	20	50	100	200	500	1000
4-Nitrophenol	10	20	50	100	200	500	1000
Pentachlorophenol	10	20	50	100	200	500	1000
Picloram	10	20	50	100	200	500	1000
Triclopyr	10	20	50	100	200	500	1000
DCAA (Surr.)	40	80	200	400	800	2000	4000
2,4-D-d_5 (Surr.)	10	20	50	100	200	500	1000
Source Std. ID	L-4	L-5	L-6	7.4.2	7.4.2	7.4.2	7.4.2
mL Added	10	10	10	1	1	2.5	5
Surrogate ID	-	-	-	7.7.4	7.7.4	7.7.4	7.7.4
mL Added	-	-	-	0.8	0.8	2	4
Final Volume (mL)	100	100	100	200	100	100	100

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TABLE 6B

SECOND SOURCE STANDARD

METHYL ESTER

Compound	Concentration (ng/mL)
2,4-D	200
2,4-DB	200
2,4,5-T	400
2,4,5-TP (Silvex)	200
Acifluorfen	800
Bentazon	200
Bromoxynil	200
Chloramben	200
Clopyralid	Not Included
Dacthal	200
Dalapon	2000
Dicamba	200
Dichlorprop	200
Diclofop	200
Dinoseb	200
MCPA	200
MCPP	200
4-Nitrophenol	200
Pentachlorophenol	200
Picloram	200
Triclopyr	200
DCAA (Surr)	800
2,4-D-d_5 (Surr)	200

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TABLE 6C

WORKING SURROGATE STANDARD PREPARATION

	2,4-D-d_5	DCAA
Stock Solution Concentration (µg/mL)	1000	1000
mL added	2.5	2.5
Final Volume (mL)	100	100
Final Concentration (µg/mL)	25.0	25.0

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TABLE 6D

STANDARD CURVE ENTRY FOR CHEMSTATION SOFTWARE

CORRECTING TO ACID FORM EQUIVALENTS

	Concentration (ng/mL)						
Compound	L-1	L-2	L-3	L-4	L-5	L-6	L-7
2,4-D	9.405	18.81	47.02	94.05	188.1	470.2	940.5
2,4-DB	9.466	18.93	47.33	94.66	189.3	473.3	946.6
2,4,5-T	18.96	37.92	94.80	189.6	379.2	948.0	1896
2,4,5-TP (Silvex)	9.507	19.01	47.53	95.07	190.1	475.3	950.7
Acifluorfen	38.51	77.02	192.6	385.1	770.2	1926	3851
Bentazon	9.450	18.90	47.25	94.50	189.0	472.5	945.0
Bromoxynil	9.518	19.04	47.59	95.18	190.4	475.9	951.8
Chloramben	9.363	18.73	46.82	93.63	187.3	468.2	936.3
Clopyralid	9.320	18.64	46.60	93.20	186.4	466.0	932.0
Dacthal	9.157	18.31	45.78	91.57	183.1	457.8	915.7
Dalapon	91.08	182.2	455.4	910.8	182.2	4554	9108
Dicamba	9.405	18.81	47.02	94.05	188.1	470.2	940.5
Dichlorprop	9.438	18.88	47.19	94.38	188.8	471.9	943.8
Diclofop	9.590	19.18	47.95	95.90	191.8	479.5	959.0
Dinoseb	9.501	19.00	47.51	95.01	190.0	475.1	950.1
MCPA	9.346	18.69	46.73	93.46	186.9	467.3	934.6
MCPP	9.386	18.77	46.93	93.86	187.7	469.3	938.6
4-Nitrophenol	9.085	18.17	45.43	90.85	181.7	454.3	908.5
Pentachlorophenol	9.501	19.00	47.51	95.01	190.0	475.1	950.1
Picloram	9.452	18.90	47.26	94.52	189.0	472.6	945.2
Triclopyr	9.482	18.96	47.41	94.82	189.6	474.1	948.2
DCAA (Surr.)	37.43	74.86	187.2	374.3	748.6	1872	3743
2,4-D-d_5 (Surr.)	10	20	50	100	200	500	1000

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TABLE 7

DFTPP TUNING CRITERIA

Mass Ion Abundance Criteria
51 10-80% of Base Peak
68 < 2% of mass 69
70 < 2% of mass 69
127 10-80% of Base Peak
197 < 2% of mass 198
198 Base peak, or > 50% of Mass 442
199 5-9% of mass 198
275 10-60% of Base Peak
365 > 1% of mass 198
441 present but < 24% of mass 442
442 Base Peak, or > 50% of mass 198
443 15-24% of mass 442

From Method 8270D.

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FIGURE 1

L-6 SUMMARY REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\A9E1504\008.D Vial: 8
Acq On : 15 May 2009 7:11 pm Operator: cps
Sample : A9E1504-CAL6 Inst : 3341A0138
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Aug 19 14:10 2009 Quant Results File: A9E1504.RES

Quant Method : D:\HPCHEM\1\METHODS\A9E1504.M (RTE Integrator)
Title : 8151 by GCMS
Last Update : Wed Aug 19 14:09:12 2009
Response via : Initial Calibration
DataAcq Meth : A9E1504

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) 2,4,5-T-d4	13.80	239	74535	1.00		0.00
System Monitoring Compounds						
3) DCAA	11.50	159	2603933	1806.94	ng/ml	0.00
11) 2,4-D-d5	12.76	204	229854	467.37	ng/ml	-0.02
Spiked Amount 200.000			Recovery =	233.69%		
Target Compounds						Qvalue
2) Dalapon	4.45	97	2803768	4454.79	ng/ml	99
4) 4-Nitrophenol	11.58	153	349815	449.46	ng/ml	97
5) Clopyralid	11.58	147	428995	455.97	ng/ml	99
6) Dicamba	11.73	205	323824	458.05	ng/ml	97
7) MCPP	11.88	169	364692	461.77	ng/ml	97
8) MCPA	12.11	214	253438	458.49	ng/ml	99
9) Dichlorprop	12.42	162	480690	456.21	ng/ml	97
10) Pentachlorophenol	12.54	265	239293	469.81	ng/ml	97
12) 2,4-D	12.77	234	145196	457.38	ng/ml	95
13) Triclopyr	12.86	210	270974	458.72	ng/ml	100
14) Bromoxynil	12.90	291	283477	468.55	ng/ml	99
15) 2,4,5-TP (Silvex)	13.42	196	342563	460.57	ng/ml	98
16) 2,4,5-T	13.80	235	273119	961.57	ng/ml	98
17) Chloramben	13.85	188	344055	468.94	ng/ml	96
18) 2,4-DB	14.31	101	805331	474.79	ng/ml	97
19) Picloram	15.52	196	312873	470.32	ng/ml	99
20) Bentazon	15.60	212	418953	465.37	ng/ml	98
21) Dacthal	15.73	301	426334	451.05	ng/ml	98
22) Dinoseb	15.88	225	318666	490.03	ng/ml	98
23) Diclofop Acid	17.88	253	221755	487.77	ng/ml	98
24) Acifluorfen	18.66	344	328217	1995.31	ng/ml	89

(#) = qualifier out of range (m) = manual integration
008.D A9E1504.M Wed Aug 19 14:10:14 2009 1 Page 1

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FIGURE 1

L-6 SUMMARY REPORT (CONT)

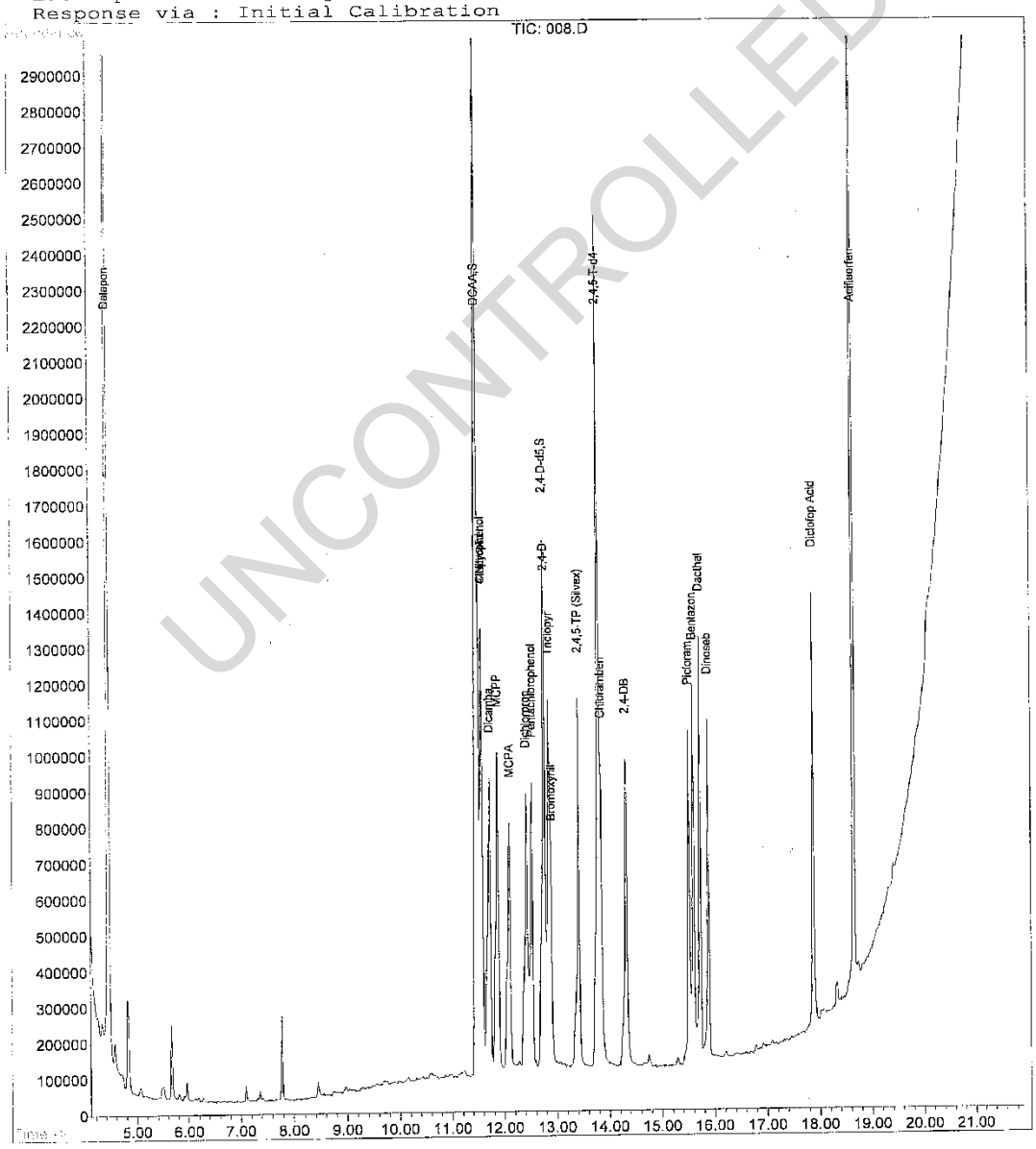
Quantitation Report

Data File : D:\HPCHEM\1\DATA\A9E1504\008.D
Acq On : 15 May 2009 7:11 pm
Sample : A9E1504-CAL6
Misc :
MS Integration Params: RTEINT.P
Quant Time: Aug 19 14:10 2009

Vial: 8
Operator: cps
Inst : 3341A0138
Multiplr: 1.00

Quant Results File: A9E1504.RES

Method : D:\HPCHEM\1\METHODS\A9E1504.M (RTE Integrator)
Title : 8151 by GCMS
Last Update : Wed Aug 19 14:09:12 2009
Response via : Initial Calibration



008.D A9E1504.M Wed Aug 19 14:10:16 2009 1 Page 2

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FIGURE 2

DIAZOMETHANE GLASSWARE APPARATUS

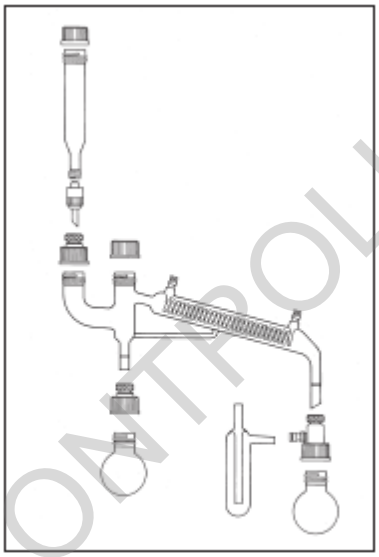

Fig. 3 Typical set-up with Diazald Glassware Set (heating and cooling baths are not included).

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FIGURE 3

MAXIMUM SENSITIVITY AUTOTUNE REPORT

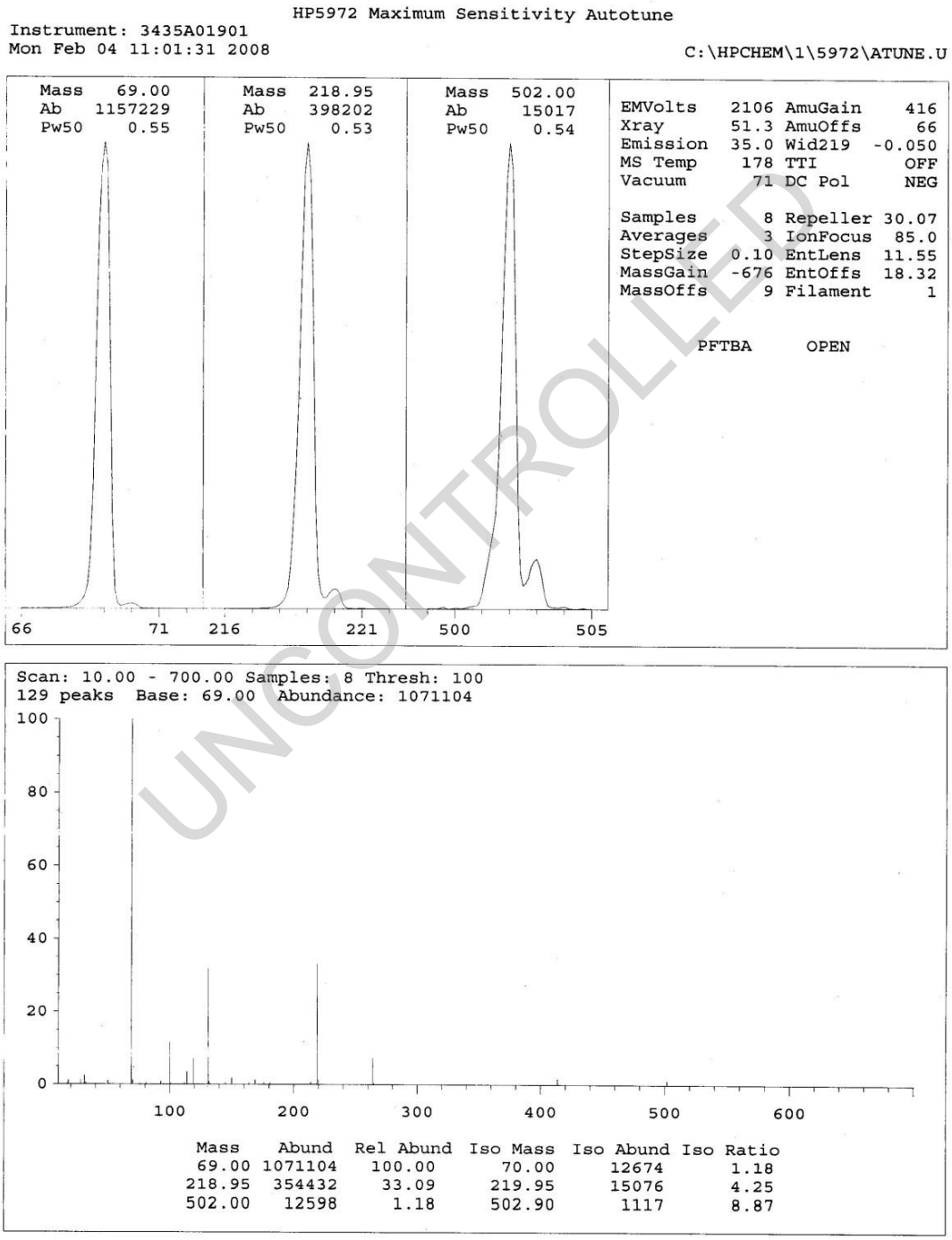

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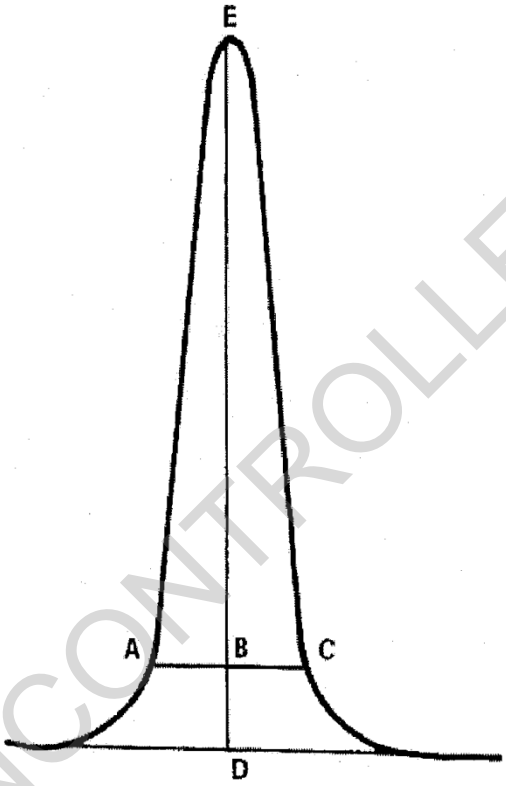
FIGURE 4

TAILING FACTOR CALCULATION



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$$TailingFactor = \frac{BC}{AB}$$

Example Calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm

$$AB = 11\ mm$$
$$BC = 12\ mm$$

$$Therefore:\ TailingFactor = \frac{12}{11} = 1.1$$

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

01/26/16

Chris Sauer, GC/MS Team Leader — Date

01/26/16

Patrick Letterer, Quality Manager — Date

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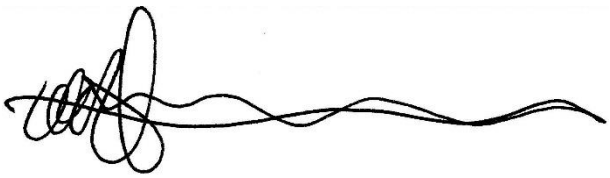
Approved By:



01/26/16

Nicholas K. Nigro, General Manager — Date

ORGANONITROGEN/ORGANOPHOSPHORUS COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) BASED ON SW846 METHOD 8270D

1 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is a capillary GC/MS method used to determine the concentration of organo-nitrogen (ON) and organo-phosphorus (OP) pesticide compounds in soil and aqueous matrices. This method has been validated for the analysis of the following compounds:

Target Analyte	CAS No.
*Acetochlor	34256-82-1
*Alachlor	15972-60-8
Ametryn	834-12-8
*Atrazine	1912-24-9
*des-ethyl-	06190-65-4
*de-isopropyl-	01007-28-9
Bromacil	314-40-9
*Butylate	2008-41-5
*Chlorpyrifos	2921-88-2
Chlorthalonil	1897-45-6
*Cyanazine	21725-46-2
Diazinon	333-41-5
*Dimethenamid	87674-68-8
*EPTC	759-94-4
*Ethalfluralin	55283-68-6
*Fonophos	944-22-9
Hexazinone	51235-04-2
Malathion	121-75-5
Methyl Parathion	298-00-0
*Metolachlor	51218-45-2
*Metribuzin	21087-64-9
Parathion	56-38-2
*Pendimethalin	40487-42-1
*Phorate	298-02-02
*Prometon	1610-18-0
Prometryn	7287-19-6
*Propachlor	1918-16-7
*Propazine	139-40-2
*Simazine	122-34-9
*Terbufos	13071-79-9
*Triallate	2303-17-5
*Trifluralin	1582-09-8

Target Analyte	CAS No.
Benfluralin	1861-40-1
Carbaryl	63-25-2
Carbofuran	1563-66-2
Clomazone	81777-89-1
Dichlobenil	1194-65-6
Dichlorvos	62-73-7
Dimethoate	60-51-5
Disulfoton	298-04-4
Ethion	563-12-2
Ethoprop	13194-48-4
Fenuron	101-42-8
Isofenphos	25311-71-1
Linuron	330-55-2
Napropamide	15299-99-7
Norflurazon	27314-13-2
Phosmet	732-11-6
Prodiamine	29091-21-2
Profenphos	41198-08-7
Promecarb	2631-37-0
Propanil	709-98-8
Propham	122-42-9
Propoxur	114-26-1
Siduron	1982-49-6
Simetryn	1014-70-6
Tebuthiuron	34014-18-1
Terbacil	5902-51-2
Terbuthylazine	5915-41-3
Terbutryn	86-50-0
Metalaxyl	57837-19-1

* Wisconsin and/or Minnesota Department of Agriculture target compound

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1.2 The analytes include several classes of compounds with organo-nitrogen and organophosphate pesticides used primarily as agricultural insecticides, herbicides and fungicides.

1.3 Compound identification is done by retention time and mass spectra of each individual standard reference compared to compounds in the unknown sample. Usually each compound has a primary quantitation ion with two secondary identification ions.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of HP MSD systems. The analyst must understand its spectra generated by the MS and how to use the spectra for identification and quantitation.

1.5 This method is based upon Method 8270D with the exception that samples from Illinois use Method 8270C DFTPP tuning criteria. All other samples follow Method 8270D tuning criteria.

2 SUMMARY OF METHOD

2.1 This method provides extraction and GC/MS conditions for the analysis of a broad range of pesticides from soil and water.

2.2 Soils are extracted with a mixture of 80% iso-octane/20% acetone, once the sample has been dried with sodium sulfate. Aqueous samples are extracted with dichloromethane and ethyl acetate. Aqueous extracts are concentrated by evaporation of the solvent and made to a final volume of 5 mL with 80% iso-octane/20% acetone.

2.3 A measured amount of the extract is transferred to an auto-sampler vial, internal standard added, and analysis performed by GC/MS. Calibration is accomplished by using an internal standard method, comparing the response of a major (quantitation) ion relative to an internal standard using a multi-point standard curve.

2.4 Quantitation is accomplished by reverse extrapolation obtaining a µg/mL or ng/mL concentration in the extract.

2.5 Final results are reported with the ECCS laboratory information management system (LIMS).

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

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4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in extracted ion chromatograms. Reagent blanks are analyzed with every analysis batch to monitor for potential interferences with a goal of the procedure being interference free.

4.2 Samples may contain interfering non-target analytes which may result in an inability to reach necessary reporting limits. See Section 4.4.

4.3 Samples may also contain high levels of target analytes resulting in elevated reporting limits for other target analytes.

4.4 GC/MS methods have limitations when a sample contains high levels of non-target compounds such as non-descript hydrocarbons. As a result, elevated reporting may occur for some target analytes. For hydrocarbon interference, the symptom is the total ion chromatogram often has a hump. An alternative approach to eliminate the hydrocarbon interference may be analysis in accordance with LAM-020 8141NP SOP. The application of GC with nitrogen phosphorus detection (NPD) will often provide more accurate quantitation of the target analytes. If the sample cannot be re-extracted in accordance with the LAM-020, the LAM-006 extract will require two of the compounds to be quantitated by a combination of GC/NP and GC/MS because atrazine-d5 and parathion-d10 co-elute with the non-deuterated analytes.

4.5 Certain groundwater samples have exhibited an interference with the quantitation of alachlor using the normal quantitation ion of 160. This method provides for an alternative quantitation ion of 237 to be used when the interference is present.

4.6 In the case of interference with the quantitation ion for any analyte, an alternative ion may be selected for quantitation. It is acceptable to use a single point calibration for this quantitation as long as the result is properly qualified with an "E1" in accordance with GEN-015.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure. Lab coats are recommended.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or

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moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 GC/MS System

6.1.1 Gas Chromatograph: HP 5890 with split/splitless injector

6.1.2 Detector: HP 5972 mass selective detector

6.1.3 Autosampler: Leap Technologies CTC A200S

6.1.4 Data System: Agilent MSD Productivity Chemstation w/ Enviroquant

6.2 GC/MS Supplies List

6.2.1 Column: Restek RTX-200 30 m x 0.32 mm, ID 0.25 µm film

6.2.2 Seals: Dual Vespel ring inlet seal, Restek Cat. # 21239 or similar

6.2.3 Septa: LB-2 septa 11mm, Supelco # 164742

6.2.4 Ferrules: 1/16 x 0.5mm Vespel/ graphite Restek Cat. #20231/ 20249

6.2.5 Liners: Gooseneck splitless liner 4mm x 6.5 x 78.5 Restek Cat. #20799

6.3 Computer Hardware

6.3.1 HP Compaq DC7600 or equivalent

6.4 Computer Software

6.4.1 Microsoft Windows 2000 operating system or equivalent current version

6.4.2 Microsoft Office 2007 or equivalent current version

6.4.3 Agilent MSD Productivity Chemstation with EnviroQuant

6.4.4 Promium Element Data System

6.5 Balances:

6.5.1 Top loader - capable of weighing to 0.01 g

6.5.2 Analytical - capable of weighing to 0.0001 g

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6.6 Vials:

6.6.1 2 mL amber auto-sampler vials LSDC Cat. # 20011A-1232 and PTFE/silicone aluminum seals, Part #21120-11

6.7 Syringes: Various sizes, 10-1000 µL, Gastight

6.8 Air displacement pipette, 200-1000 µL variable, and 1 mL tips (Eppendorf or equivalent)

6.9 Repeater Plus with Combitips of 1ml or 2.5 ml

6.10 Optifix solvent dispensers capable of 10 mL, 50 mL and 100 mL volume (EM Science or equivalent)

6.11 Compressed gas:

6.11.1 Helium, Grade 5

6.11.2 Nitrogen, Grade 5

6.12 Refrigerator capable of maintaining 4 °C

6.13 Freezer capable of maintaining temperatures below -15 °C

7 REAGENTS

7.1 Solvents

7.1.1 Carbon disulfide, CS_2 – pesticide quality or equivalent.

7.1.2 Acetone, CH_3COCH_3 - pesticide quality or equivalent

7.1.3 Iso-octane, C_8H_{18} – pesticide quality or equivalent

7.1.4 80% iso-octane/20% acetone: Prepare 4 L by mixing 3200 mL of iso-octane with 800 mL of acetone in a 4 L solvent bottle.

7.1.5 Chloroform, $CHCl_3$ – EMD CX1055-13

7.1.6 Internal standard diluent – 70% carbon disulfide/30% chloroform: Combine 140 mL of carbon disulfide and 60 mL of chloroform in a bottle and mix well. Discard this solution when the internal standard preparation is complete.

7.2 Solid Reagents

7.3 Acids and Bases

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7.4 Stock Standards

7.4.1 GC/MS Mix 1 is purchased from Absolute Standards at a concentration of 20 µg/mL (Part # 93725). See Table 4-1 for a list of the 32 components.

7.4.2 GC/MS Mix 2 is purchased from Absolute Standards at a concentration of 20 µg/mL (Part # 94174). See Table 4-2 for a list of the 29 components.

7.4.3 Stock standards are stored frozen and assigned an expiration date of 2 years from receipt.

7.5 Intermediate Standards – Not applicable to this method

7.6 Calibration Standards

7.6.1 Initial calibration (ICAL)

7.6.1.1 Example preparation: ICAL Level 8 (2000 ng/mL)

7.6.1.1.1 Using a 10 mL volumetric pipette, aliquot 10 mL of Mix 1 (Section 7.4.1) and 10 mL of Mix 2 (Section 7.4.2) into a 100 mL volumetric flask. Using a 20 mL volumetric pipette, aliquot 20 mL of surrogate spike mix at 10 µg/mL (Section 7.7.3) into the same 100 mL volumetric flask.

7.6.1.1.2 Make to volume with 80% iso-octane/20% acetone.

7.6.1.2 Refer to Table 6 for the preparation of the remaining 7 ICAL levels.

7.6.1.3 Transfer to amber VOA vials labeled with a LIMS number, store frozen, and assign a 1 year expiration date.

7.7 Surrogate

7.7.1 Surrogate neat standards

7.7.1.1 Surrogate, atrazine-d5, CDN Isotopes # D-4389

7.7.1.2 Surrogate, parathion-d10, CDN Isotopes # D-4288

7.7.1.3 Surrogate, triphenylphosphate (TPP), Chem. Service

7.7.2 Surrogate stock solutions

7.7.2.1 Atrazine-d5 (500 µg/mL) - Weigh 0.050 g neat material (Section 7.7.1.1) using an analytical balance into a 100 mL volumetric flask. Dilute to volume with acetone.

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7.7.2.2 Parathion-d10 (500 µg/mL) - Weigh 0.050 g neat material (Section 7.7.1.2) using an analytical balance into a 100 mL volumetric flask. Dilute to volume with acetone.

7.7.2.3 TPP (1000 µg/mL) - Weigh 0.100 g neat material (Section 7.7.1.3) using an analytical balance into a 100 mL volumetric flask. Dilute to volume with acetone.

7.7.2.4 Transfer contents of each stock surrogate solution to amber 40 mL VOA vials labeled with LIMS numbers, store frozen, and assign an expiration date of two years from preparation.

7.7.3 Surrogate spiking solution (10 µg/mL)

7.7.3.1 Using volumetric pipettes aliquot 4 mL of parathion-d10 500 µg/mL (Section 7.7.2.2), 4 mL of atrazine-d10 500 µg/mL (Section 7.7.2.1), and 2 ml of TPP 1000 µg/mL (Section 7.7.2.3) into a 200 mL volumetric flask. Make to volume with acetone. Transfer to amber VOA vials labeled with LIMS numbers, store frozen, and assign an expiration date of one year from preparation. See Table 7-1 for dilution scheme. Store frozen and assign a one year from preparation expiration date.

7.7.3.2 Surrogate spiking volume is usually 100 µL for soils and 50 µL for waters.

7.8 Laboratory Control Sample (LCS) Spiking Solutions

7.8.1 The performance of the extraction, cleanup (when used) and analytical system is monitored by spiking reagent water or silica sand with the compounds of interest.

7.8.2 The LCS spiking solutions are the Mix 1 and Mix 2 stock standard solutions (See Section 7.4.1 and 7.4.2).

7.8.3 LCS spiking volume is usually 100 µL for soils and 50 µL for waters.

7.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Spiking Solutions

7.9.1 Precision and the effect of the matrix are monitored by spiking a sample in duplicate with the compounds of interest.

7.9.2 The MS/MSD spiking solutions are the same Mix 1 and Mix 2 stock standard solutions used for spiking the LCS (See Section 7.4.1 and 7.4.2).

7.9.3 MS/MSD spiking volume is usually 100 µL for soils and 50 µL for waters.

7.10 Second Source Standard Solution

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7.10.1 The second source stock standard solutions are purchased as custom mixes from Ultra Scientific (Part #s CUS-14980; CUS-14981 and CUS 11342) with each analyte at a concentration of 20 µg/mL.

7.10.2 Second source injection standards (100 and 1000 ng/mL)

7.10.2.1 100 ng/mL: Using a 1 mL Gastight syringe aliquot 0.5 mL of each second source stock standard solution (Section 7.10.1) into a 100 mL volumetric flask and make to volume with 80% iso-octane/20% acetone.

7.10.2.2 1000 ng/mL: Using a 5 mL volumetric, pipette 5.0 mL of each second source stock solutions (Section 7.10.1) into a 100 mL volumetric flask and make to volume with 80% iso-octane/20%acetone.

7.10.2.3 Transfer to amber VOA vials labeled with LIMS numbers, store frozen, and assign an expiration date of one year from preparation.

7.11 Internal Standard

7.11.1 Internal standard neat standards

7.11.1.1 EPTC-d14, CDN Isotopes # D-5645

7.11.1.2 Phorate-d10, CDN Isotopes # D-5817

7.11.1.3 Simazine-d10, CDN Isotopes # D-5654

7.11.2 Stock internal standard solutions

7.11.2.1 EPTC-d14 (500 µg/mL): Weigh 0.050 g neat material using an analytical balance into a 100 mL volumetric flask. Add 30 mL of 70% carbon disulfide/30% chloroform to dissolve and make to volume with acetone. Transfer to amber VOA vials labeled with LIMS numbers, store frozen, and assign an expiration date of two years from preparation.

7.11.2.2 Phorate d-10 (500 µg/mL): Weigh 0.05 g neat material using an analytical balance into a 100 mL volumetric flask. Add 30 mL of 70% carbon disulfide/30% chloroform to dissolve and make to volume with acetone. Transfer contents to 3 labeled 40 mL VOA vials and assign an expiration date of two years from preparation.

7.11.2.3 Simazine-d10 (500 µg/mL): Weigh 0.05 g neat material using an analytical balance into a 100 mL volumetric flask. Add 30 to 60 mL of 70% carbon disulfide/30% chloroform to dissolve and make to volume with 70% carbon disulfide/30% chloroform. Transfer contents to 3 labeled 40 mL VOA vials and assign an expiration date of two years from preparation.

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7.11.3 Working internal standard solution (12.5 µg/mL)

7.11.3.1 Using volumetric pipettes, aliquot 5 mL of EPTC-d14 (Section 7.11.2.1), 5 mL of simazine-d10 (Section 7.11.2.3), and 5 mL of phorate-d10 (Section 7.11.2.2) into a 200 mL volumetric. Make to volume with acetone. Transfer to amber VOA vials labeled with LIMS numbers, store frozen, and assign an expiration date of one year from preparation. See Table 7-2 for dilution scheme.

7.11.3.2 A volume of 40 µL is added to every injection vial prior to analysis on the GC/MS. The nominal concentration of the internal standard in the vial is 0.625 µg/mL.

7.12 Tune Performance Check Standard

7.12.1 Stock Tune Performance Check Solution: CLP semi-volatile tuning solution at 500 µg/mL (Absolute Part #43030). The working tuning solution is prepared at 2000 ng/mL. The solution contains DFTPP, benzidine, pentachlorophenol and 4,4'-DDT. Using a 500 µL gastight syringe, transfer 400 µL of the stock tuning solution to a 100 mL volumetric flask. Fill to the mark with 80/20 Isooctane/Acetone and transfer to amber 40 mL VOC vials. Store frozen and assign an expiration date of one year from preparation.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples should be collected in 1 L amber glass bottles with Teflon-lined caps and refrigerated at 4 °C after collection. Request one of the samples to be collected in triplicate for MS/MSD analysis. Aqueous samples should be extracted within 7 days of collection.

8.2 Soil samples should be collected in amber glass jars with Teflon-lined caps and refrigerated at 4 °C after collection. Soil samples should be extracted within 14 days of collection. Soil samples for most compounds of interest can be frozen for longer periods until extraction if regulatory rules allow.

8.3 Pesticide mix tank waste samples are often highly contaminated and should be analyzed by dilute and shoot techniques (GEN-007, Waste Dilution)

8.4 Store sample extracts in a freezer and analyze within 40 days of extraction.

9 PROCEDURE

9.1 Preparation of Samples – Choose the appropriate preparation method below.

9.1.1 Water samples

9.1.1.1 PRE-001, Separatory Funnel Extraction

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9.1.1.2 PRE-002, Self-Contained Water Extraction

9.1.2 Soil/sediment samples

9.1.2.1 PRE-003, Micro-Scale Soil Extraction

9.1.3 Wipe Samples

9.1.3.1 PRE-007, Wipe Sample Extraction

9.1.4 Waste

9.1.4.1 PRE-006, Waste Dilution

9.2 Clean-up of Samples – Clean-up procedures are not applicable to this method.

9.3 Instrument Conditions

9.3.1 GC Conditions

Column:	RTX-200MS, 30 m x 0.32 mm ID, 0.25µ film (Restek part #15624)
Guard Column:	5m x 0.32 mm ID, 0.25u film of RTX-200MS
Alternate Guard Column:	5m x 0.32mm ID RXI Guard Column (Restek part# 10039-600)
Liner:	4 mm standard gooseneck
Injection Seal:	Stainless steel
Head Pressure:	5 PSI at 80 °C
Column Flow:	1.5 mL/minute
Split Flow:	35 mL/minute
Purge Valve:	Initial off
On Time:	4.0 minutes
Transfer Line Temperature:	320 °C
Injector Temperature:	300 °C
GC Temperature Program	
Initial Temperature:	80 °C
Initial Hold:	4.0 minutes
1st Ramp:	20 °C/minute
1st Final Temperature:	120 °C
1st Hold Time:	1.0 minutes
2nd Ramp:	6 °C/minute
2nd Final Temperature:	160 °C
2nd Hold Time:	2 minutes
3rd Ramp:	20 °C/minute
3rd Final Temperature	320 °C

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3rd Final Hold Time: 1.0 minutes

9.3.2 MS conditions

Tune:	Maximum sensitivity autotune
EM Voltage:	Set to produce response of m/z 69 at 4.5 million counts
Scan Range:	45-550 amu
Scan Time:	0.83 scans/second
Solvent Delay:	6.5 minutes

9.3.3 The mass spectrometer is tuned prior to calibration using the maximum sensitivity autotune program. This program is designed to adjust ion ratios to match those found in mass spectral library reference spectra while also providing high MS sensitivity. A valid autotune should provide peak widths of < 0.55 amu with mass assignments not differing by more than 0.10 amu for masses 69, 219, and 502 from the PFTBA (perfluorotributylamine) tuning compound. The relative intensities for mass/charge 69, 219, and 502 should be 100%, 25-45%, and 0.2-4%, respectively. Isotope masses should be present for mass/charge 70, 220, and 503. Figure 2 shows a typical report for a successful maximum sensitivity autotune.

9.4 Preventive Maintenance/Troubleshooting

9.4.1 Preventative maintenance should be performed prior to the start of each analytical sequence. This includes clipping the front end of the guard column and replacing the septa, inlet liner, inlet seal and their associated o-rings and ferrules.

9.4.2 System performance can be evaluated by running low level and mid level ICAL standards known as CRLs. If general guidelines are not met for CRL standards refer to the subsequent topics in this section for further troubleshooting.

9.4.2.1 Mid level CRL: L-5 of the ICAL. Evaluate this mid level standard for acceptable minimum areas for target compounds, surrogates and internal standards. This CRL is also used to update retentions times of analytes prior to the analysis of the low level CRL.

9.4.2.2 Low level CRL: L-1 of the ICAL. Evaluate this low level standard for acceptable peak shape and signal to noise ratio for target compounds.

9.4.2.3 Tune performance check: Evaluate this tune standard for peak tailing, compound degradation, and MS tune performance. Refer to section 9.6.1.2 for specific acceptance criteria regarding the tune performance check.

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9.4.3 If the guidelines established above have not been met, further instrument maintenance may be required to optimize system performance. The following sections outline potential steps in optimizing the GC/MS system.

9.4.3.1 Evaluate the guard column for sufficient length. If less than one meter of the guard column is left, it is recommended that it be replaced. Moreover, prior to installing a new guard column, one meter should be clipped from the analytical column.

9.4.3.2 If replacing the guard column does not correct the problem, the analytical column may then need to be replaced.

9.4.3.3 If problems still persist in the GC/MS system, refer to the next section regarding MS preventative maintenance.

9.4.4 Less routinely, the MS source should be cleaned, filaments examined/changed, and electron multipliers replaced. This may aid in the optimization of the overall GC/MS system.

9.4.5 Advanced MS troubleshooting and other topics.

9.4.5.1 Detection of leaks in the GC/MS system: the presence of a base peak at m/z 28 (N_2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of a leak is a loose transfer line nut sealing the GC capillary column to the MS transfer line. Less commonly, a break or improper alignment of the gasket between the MS source and the vacuum manifold may be the source of the leak.

9.4.5.2 Leak Checking: Leak checking may be done by lightly spraying an air duster around areas of the MS that pose the most chance for a leak. These areas include around the gasket of the vacuum manifold, the seal between the MS and the MS interface, and the transfer line nut. Most commercially available air dusters (air in a can) contain difluoroethane, which will be quickly sucked into the MS system at any potential leak point. Actively scanning the MS while spraying the air duster will yield base peaks at m/z 51 and 65 at the point of any potential leak.

9.4.5.3 Water in the GC/MS system: The presence of a base peak at m/z 18 (H_2O) that is higher than m/z 69 in the tune report indicates excessive water remains in the MS manifold. The MS vacuum system does not efficiently remove water and this condition indicates that a long equilibration time is needed prior to the initial calibration. To aid in accelerating the equilibration process, ramp the GC oven to 115 °C during the pump down process. Injecting methanol or acetone can also aid in removing water.

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9.4.5.4 Electron Multiplier Voltage: Raising the electron multiplier voltage by 200 V should approximately double the abundance of the m/z 69 ion in the tune report for a properly functioning electron multiplier. Over time this ratio will decrease. Nominal increase of the m/z 69 ion abundance as the voltage is increased in the MS is an indicator that the electron multiplier is damaged or worn out and requires replacement. An electron multiplier voltage higher than 2700 V in the tune report indicates that the multiplier needs replacement and/or the MS source needs cleaning.

9.4.5.5 Peak shape/resolution of PFTBA (perfluorotributylamine) Calibration peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MS should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the tune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MS source needs to be cleaned. Refer to section 9.6.1.1 for more specific criteria when evaluating the PFTBA spectrum.

9.4.5.6 Standard spectra autotune: In the event that a maximum sensitivity autotune program does not yield acceptable spectra a standard spectra autotune program may be employed to tune the MS. This autotune program is designed to adjust ion ratios to match those found in mass spectral library reference spectra, but does typically provide high MS sensitivity. As a consequence, the MS lenses (i.e. the entrance lens offset, entrance lens, ion focus repeller) may need adjustment to provide high MS sensitivity while also keeping the relative intensities of m/z 69, 219, & 502 within acceptable limits.

9.4.6 All maintenance performed on the GC/MS system should be performed by a chemist skilled in GC/MS maintenance and recorded in the GC and/or MS maintenance logs.

9.5 Retention Time Windows

9.5.1 The retention time window of the sample component is ± 0.06 minutes of the standard component after time adjustment using the internal standards as time reference peaks. See Table 8 for which target analytes are associated with which internal standards.

9.5.2 Fluctuating retention times are typically due to a leak or loose connection in the GC/MS system, and require system maintenance.

9.6 Instrumental Analysis

9.6.1 Tuning

9.6.1.1 Maximum sensitivity autotune: The mass spectrometer is tuned prior to

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calibration using the maximum sensitivity autotune program. This autotune program is designed to adjust ion ratios to match those found in mass spectral library reference spectra while also providing high MS sensitivity. A valid autotune should provide peak widths < 0.60 amu with mass assignments not differing by more than 0.20 amu for masses 69, 219, and 502 from the PFTBA tuning compound. The relative intensities for m/z 69, 219, and 502 should be 100%, 25-45%, and 0.5-1.5%, respectively. Isotope masses should be present for m/z 70, 220, and 503. Figure 3 shows a typical report for a successful maximum sensitivity autotune.

NOTE: If a maximum sensitivity autotune program does not yield an acceptable tune, a standard spectra autotune program may be used to tune the MS. Refer to the preventative maintenance/troubleshooting section of this SOP for further details regarding this tune program.

9.6.1.2 After running the maximum sensitivity autotune program, the tuning parameters must be verified by injecting 6 uL of a 2000 ng/ml solution (12 ng) of DFTPP (Section 5.12). This must occur before initial calibration and every twelve hours thereafter.

9.6.1.3 Using the exact run parameters when analyzing samples, inject the DFTPP and acquire data. Once the DFTPP elutes use any combination of the top 3 scans to determine the fragment ratios for the ions listed in Figure 3. The spectra must meet the ranges listed in Table 9 for Method 8270C and Table 10 for Method 8270D before analysis of samples or standards continues. Method 8270C tuning criteria are only used for Illinois samples. DFTPP must be injected at a minimum of the beginning of every twelve hours of instrument operation. Figure 3 provides a typical Method 8270D DFTPP tuning report.

9.6.2 Tune performance check: The tune performance check is used to evaluate GC/MS performance. DFTPP (decafluorotriphenylphosphine) is used to validate MS performance by evaluating relative mass intensities of DFTPP ions. GC column performance and injection port inertness is also validated by calculating the tailing factors of benzidine and pentachlorophenol as well as the breakdown of DDT to DDE and DDD.

9.6.2.1 Inject 6 µL of a 2000 ng/mL GC/MS tuning standard.

9.6.2.2 Refer to Table 10 (Table 9 for Illinois samples) for required DFTPP acceptance criteria.

9.6.2.3 Benzidine and pentachlorophenol should not exceed a tailing factor of 2 as given by the following equation:

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9.6.2.4 $TailingFactor = \frac{BC}{AB}$

9.6.2.5 The equation compares the width of the back half of the peak to the width of the front half of the peak at 10% height of the peak. See Figure 4 for an example tailing factor calculation.

9.6.2.6 Degradation of DDT to DDE and DDD should not exceed 20%.

9.6.2.7 If the acceptance criteria are not met, corrective action should be taken. This may include system maintenance or retuning the instrument prior to continuation of analysis.

9.6.3 Initial calibration (ICAL) is performed using the internal standard technique. Typically eight different levels of calibration standards are used to plot calibration curves using a quadratic regression fit with the weight on the inverse of the concentration.

9.6.4 Add 40 µL of the internal standard solution (see Section 7.11.3) to a 2 mL injection vial. Transfer 0.80 mL of each calibration standard to labeled vials containing the internal standard. Preparation of vials in sets of five at a time is recommended. All vials to be analyzed must be prepared in the same manner.

9.6.5 The nominal concentration of the internal standard in the vial is 0.625 µg/mL.

9.6.6 Refer to Table 8 for which analytes are associated with which internal standards.

9.6.7 Inject each calibration standard, collect the data and tabulate the area response of the characteristic ions against the concentration for each target analyte and each internal standard. Select the quadratic curve fit analysis from the calibration options for each compound with the weight on the inverse of the concentrations. An acceptable calibration curve has a coefficient of determination (r^2) of 0.990 or greater.

9.6.8 Quadratic regression with inverse of concentration weighting

$$Y = AX^2 + BX + C$$

Where: Y = Peak Area Ratio of each analyte

$$\text{Peak Area Ratio} = \frac{\text{Peak Area of Target Analyte}}{\text{Peak Area of Internal Standard}}$$

A = Second order constant term (quadratic term)
B = First order constant term (linear term)
C = Constant term
X = Concentration Ratio of each target analyte

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$$\text{Concentration Ratio} = \frac{\text{Concentration of Target Analyte}}{\text{Concentration of Internal Standard}}$$

$$\text{Weighting Factor} = \frac{1}{X}$$

9.7 Sample Analysis

9.7.1 Samples are analyzed in a group referred to as a Sequence which is obtained from the LIMS. The Sequence number is a date keyed numbering system and is unique for every analytical run. The sequence begins with a mid and low range level calibration standard (200; 10 ng/ml) as instrument performance checks. These checks are designated as CRL sample types in the LIMs sequence. Then performance evaluation/tune verification and initial calibration standards are analyzed followed by sample extracts interspersed with CCV standards and performance evaluation/tune verification standards . The sequence ends when the entire sequence has been injected or when qualitative and/or quantitative QC criteria are exceeded.

9.7.2 Add 40 µL of the internal standard solution (see 7.11.3) to a 2 mL injection vial. Transfer 0.80 mL of each sample to the vial. Preparation of vials in sets of five at a time is recommended. Prepare each vial (standard, sample or QC sample) in the same manner. Inject the samples and collect and process the data using a chromatography workstation. Area counts of the internal standard peaks should be between 50-200% of the area of the internal standards in the initial calibration verification (ICV) standard. If great variation occurs, samples may need to be re-aliquoted and re-injected.

9.7.3 If a response exceeds the theoretical concentration of the highest standard dilute the sample extract and re-analyze. Dilute such that the concentration is in the upper half of the standard curve.

9.7.4 The system requires a tune performance check at the beginning of every sequence and with every 12 hours of additional analysis time. A tune performance check routinely occurs every ten samples and is included with bracketing continuing calibration standards.

9.7.4.1 The tune performance check is performed as outlined in Section 9.6.2 and must meet DFTPP requirements as listed in Table 9 for Method 8270C and Table 10 for Method 8270D for analysis to continue. Method 8270C tuning criteria are only used for Illinois samples. See Figure 3 for a typical Method 8270D tuning report.

9.7.4.2 Two levels of continuing calibration, Level-4 (100 ng/mL) and Level 7 (1000 ng/mL) are used for this method. CCVs are evaluated on an individual compound basis with two control limit levels. Wisconsin and Minnesota Department of Agriculture target pesticides must have a

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percent difference, based on concentration, of less than 20%, except for cyanazine, ethalfluralin and pendimethalin which must have a percent difference less than 30%. Other method analytes must also exhibit a percent difference of less than 30%. If a compound fails for any of the prescribed CCV limits, all samples that were injected after the last passing CCV must be re-injected or appropriately qualified. When a CCV fails, inspect the system for cause and perform necessary maintenance before recalibrating and proceeding with sample analysis.

$$Percent\ Difference = \frac{R2 - R1}{R1} \times 100$$

Where: R1 = Theoretical Concentration.
R2 = Calculated Concentration from succeeding analyses.

9.7.5 Initial calibration verification (ICV) is done with two levels (100 and 1000 ng/ml) prior to injection of any unknowns or QC samples. The accepted limits are the same as a CCV. See Section 10.7.

9.7.6 Qualitative analysis

9.7.6.1 The qualitative identification of each compound determined by this method is based on retention time and on comparison of the extracted ion chromatograms of the sample with the characteristic extracted ion chromatograms from a reference standard. The reference standard should be generated using the conditions of the method and/or library reference spectra. The characteristic ions for this method are provided in Table 3. Compounds are identified as present when the following criteria are met.

9.7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

9.7.6.1.2 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum (e.g. for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). See Figures 5 and 6 for detailed spectra reports.

9.7.6.2 Identification is hampered when sample compounds are not resolved and produce mass spectra containing ions contributed by more than one compound. When peaks obviously represent more than one compound

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(i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of background spectra is important. If two compounds co-elute directly over one another, it becomes impossible to collect a pure spectra of either component. If one compound is a shoulder peak of another compound, the first spectra is taken from the far side of the target analyte peak of interest and the second spectra is taken from the other side of the shoulder peak. Background subtraction then yields relatively pure spectra of the target analyte of interest.

9.7.6.3 Examination of extracted ion current profiles can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute, the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

9.7.6.4 Two isomers of siduron are separated and identified as two distinct peaks during the chromatographic analysis. The peak area proportions of these two peaks are approximately 10%/90% in the initial calibration standard, while they are approximately 50%/50% in the second source standard. The two peaks are commonly summed together during automated integration of the initial calibration standard. However, they are not summed during automated integration of the second source standard and require summing as manual integration for accurate recovery results. The GC/MS chemist must be aware that these two isomers exist during data analysis and review for siduron.

9.7.6.5 In certain groundwater samples, identification, as described above, and quantitation of alachlor is hampered by an interference suspected to be associated with samples containing high levels of metolachlor. An alternative ion, 237, is used for quantitation in these cases. Report limits for alachlor may need to be increased when this interference is present.

9.7.7 Data package and review

9.7.7.1 Refer to “GEN-016, Data Review Procedures” for preparation and review of each sequence data package.

9.7.7.2 Manual integrations must be properly documented in accordance with GEN-018, Manual Chromatographic Peak Integration.

9.7.7.3 Prior to reporting results via LIMS, false positives must be removed by Q-deletion.

9.8 Calculations

9.8.1 Once a compound has been identified, the quantitation of that compound is based on the integrated abundance from the extracted ion current chromatogram of the primary characteristic ion. The concentration in ng/mL

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of each compound found in the sample extract is calculated by the chromatography workstation using reverse extrapolation. The standard curve regression is established by plotting the concentration ratio against the response ratio using a quadratic regression curve fit from the ICAL analyzed as described in Sections 9.6.3 thru 9.6.8..

9.8.2 In certain groundwater samples, identification and quantitation of alachlor is hampered by an interference suspected to be associated with samples containing high levels of metolachlor. An alternative ion, 237, is used for quantitation in these cases. The use of the 237 ion is only effective at 50 ng/mL and above. If the interference exists at levels below that level, the alachlor reporting limit should be raised and a note in the case narrative should be added for that sample.

9.8.3 The concentration of the sample in µg/L, or µg/kg, is then calculated as follows:

$$Concentration\ (\mu g/L) = \frac{A_x \times D \times V_e}{V_s}$$

Where: A_x = Concentration of compound in the extract in µg/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
V_s = Volume of sample extracted in liters

$$Concentration\ (\mu g/kg) = \frac{A_x \times D \times V_e}{W_s}$$

Where: A_x = Concentration of compound in the extract in µg/mL
D = Dilution factor, if applicable
V_e = Volume of extract in mL
W_s = Weight of sample extracted in kg

10 QUALITY CONTROL

10.1 Refer to SW-846 Method 8000 for general quality control procedures for chromatography methods.

10.2 An analytical batch consists of 20 or fewer samples. Batch quality control samples should be analyzed with each set with the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

Note: If an MS/MSD cannot be prepared because of limited sample volume, a second LCS must be prepared.

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10.3 Method blanks consist of an aliquot of laboratory reagent water or silica sand that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. If target analytes or interferences are present at concentrations that impact the analytical results for samples, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.4 LCSs consist of an aliquot of laboratory reagent water or silica sand spiked with the target analytes, prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. LCS control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. If the recovery of any of the target analytes is outside control limits, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.5 MS/MSD samples consist of duplicate aliquots of sample spiked with the target analytes, prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. MS/MSD control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. MS/MSD control limits are advisory. If the recovery or RPD of any of the target analytes or the RPD is outside control limits, data should be appropriately qualified in accordance with GEN-015, Qualification of Data.

10.6 Initial calibration (ICAL) is performed using the internal/external standard technique by injecting a minimum of 5 of the available calibration standards. The lowest calibration point must be at or below the reporting limit. An acceptable calibration curve has a coefficient of determination (r^2) of 0.990 or greater.

10.7 Two levels of CCVs, Level 4 (100 ng/mL) and Level 7 (1000 ng/mL) are used for this method. CCVs are evaluated on an individual compound basis with two control limit levels. Wisconsin and Minnesota Department of Agriculture target pesticides must have a percent difference, based on concentration, of less than 20%, except for cyanazine, ethalfluralin and pendimethalin which must have a percent difference less than 30%. Other method analytes must also exhibit a percent difference of less than 30% . If the response for any analyte varies from the theoretical concentration by more than the limits described above, a new calibration curve must be prepared or data appropriately qualified in accordance with GEN-015, Qualification of Data.

10.8 Surrogates are added to every sample and QC sample. Surrogate control limits are generated on at least a yearly basis. If a surrogate recovery is outside of control limits, the sample should be re-extracted and re-analyzed, if possible. If not, the data should be appropriately qualified in accordance with GEN-015, Qualification

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of Data.

10.9 Two levels of second source calibration verification standard (SCV) are analyzed with every analysis sequence immediately after the ICAL. The acceptance limits are the same as those used for CCVs. If an SCV fails, immediate corrective action is required before proceeding with sample analysis. Affected data should be qualified according to GEN-015, Qualification of Data.

10.10 A tune performance check is injected and required to pass DFTPP, tailing, and degradation criteria before initial analysis proceeds. Refer to section 9.6.2 for specific tune performance check criteria. Subsequent tune performance checks are analyzed every 12 hours or less, typically alongside CCV standards, and are required to pass DFTPP criteria and should pass tailing and degradation criteria.

10.11 Internal standards are added to every injection vial. The response of the internal standards must not vary by < 50% or > 200% from the initial response of the mid-point standard in the initial calibration. Samples failing to meet the limits must be re-injected.

11 METHOD PERFORMANCE

11.1 Typical limit of detection (LODs) for laboratory reagent water and silica sand are listed in Table 1.

11.2 Typical demonstration of capability (DOC) data for laboratory reagent water and silica sand are summarized in Table 2.

12 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

12.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

13 WASTE MANAGEMENT / POLLUTION PREVENTION

13.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

14 REFERENCES

14.1 "Method 8270C," SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, U.S. EPA, OSWER, November 2004.

UNCONTROLLED

14.2 "Method 8270D", SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update IV, U.S. EPA, OSWER, February 2007.

14.3 "Method 8141A"; SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update II; U.S. EPA, OSWER; September 1994.

14.4 Taylor, V.; Hickey, D. M.; Marsden, P.J. "Single Laboratory Validation of EPA Method 8140"; U.S. EPA, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, 1987; EPA-600/4-87-009.

14.5 Pressley, T.A; Longbottom, J.E. "The Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: Method 614"; U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, OH, 1982; EPA-600/4-82-004.

14.6 "Method 622, Organophosphorus Pesticides"; U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, OH, 45268.

14.7 LAM-022, Organonitrogen/Organophosphorus Compounds by GC/NPD, ECCS SOP.

14.8 "Method 3570, Microscale Solvent Extraction (MSE)", SW-846, Test Methods for Evaluating Solid Waste, Third Edition, U.S. EPA, OSWER.

14.9 "Method 3580A, Waste Dilution", SW-846, Test Methods for Evaluating Solid Waste, Third Edition, U.S. EPA, OSWER, Revision 1, July 1992.

UNCONTROLLED

TABLE 1

LODS

Compound	Water LODs (µg/L)	Soil LODs (µg/kg)
Acetochlor	0.0247	9.47
Alachlor	0.0190	7.92
Alachlor 237	0.0614	44.1
Ametryn	0.0186	8.37
Atrazine	0.0237	9.27
Baygon	0.0128	7.94
Benfluralin	0.0144	9.69
Bromacil	0.0137	6.17
Butylate	0.0250	7.49
Carbaryl	0.0186	7.55
Carbofuran	0.0212	7.82
Chlorothalonil	0.0163	7.01
Chlorpyrifos	0.0202	11.9
Clomazone	0.0132	4.97
Cyanazine	0.0346	9.40
Des-ethyl atrazine	0.0154	6.54
De-isopropyl atrazine	0.0327	12.4
Diazinon	0.0519	7.75
Dichlorbenil	0.0157	4.92
Dichlorvos	0.0165	8.23
Dimethenamid	0.0134	7.20
Dimethoate	0.0090	7.54
Disulfoton	0.0285	6.62
EPTC	0.0322	9.95
Ethalfluralin	0.0489	11.9
Ethion	0.0125	7.69
Ethoprop	0.0160	7.56
Fenuron	0.0146	5.84
Fonophos	0.0229	8.61
Hexazinone	0.0224	8.04
Isophenphos	0.0161	7.01
Linuron	0.0205	6.57
Malathion	0.0209	7.49
Methyl parathion	0.0181	11.2
Metolachlor	0.0137	6.78
Metribuzin	0.0066	13.0
Napropamide	0.0179	4.69

UNCONTROLLED

TABLE 1 CONTINUED

LODS

Compound	Water LODs (µg/L)	Soil LODs (µg/kg)
Norflurazon	0.0302	10.4
Parathion	0.0196	8.77
Pendimethalin	0.0100	9.62
Phorate	0.0376	14.8
Phosmet	0.0172	10.2
Prodiamine	0.0271	6.44
Promecarb	0.0156	4.43
Prometon	0.0240	7.91
Prometryn	0.0135	5.69
Propachlor	0.0147	9.84
Propanil	0.0095	7.51
Propazine	0.0210	9.94
Propham	0.0264	7.47
Prophenphos	0.0218	21.6
Siduron	0.0231	6.18
Simazine	0.0223	8.46
Simetryne	0.0152	9.73
Tebuthiuron	0.0250	7.61
Terbuthylazine	0.0119	7.94
Terbutryn	0.0118	10.5
Terbacil	0.0464	7.73
Terbufos	0.0125	6.61
Triallate	0.0258	10.5
Trifluralin	0.0168	11.5

Water LODs: GC Run # A9B0902, 02/05/09
Soil LODs: GC Run # A9C0905, 03/04/09

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TABLE 2-1
DOCS FOR WATER

Compound	Spike Level (µg/L)	Average Rec (%)	RSD (%)
Acetochlor	200	96.6	3.15
Alachlor	200	92.7	5.97
Alachlor 237	200	93.7	8.33
Ametryne	200	95.5	5.39
Atrazine	200	95.2	3.90
Benfluralin	200	88.7	6.26
Bromacil	200	93.4	9.96
Butylate	200	87.8	6.55
Chlorothalonil	200	107	4.25
Chlorpyrifos	200	92.8	3.52
Cyanazine	200	108	18.4
Des-ethyl atrazine	200	92.6	3.70
De-isopropyl atrazine	200	79.7	9.94
Diazinon	200	106	6.33
Dimethenamid	200	93.2	6.27
EPTC	200	86.6	6.00
Ethalfluralin	200	104	7.87
Fonophos	200	105	7.41
Hexazinone	200	98.0	45.3
Malathion	200	98.2	9.80
Metolachlor	200	95.7	5.67
Metribuzin	200	94.7	6.04
Parathion	200	126	13.0
Parathion-methyl	200	124	8.70
Pendamethalin	200	94.3	8.54
Phorate	200	94.3	6.16
Prometon	200	90.8	4.33
Prometryne	200	93.5	4.51
Propachlor	200	105	4.94
Propazine	200	95.6	5.52
Simazine	200	99.7	3.32
Terbufos	200	96.7	6.79
Triallate	200	102.7	8.64
Trifluralin	200	92.5	3.22
Carbaryl	200	81.9	6.73
Carbofuran	200	88.1	13.0
Clomazone	200	95.4	1.45

UNCONTROLLED

TABLE 2-1 CONTINUED
DOCS FOR WATER

Compound	Spike Level (µg/L)	Average Rec (%)	RSD (%)
Dichlobenil	200	84.8	2.26
Dichlorvos	200	91.9	1.70
Dimethoate	200	95.5	7.20
Disulfoton	200	82.2	7.41
Ethion	200	99.9	17.6
Ethalfluralin	200	104	7.87
Ethoprop	200	98.2	6.48
Fenuron	200	83.3	9.02
Isofenphos	200	99.6	15.1
Linuron	200	87.6	7.77
Napropamide	200	81.9	11.3
Norflurazon	200	90.1	31.7
Phosmet	200	96.7	30.8
Prodiamine	200	93.2	8.30
Profenphos	200	101	24.2
Promecarb	200	92.0	7.76
Propanil	200	89.4	9.31
Propham	200	89.9	4.93
Propoxur	200	92.8	9.31
Siduron	200	85.0	22.1
Simetryn	200	89.2	7.85
Tebuthiuron	200	81.1	10.8
Terbacil	200	90.2	9.90
Terbuthylazine	200	92.0	3.97
Terbutryn	200	90.0	5.82
Atrazine-d5 (Surr.)	100	91.8	7.27
TPP (Surr.)	100	105	27.3
Parathion-d10 (Surr.)	100	120	21.9

GC Run# A9H0501 Analyzed 02/06/09

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TABLE 2-2

DOCS FOR SOIL

Compound	Spike Level (µg/kg)	Average Rec (%)	RSD (%)
Acetochlor	100	99.0	9.17
Alachlor	100	99.7	6.40
Alachlor 237	100	105	5.31
Ametryne	100	102	4.89
Atrazine	100	102	5.64
Baygon	100	93.4	6.41
Benfluralin	100	95.5	6.92
Bromacil	100	93.3	4.66
Butylate	100	105	3.31
Carbaryl	100	94.4	4.29
Carbofuran	100	89.7	5.94
Chlorpyrifos	100	100	7.52
Chlorthalonil	100	98.2	6.98
Clomazone	100	99.7	4.84
Cyanazine	100	94.4	6.16
Des-ethyl atrazine	100	103	4.47
De-isopropyl atrazine	100	100	9.13
Diazinon	100	106	4.22
Dichlorbenil	100	105	2.94
Dichlorvos	100	81.7	6.86
Dimethenamid	100	103	5.25
Dimethoate	100	85.4	5.49
Disulfoton	100	99.9	5.26
EPTC	100	109	4.96
Ethalfluralin	100	89.2	1.95
Ethion	100	89.2	8.53
Ethoprop	100	94.5	4.26
Fenuron	100	95.6	6.29
Fonophos	100	103	4.89
Hexazinone	100	101	3.74
Isophenphos	100	99.5	5.45
Linuron	100	93.8	4.59
Malathion	100	90.4	10.4
Metolachlor	100	102	5.64
Metribuzin	100	102	5.31
Napropamide	100	103	7.71
Norflurazon	100	88.0	6.88
Parathion	100	93.9	7.23
Parathion-methyl	100	82.8	4.48

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TABLE 2-2 CONTINUED

DOCS FOR SOIL

Compound	Spike Level (µg/kg)	Average Rec (%)	RSD (%)
Pendamethalin	100	89.4	10.2
Phorate	100	107	4.09
Phosmet	100	89.8	5.41
Prodiamine	100	93.5	5.11
Promecarb	100	93.1	6.75
Prometon	100	102	8.03
Prometryne	100	99.1	3.96
Propachlor	100	105	4.35
Propanil	100	99.4	3.27
Propazine	100	103	3.58
Propham	100	102	4.81
Prophenphos	100	95.0	15.2
Siduron	100	102	3.38
Simazine	100	105	5.29
Simetryne	100	95.3	4.93
Tebuthiuron	100	88.4	7.27
Terbuthylazine	100	97.6	3.06
Terbutryn	100	97.6	5.60
Terbacil	100	83.7	10.8
Terbufos	100	89.2	8.53
Triallate	100	105	6.16
Trifluralin	100	101	4.88
Atrazine-d5 (Surr.)	50	103	8.25
TPP (Surr.)	50	111	9.69
Parathion-d10 (Surr.)	50	86.7	11.4

GC Run# A9A2702
Analyzed 02/06/09

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TABLE 3

CHARACTERISTIC IONS AND RETENTION TIMES

Compound	Retention Time (min)	Quantitation Ion	Qualifier Ions
EPTC-d14 (I.S.)	7.90	94	142, 203
EPTC	7.93	128	86, 189
Dichlorvos	8.59	109	185, 79, 220
Butylate	8.94	146	174, 156, 217
Dichlobenil	9.80	171	136, 100, 75
Propham	10.54	93	179, 120, 137
Tebuthiuron	13.10	156	171, 88, 74
De-isopropyl Atrazine	13.60	173	158, 145, 175
Des-ethyl Atrazine	13.89	172	187, 174, 189
Phorate-d10 (IS)	13.95	131	99, 270
Phorate	13.98	121	260, 153, 231
Ethoprop	14.11	158	242, 200, 97
Baygon	14.74	110	152, 81
Propachlor	15.12	120	176, 104
Promecarb	15.19	135	65, 91, 77
Prometon	15.25	168	210, 225, 183
Triallate	15.30	268	226, 228
Simazine-d10 (IS)	15.43	211	193, 179
Simazine	15.47	201	186, 203, 202
Diazinon	15.69	304	152, 276, 199
Terbufos	15.74	231	153, 103, 288
Atrazine-d5 (Surr.)	15.78	220	205, 178, 58
Atrazine	15.80	200	215, 217, 202
Fonofos	15.89	246	137, 109
Propazine	16.06	172	229, 214 216
Disulfoton	16.35	88	274, 186, 97
Terbuthylazine	16.42	173	229, 214, 132
Carbofuran	16.50	149	164, 221, 122
Ethalfluralin	16.57	276	55, 292, 333
Fenuron	16.61	72	164, 119, 91
Clomazone	16.73	125	204, 89, 127
Trifluralin	16.95	306	264, 290, 335
Benfluralin	17.03	292	335, 264, 276
Metribuzin	17.17	198	171, 103, 182
Simetryne	17.56	213	198, 170, 155
Ametryne	17.65	227	212, 170, 183

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TABLE 3 (CONTINUED)

CHARACTERISTIC IONS AND RETENTION TIMES

Compound	Retention Time (min.)	Quantitation Ion	Qualifier Ions
Prometryne	17.72	241	184, 226, 242
Dimethoate	17.83	87	125, 229, 93
Terbutryn	17.98	226	241, 185, 170
Terbacil	18.26	161	160, 117, 144
Acetochlor	18.37	146	117, 223, 162
Chlorpyrifos	18.37	199	197, 314, 258
Dimethenamid	18.42	154	203, 230
Alachlor	18.53	160	188, 146
Propanil	18.66	161	163, 217, 57
Chlorthalonil	18.70	266	264, 229, 124
Carbaryl	19.06	144	115, 116, 201
Methyl Parathion	19.19	263	109, 200, 246
Metolachlor	19.17	162	238, 146
Malathion	19.30	173	158, 143, 99
Linuron	19.35	61	248, 160, 250
Bromacil	19.52	205	207, 231, 260
Prodiamine	19.52	321	279, 216, 148
Isophenphos	19.56	213	255, 185, 58
Pendamethalin	19.78	252	281, 192, 208
Parathion-d10	19.83	301	115, 99
Parathion	19.85	291	97, 139, 109
Cyanazine	20.07	225	198, 172, 68
Prophenphos	20.14	208	297, 337, 139
Napropamide	20.38	128	271, 100, 72
Siduron	20.53	93	232, 119, 55
Ethion	20.66	231	153, 125, 97
Triphenylphosphate (Surr)	21.43	326	170, 77
Phosmet	22.14	160	133, 104, 76
Norflurazon	22.25	303	145, 102, 173
Hexazinone	22.60	171	128, 252, 83

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TABLE 4-1

STOCK STANDARD CONCENTRATIONS

ABSOLUTE PART # 93725

Mix 1 Compound	Concentration (µg/mL)
Acetochlor	20
Alachlor	20
Ametryn	20
Atrazine	20
Des-ethyl atrazine	20
De-isopropyl atrazine	20
Butylate	20
Bromacil	20
Chlorpyrifos	20
Chlorthalonil	20
Cyanazine	20
Diazinon	20
Dimethenamid	20
EPTC	20
Ethalfluralin	20
Fonophos	20
Hexazinone	20
Malathion	20
Metolachlor	20
Metribuzin	20
Parathion	20
Parathion-methyl	20
Pendimethalin	20
Phorate	20
Prometon	20
Prometryne	20
Propachlor	20
Propazine	20
Simazine	20
Terbufos	20
Triallate	20
Trifluralin	20

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TABLE 4-2

STOCK STANDARD CONCENTRATIONS

ABSOLUTE PART # 94174

Mix 2 Compound	Concentration (µg/mL)
Baygon	20
Benfluralin	20
Carbaryl	20
Carbofuran	20
Clomazone	20
Dichlobenil	20
Dichlorvos	20
Dimethoate	20
Disulfoton	20
Ethion	20
Ethoprop	20
Fenuron	20
Isofenphos	20
Linuron	20
Napropamide	20
Norflurazon	20
Phosmet	20
Prodiamine	20
Profenophos	20
Promecarb	20
Propanil	20
Propham	20
Siduron	20
Simetryne	20
Tebuthiuron	20
Terbacil	20
Terbuthylazine	20
Terbutryn	20

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TABLE 5

INTERMEDIATE MIX CONCENTRATIONS

This page intentionally left blank as there are no intermediate concentrations for this method.

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TABLE 6

CALIBRATION STANDARDS

ICAL Level	8	7	6	5	4	3	2	1
Final Concentration (ng/mL)	2000	1000	500	200	100	50	25	10
Stock Solution ID	7.4.1	7.4.1	7.4.1	7.4.1	L-8	L-7	L-6	L-5
mL Added	10	10	2.5	1	10	5	5	5
Stock Solution ID	7.4.2	7.4.2	7.4.2	7.4.2	-	-	-	-
mL Added	10	10	2.5	1	-	-	-	-
Surrogate Mix ID	7.7.3	7.7.3	7.7.3	7.7.3	-	-	-	-
mL Added	20	20	5	2	-	-	-	-
Final volume (mL)	100	200	100	100	200	100	100	100

All solutions prepared in 80% iso-octane/20% acetone

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TABLE 7

WORKING SURROGATE STANDARD SOLUTION

Atrazine-d5	
Stock Solution	7.7.2.1
(µg/mL)	500
mLs added	4
Parathion-d10	
Stock Solution	7.7.2.2
(µg/mL)	500
mLs added	4
TPP	
Stock Solution	7.7.2.3
(µg/mL)	1000
mLs added	2
Final Volume (mL)	200
Final Concentration (µg/mL)	10

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TABLE 7-2

WORKING INTERNAL STANDARD SOLUTIONS

EPTC-d14	
Stock Solution	7.11.2.1
(µg/mL)	500
mLs added	5
Phorate-d10	
Stock Solution	7.11.2.2
(µg/mL)	500
mLs added	5
Simazine-d10	
Stock Solution	57.11.2.3
(µg/mL)	500
mLs added	5
Final Volume (mL)	200
Final Concentration (µg/mL)	12.5

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TABLE 8

ANALYTES ASSOCIATED WITH EACH INTERNAL STANDARD

EPTC-d14		
EPTC	Butylate	Dichlorvos
Dichlobenil	Propham	

Simazine-d10		
De-isopropyl atrazine	Des-ethyl Atrazine	Prometon
Simazine	Atrazine-d5 (Surr.)	Atrazine
Propazine	Trifluralin	Metribuzin
Ametryn	Prometryn	Acetochlor
Chlorpyrifos	Dimethenamid	Alachlor
Chlorthalonil	Metolachlor	Bromacil
Pendimethalin	Cyanazine	Hexazinone
Tebuthiuron	Propoxur	Promecarb
Terbuthylazine	Carbofuran	Fenuron
Clomazone	Benfluralin	Simetryn
Terbutryn	Terbacil	Propanil
Carbaryl	Linuron	Prodiamine
Napropamide	Siduron	Norflurazon
Triphenylphosphate (Surr.)		

Phorate-d10		
Phorate	Butylate	Dichlorvos
Dichlobenil	Propham	Propachlor
Triallate	Diazinon	Terbufos
Fonophos	Ethalfluralin	Parathion-methyl
Malathion	Parathion-d10 (Surr.)	Parathion
Ethoprop	Disulfoton	Dimethoate
Isofenphos	Profenphos	Ethion
Phosmet		

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TABLE 9

METHOD 8270C

DFTPP TUNING CRITERIA

ONLY FOR ILLINOIS SAMPLES

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

From Method 8270C

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TABLE 10

METHOD 8270D

DFTPP TUNING CRITERIA

Mass Ion	Abundance Criteria
51	10-80% of Base Peak
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of Base Peak
197	< 2% of mass 198
198	Base peak, or > 50% of Mass 442
199	5-9% of mass 198
275	10-60% of Base Peak
365	> 1% of mass 198
441	present but < 24% of mass 442
442	Base Peak, or > 50% of mass 198
443	15-24% of mass 442

From Method 8270D

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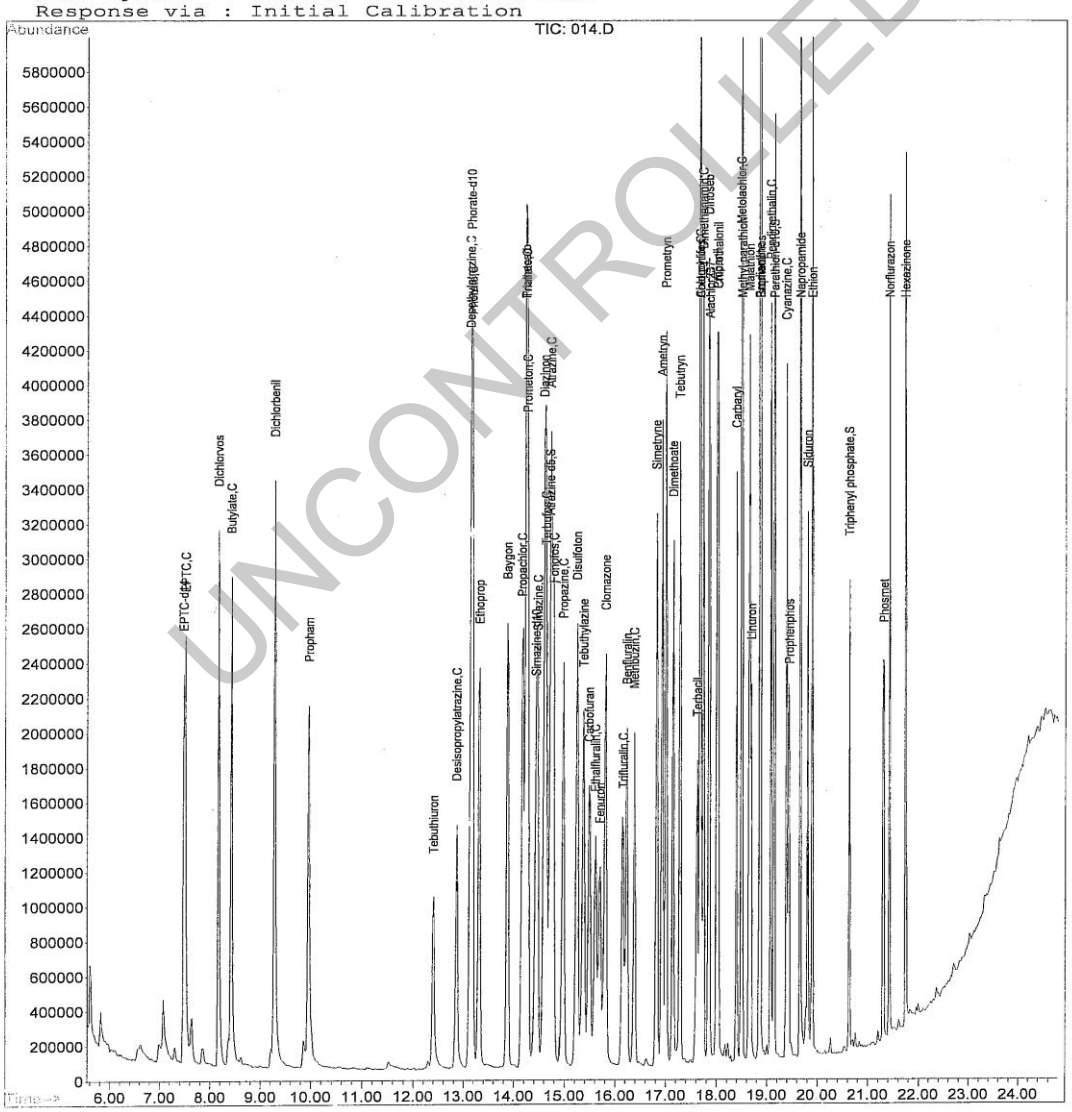
FIGURE 1

TOTAL ION CHROMATOGRAM FOR A 1000 NG/ML CCV

Quantitation Report

Data File : F:\HPCHEM\1\DATA\A9G2101\014.D Vial: 14
Acq On : 21 Jul 2009 5:24 pm Operator: BG
Sample : A9G2101-CCV2 Inst : 3307A0319
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Jul 22 7:59 2009 Quant Results File: A9G2101.RES

Method : F:\HPCHEM\1\METHODS\A9G2101.M (RTE Integrator)
Title : 8141 by GCMS Rtx 200 .032 ID .25 u S# 720299
Last Update : Tue Jul 21 15:44:13 2009
Response via : Initial Calibration



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014.D A9G2101.M Tue Jul 28 15:18:19 2009 Page 3

FIGURE 2

TYPICAL MAXIMUM SENSITIVITY AUTOTUNE REPORT

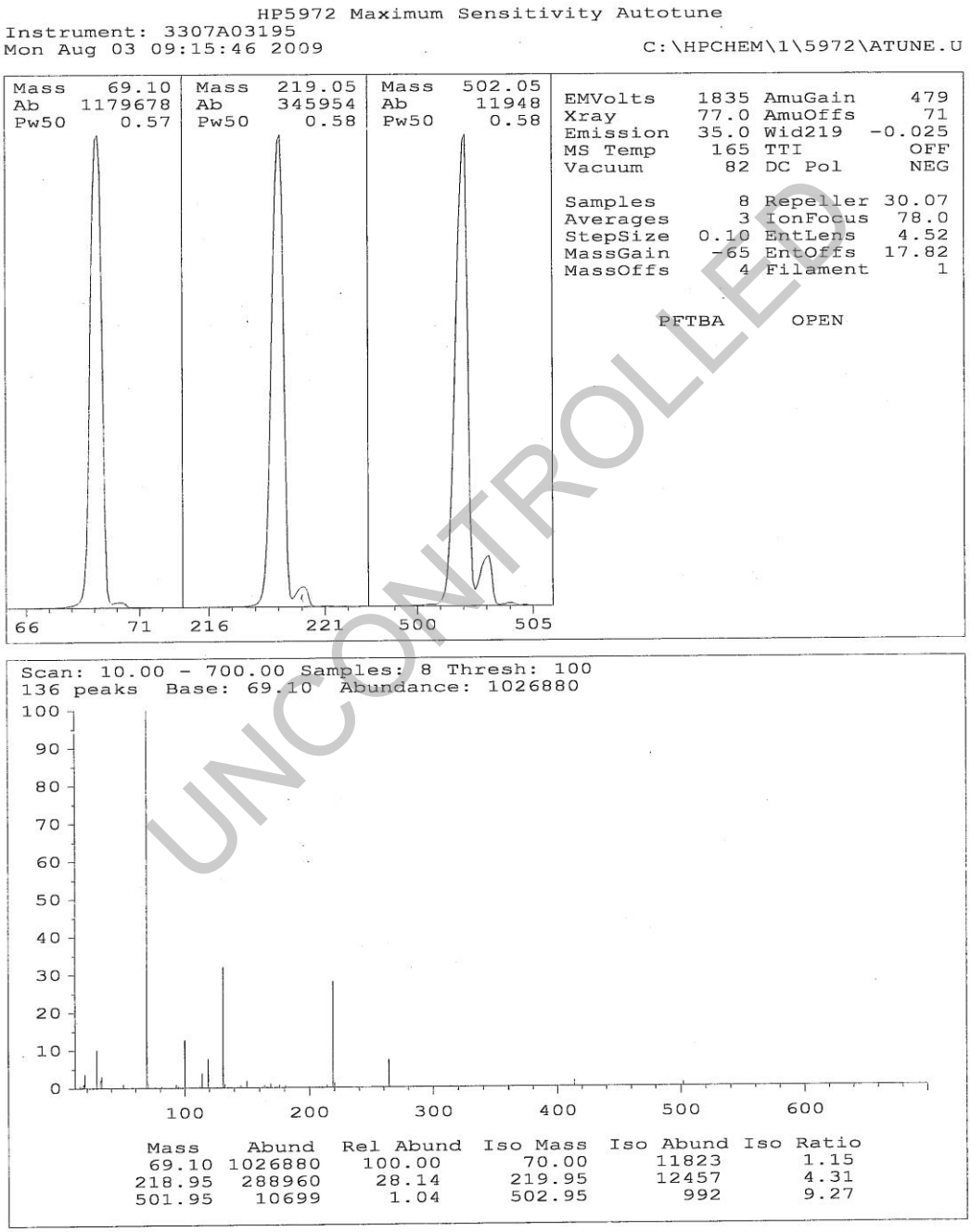

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FIGURE 3

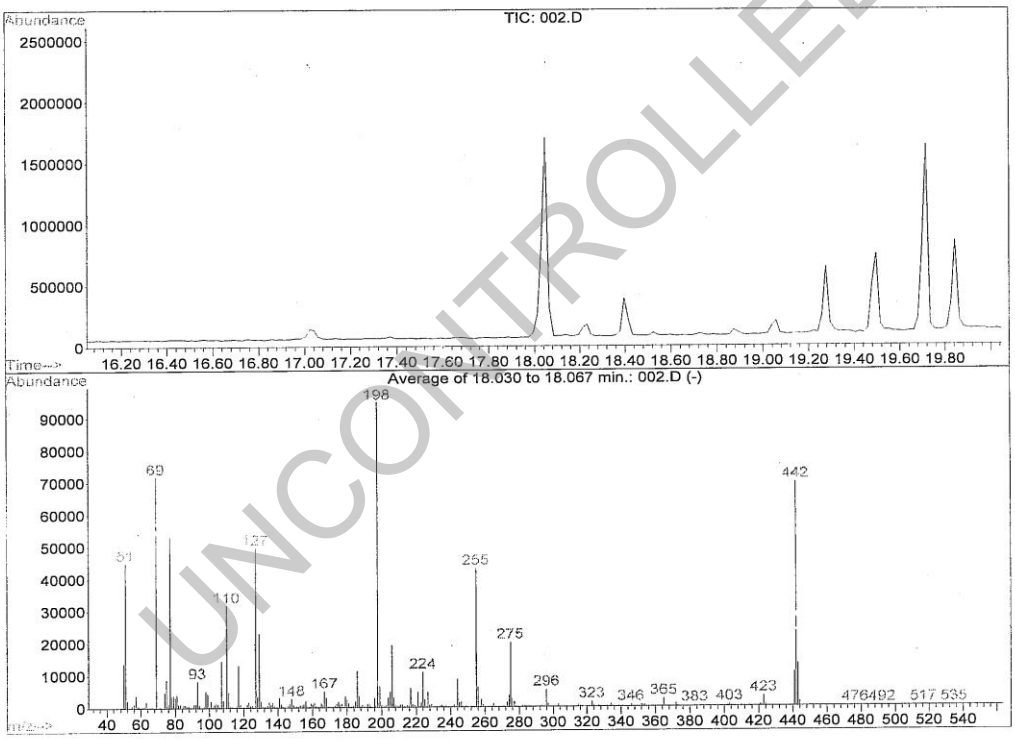
TYPICAL DFTPP TUNING REPORT

FOR METHOD 8270D

DFTPP

Data File : F:\HPCHEM\1\DATA\A9G2101\002.D
Acq On : 21 Jul 2009 11:13 am
Sample : A9G2101-TUN1
Misc :
MS Integration Params: RTEINT.P
Method : F:\HPCHEM\1\METHODS\A9G2101.M (RTE Integrator)
Title : 8141 by GCMS Rtx 200 .032 ID .25 u S# 720299

Vial: 2
Operator: BG
Inst : 3307A0319
Multiplr: 1.00



AutoFind: Scans 682, 683, 684; Background Corrected with Scan 676

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
51	198	10	80	47.4	44968	PASS
68	69	0.00	2	0.0	0	PASS
69	198	0.00	100	75.5	71708	PASS
70	69	0.00	2	0.0	0	PASS
127	198	10	80	52.3	49634	PASS
197	198	0.00	2	0.0	0	PASS
198	198	100	100	100.0	94917	PASS
199	198	5	9	6.9	6557	PASS
275	198	10	60	21.1	20024	PASS
365	198	1	100	2.6	2474	PASS
441	442	0.01	24	15.2	10603	PASS
442	198	50	100	73.4	69677	PASS
443	442	15	24	19.1	13297	PASS

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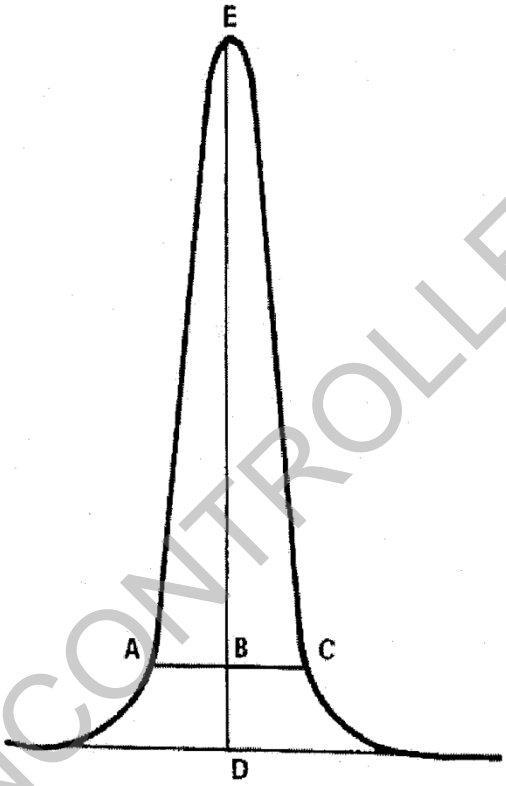
FIGURE 4

TAILING FACTOR CALCULATION



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$$TailingFactor = \frac{BC}{AB}$$

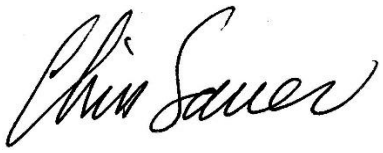
Example Calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm

$$AB = 11\ mm$$
$$BC = 12\ mm$$

$$Therefore:\ TailingFactor = \frac{12}{11} = 1.1$$

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede any prior revisions effective immediately as dated.

Reviewed By:



01/26/16

Chris Sauer, GC/MS Team Leader — Date

01/26/16

Patrick Letterer, Quality Manager — Date

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Approved By:

01/26/16

Nick Nigro, General Manager — Date

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ENVIRONMENTAL LABORATORY QUALITY ASSURANCE MANUAL

TABLE OF CONTENTS

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1 INTRODUCTION AND SCOPE

The purpose of this Quality Manual is to outline the management system for Minnesota Department of Health's Public Health Laboratory Division (PHL) Environmental Laboratory Section (ENV). The Quality Manual defines the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality that is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance.

The Quality Manual sets the standard under which all laboratory operations are performed, including ENV's organization, objectives, and operating philosophy. This Standard is consistent with ISO/IEC 17025:2005 requirements that are relevant to the scope of environmental testing services and thus, ENV operates a quality system in conformance with ISO/IEC 17025:2005(E). In addition, the Quality Manual has been prepared to be consistent with the USEPA's requirements for certification of drinking water laboratories.

More information:

Manual for the Certification of Laboratories Analyzing Drinking Water
Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water

1.1 Scope of Testing

ENV's scope of analytical testing services includes those listed in the Laboratory Information Management System (LIMS). The Environmental Laboratory Section supports public health and environmental protection functions of state government by performing chemical, bacteriological and radiological analyses of environmental samples including, but not limited to, drinking water, surface water, wastewater, soil, air and commercial products. ENV provides these testing services for programs in the Environmental Health Division at the Minnesota Department of Health, for the Minnesota Pollution Control Agency, the Minnesota Department of Transportation, and various agencies of local government. ENV maintains the capability to respond to chemical, microbiological, and radiological emergencies within Minnesota and with abilities to analyze clinical specimens. The analysis of clinical specimens, including, but not limited to, blood, serum and urine, do not fall under the purview of this Quality Manual.

ENV also develops new analytical methods and provides technical training and consultation at the request of its clients. The Environmental Laboratory ensures that testing capacity is available to support the public health and environmental protection objectives of the state.

1.2 Table of Contents, References and Appendices

The Table of Contents precedes Section 1 and Appendices are in Section 27.

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This Quality Manual uses the references included in ISO/IEC 17025:2005(E) requirements that are relevant to the scope of environmental testing, and USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition.

1.3 Glossary and Acronyms Used

Quality control (QC) terms are generally defined within ENV that describes the activity. The QC definitions and terms listed herein are standardized for use in this laboratory. Employees in this laboratory recognize that, in some cases, a particular USEPA-approved method and, in turn, a particular Standard Operating Procedure (SOP) may use different QC definitions and QC terms. In those situations, the QC in those particular SOPs supersedes the QC definitions and terms in this Quality Assurance Manual.

1.3.1 Glossary

Acceptance Limits: A range within which specified measurement results must fall to be compliant. Exceedance of acceptance limits require corrective action or that noncompliant data be qualified, based on method SOP criteria.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy is a data quality indicator that includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations.

Aliquot: A representative portion of a sample taken for sample preparation and/or analysis and assumed to have been taken with negligible sampling error.

Analyte: The element, ion, compound, or other substance that an analytical procedure determines.

Analytical Sequence: Prepared samples which are analyzed together as a group. An analytical sequence can include prepared samples originating from various matrices and can exceed 20 samples. Analytical sequence may also include instrument calibration and QC samples, such as SCV and CCV.

Analytical Uncertainty: An estimate of the error in a measurement that includes all laboratory activities performed as part of the analysis.

Batch: Client and QC samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A batch is composed of one to 20 samples of the same matrix, and with a maximum time between the start of processing of the first and last sample in the batch not to exceed method-defined time limit.

Bias: The systematic or persistent deviation of a measurement process which causes errors in one direction.

Blind Sample: A sample submitted for analysis to the laboratory with the true value(s) known only by the submitter. It is used to test the laboratory's proficiency in the execution of the measurement process.

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Bottle Blank (BB): A blank sample (reagent water, solvent rinse, etc.) taken from a new lot of sample collection containers to asses any potential contamination in the containers before releasing for use. For more information, see the Bottle Blank Quality Assurance procedure.

Calibration: The process of quantifying an instrument's response to known values under specified conditions.

Calibration Blank: A zero standard that contains the reagents present in the calibration standards, but does not contain the target analyte(s). It can be used as a zero point standard in a calibration or for background subtraction.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and the instrument response to a single analyte.

Calibration Range: The working range between (and including) the lowest and highest calibration standards, from which the value of unknown samples can be determined.

Calibration Standard: A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): Reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

Chain of Custody: The procedures and records that document the possession and handling of samples from collection through disposal.

Chain of Custody Form (COC): A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: a unique Chain of Custody identification number; the number and types of containers; collector; time of collection; preservation; and requested analyses.

Class A Glassware: Volumetric glassware of the highest accuracy. Class A volumetric glassware complies with the Class A tolerances defined in ASTM E694 and must be permanently labeled as Class A.

Clean Water Act, CWA (Federal Water Pollution Control Act): The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816, that empowers USEPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Continuing Calibration Blank (CCB): A blank that is run within an analytical sequence. The CCB may indicate contamination, carryover, baseline drift or other instrument or reagent changes occurring over the course of an analytical run that contributes to the value obtained for the quantity in the analytical procedure.

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Continuing Calibration Verification (CCV): A standard analyzed within an analytical sequence that verifies the previously established calibration curve and confirms accurate analyte quantitation for all samples. The concentration of the CCV should be near the mid-point of the calibration curve. Also known as a Calibration Verification Standard (CVS).

Control Charts: Plots of quality control data, such as precision or accuracy, to visually monitor a process or analysis.

Control limits: Statistically determined limits that reflect the expected variation in data. When data points fall outside the limits, it may be due to random error or it may indicate the analytical system is out control. Control limits are usually defined as three standard deviations on either side of the mean.

Corrective Action: The action taken to eliminate the cause(s) of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Daily: Applies to the days during which the analytical process (including preparation of samples) is performed.

Data Quality Objectives (DQO): A statement of the appropriate type of data and overall level of uncertainty that a decision-maker is willing to accept in results derived from analytical data. DQOs are often expressed in terms of precision, accuracy, reliability, representativeness, and comparability.

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Data Validation: A process used to determine if data are accurate, complete, or meet specified criteria.

Dissolved Analyte: A dissolved analyte is an analyte within in an aqueous sample that will pass through a 0.45 µm membrane filter prior to any sample preservation.

External Standard Calibration: The process of creating a mathematical relationship by directly comparing the concentrations of target analytes to their instrument responses in calibration standards. Samples are quantitated by using this mathematical relationship to calculate the concentrations of target analytes from the instrument responses to the same target analytes in samples.

Field Blank: An aliquot of reagent water or other appropriate blank matrix that is placed in a sample container in the field and treated as a sample in all respects, including exposure to sampling site conditions, equipment, storage, preservation (if necessary), and all analytical procedures. The purpose of the field blank is to determine if the field procedures or sample transporting procedures and environments could have contaminated the samples.

Filter Blank (FB): For each batch of laboratory filtered or field filtered samples, reagent water is passed through one or more unused filter(s) and the filtrate from each is

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collected. The filtrate is treated like all other dissolved samples in the batch. Analysis of the filter blank will reveal contamination from the filter or filtration process.

Holding Time: The maximum time that a sample may be held prior to preparation and/or analysis and still be considered valid or not compromised.

Initial Demonstration of Capability (IDC): A procedure by which an analytical team must demonstrate acceptable precision, accuracy, sensitivity, and specificity for the analysis prior to its initial use. For additional information see the Initial and Ongoing Demonstration of Capability procedure.

Intermediate Standard: A solution made up from the stock standard solution and diluted as necessary to prepare working standard solutions.

Internal Standard (IS): A constant amount of non-target analyte that is added to all samples, blanks, and standards. The internal standard should not be present in the original test sample at interfering levels and should behave similarly to the target analyte(s). Ideally, the retention times of internal standards should be near the retention times of the associated target analytes. See individual SOPs for additional criteria applicable to the use of internal standards.

Internal Standard Calibration: The process of creating a mathematical relationship by comparing the instrument response of a target analyte in a calibration standard to the response of an internal standard added to the calibration standard. The relative response factor (RRF) created by this process is used to calculate the concentration of the target analyte in other samples to which the internal standard has also been added. The internal standard(s) is added to all samples, blanks and standards at a constant amount, should not be present in the original test samples in interfering amounts, and should behave similarly to the target analyte.

Laboratory Control Sample (LCS): An aliquot of reagent water or other blank matrix, known to be free of interfering amounts of target analytes or other interferences, to which known quantities of the target analytes are added in the laboratory. It is prepared and analyzed exactly like a sample. Its purpose is to verify that the procedure is in control and that the laboratory is capable of making accurate measurements. A LCS is also known as a Laboratory Fortified Blank (LFB) or a Blank Spike (BS).

Laboratory Control Sample Duplicate (LCSD): A second aliquot of reagent water or other blank matrix, known to be free of interfering amounts of target analytes or other interferences, to which known quantities of the target analytes are added in the laboratory. The LCSD is prepared the same as the LCS. It is also known as a Laboratory Fortified Blank Duplicate (LFBD) or Blank Spike Duplicate (BSD).

Laboratory Duplicates (DUP): Two aliquots taken from a single sample in the laboratory and analyzed separately using identical procedures. Analysis of DUP indicates precision associated with laboratory procedures for a specific sample matrix, but not with sample collection, preservation, or storage procedures.

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Laboratory Information Management System (LIMS): A computer software system that is used to track samples and the associated quality control, assign permissions, generate client reports, manage stock chemicals and prepared solutions, and to manage user permissions from sample receipt through sample disposal.

Linear Calibration Range (LCR): The concentration range, as determined by the analysis of calibration standards, over which the calibration curve is linear.

Linear Dynamic Range (LDR): The concentration range over which the instrument response is linear. The LDR may extend beyond the calibration range. A LDR study is required to confirm the validity of reporting data beyond the calibration range.

Management System: A set of policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. It is also known as Quality System.

Matrix: The predominant material of the sample to be analyzed. Matrices include, but are not limited to: air, drinking water, non-potable water, biological materials and solids/chemical materials.

Matrix Spike (MS): An aliquot of a field sample to which known quantities of the target analytes are added in the laboratory prior to sample preparation and analysis. The MS is prepared and analyzed exactly like a sample. The background concentrations of the analytes in the sample matrix must be determined in an unspiked aliquot of sample and subtracted from the MS concentrations. The purpose of the MS is to determine whether the sample matrix contributes bias to the analytical results. It is also known as Laboratory Fortified Matrix (LFM).

Matrix Spike Duplicate (MSD): A second aliquot of sample to which known quantities of the target analytes are added in the laboratory prior to sample preparation and analysis. The MSD is treated exactly the same as the MS. It is also known as Laboratory Fortified Matrix Duplicate (LFMD).

Maximum Contaminant Level (MCL): The maximum permissible level of a contaminant in water set by the USEPA which is delivered to the free flowing outlet of the ultimate user of a public water system. See 40 CFR Part 141.2.

May: Denotes a permitted, but not a required action.

Method: A scientific technique for performing a specific measurement as published by a recognized authority or validated by ENV.

Method Blank (MB): An aliquot of reagent water or other blank matrix known to be free of interfering amounts of target analytes or other interferences. The MB is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, acids, internal standards and surrogates that are used with samples. The MB is used to

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determine if target analytes or other interferences are present in the laboratory environment, reagents or apparatus that may give false positive results. It is also known as a Laboratory Reagent Blank (LRB) or Blank (BLK).

Method Detection Limit (MDL): The minimum concentration of an analyte that can be distinguished from a blank. The MDL is determined from multiple analyses of laboratory control samples for a given matrix. See 40 CFR 136 App. B for the procedure used to determine the MDL.

Monthly: Applies to those months during which the analysis is performed.

Must: Describes an action, activity or procedural step that is required. Must is synonymous with shall.

Percent Recovery: A measure of the accuracy of a measurement in a given matrix. A known amount of analyte is added to a blank or sample and the concentration found is divided by the concentration of the spike. The result is multiplied by 100 to express the value in percent. The formula is as follows:

$$\% \text{ Recovey} = \frac{C_s - C_u}{C_t}$$

where: C_s = Measured concentration of the spiked sample aliquot or blank
C_u = Measured concentration of the unspiked sample aliquot (Use 0 for an LCS)
C_t = True value of the concentration of the spike added to the sample or blank

Percent Relative Standard Deviation (%RSD): A measurement of the precision of a series of replicate analyses where the Standard Deviation (S) of the replicates is expressed as a percent of the mean (X) value. To calculate:

$$\% \text{ RSD} = \frac{S}{X} \times 100$$

where: S = Standard Deviation
X = Mean value

Post Digestion Spike (PDS): An aliquot of a sample to which known quantities of the target analytes are added after digestion to determine matrix effects.

Precision: The measure of mutual agreement among individual measurements of replicate samples under similar conditions. The most commonly used estimates of precision are standard deviation (S), percent relative standard deviation (%RSD), and relative percent difference (RPD).

Preservation: Chemical or physical treatment of the sample to slow down the chemical and biological changes that occur after the sample was collected from the parent source.

Procedural Standard Calibration: A calibration method in which aqueous calibration standards are prepared and processed (e.g., extracted, and/or derivatized) in exactly the

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same manner as the samples. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiencies in the processing procedure.

Proficiency Testing (PT): A procedure for evaluating an analyst's or laboratory's performance relative to a given set of criteria through the analysis of unknown samples provided by an external source.

Proficiency Test Sample: A sample obtained from an approved provider to evaluate the ability of the laboratory to produce an analytical test result meeting the definition of acceptable performance. The concentration of the analyte(s) in the sample is unknown to the laboratory at the time of analysis.

Purchasing Coordinator: A member of ENV who reviews order requests for supplies and services to ensure the requests meet the purchasing requirements of PHL.

Quality Assurance (QA): An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance Manual (QAM): A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of a laboratory or other organization, to ensure the quality and the utility of its product to its users.

Quality Assurance Project Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): The routine technical activities that give insight into the precision and accuracy of analysis results.

Raw Data: Describes any original factual information from a measurement activity or study recorded, electronically or by hand, in laboratory notebooks, worksheets, records, memoranda, notes, or photo copies thereof, that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, computer printouts, and recorded data from automated instruments.

Reagent Water: Water known to be free of interfering amounts of target analytes or other interferences. Individual SOPs may have additional requirements.

Reference Method: An analytical method issued by a nationally recognized organization from which ENV's analytical SOP is derived. Also known as a standard method.

Relative Percent Difference (RPD): A measure of precision between two values, such as analysis of DUP, MS/MSD, or LCS/LCSD. It is calculated with the formula below:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 - C_2}{2}\right)} \times 100$$

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where: C1 = Measured concentration of the first sample aliquot.
C2 = Measured concentration of the second sample aliquot.

Replicate: Two or more aliquots of a sample analyzed independently and used to determine precision. In some analytical methods, the reported value is an average of all of the replicate analyses.

Reporting Limit (RL): The minimum concentration that can be reported as a quantitated value for a target analyte in a sample following analysis. Typically, this defined concentration can be no lower than the concentration of the lowest calibration standard for that analyte, and can only be used if acceptable quality control criteria for the analyte at this concentration are met.

Reporting Limit Verification (RLV): A procedure that determines whether the established RL is valid for a target analyte within an analysis and/or analytical sequence. This procedure is performed by the analysis of a standard at or below the RL. The percent recovery of the RLV standard must meet the method acceptance criteria. The RL must be verified each time the instrument is calibrated, or monthly at a minimum. Also known as MRL-Level Calibration Verification (CRL) or MRL Check (MRL).

Requirement: Denotes a mandatory specification, often designated by the terms “shall” or “must”.

Resource Conservation and Recovery Act (RCRA): The enabling legislation under 42 USC 321 et seq. (1976), that gives USEPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal.

Safe Drinking Water Act (SDWA): The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the USEPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Safety Data Sheet (SDS): Written information provided by vendors concerning a chemical’s toxicity, health hazards, physical properties, fire hazard, and reactivity including storage, spill, and handling precautions. Formerly known as Material Safety Data Sheet (MSDS).

Sample: A representative portion of material (drinking water, non-potable water, biological material etc.) collected for analysis in the laboratory. A sample must be uniquely identified through sample analysis, reporting and archiving.

Second Source Calibration Verification Standard (SCV): A standard containing target analytes of known concentrations which is used to verify the initial calibration. The SCV is obtained from a source different from the source of the calibration standards or from a different lot if a second source is not available. It is also known as Quality Control Sample (QCS).

Shall: Denotes a mandatory requirement. Shall is synonymous with must.

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Should: Denotes a recommended but not required action.

Standard: A solution or other material with a known value that is used in the laboratory to perform calibrations or QC checks.

Standard Reference Material (SRM): A certified reference material issued by the U.S. National Institute of Standards and Technology (NIST) and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material.

Standard Operating Procedure (SOP): A controlled document that details the techniques and procedures of an operation, analysis, and/or action and is officially approved as the method for performing certain routine functions. The SOP ensures the generation of usable and consistent results.

Stock Standard: A concentrate containing one or more target analytes that is purchased from a commercial source or prepared in the laboratory and is traceable to a NIST standard, when commercially available. The stock standard is used to prepare intermediate standards, and calibration standards.

Surrogate: A non-target analyte added to samples, blanks, and standards before sample preparation. The surrogate is added at a known concentration and is used to determine the efficiency of the sample preparation process. Surrogates should possess chemical properties similar to those of the target analytes, but should not be present in the original test sample.

Target Analyte: The analyte in a given matrix that is determined by an analytical procedure.

Test Sample: The prepared sample from which test portions are removed for analysis.

Trip Blank: A trip blank consists of a sample container or set of containers filled at the laboratory with water demonstrated to be target analyte free. The trip blank travels to the sampling site with the empty sample containers and returns from the site with the full sample containers.

Turnaround Time: The time from when a sample is received through when the data is reported to the client. Turnarounds times are established through Interagency Agreements, Quality Assurance Project Plans, or similar contract documents per analysis.

Weekly: Applies to the weeks during which the analytical process (including preparation of samples) is performed.

1.3.2 Acronyms

A list of acronyms used in this document and their definitions are:

AB – Accrediting body
ASTM – American Society for Testing and Materials
°C – Degrees Celsius

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CCV – Continuing calibration verification
CFR – Code of Federal Regulations
COC – Chain of custody form
DOC – Demonstration of capability
ENV – Environmental Laboratory Section
ICV – Initial calibration verification
ISO/IEC – International Organization for Standardization/International Electrochemical Commission
LCS – Laboratory control sample
LIMS – Laboratory information management system
LFB – Laboratory fortified blank
MDH – Minnesota Department of Health
MDL – Method detection limit
MS – Matrix spike
MSD – Matrix spike duplicate
NIST – National Institute of Standards and Technology
PHL – Public Health Laboratory Division
PT – Proficiency test(ing)
PTP – Proficiency testing provider
PTPA – Proficiency testing provider accreditor
QA – Quality assurance
QC – Quality control
QAM – Quality Assurance Manual
RL – Reporting limit
RPD – Relative percent difference
RSD – Relative standard deviation
SOPs – Standard operating procedures
USEPA – United States Environmental Protection Agency

1.4 Management of the Quality Manual

The Quality Assurance Officer is responsible for maintaining the currency of the Quality Manual.

The Quality Manual is reviewed annually by the Quality Assurance Officer and laboratory personnel to ensure it still reflects current practices and meets the requirements of any applicable regulations or client specifications. Sections of the manual are updated by making a change to the Section and then increasing the revision number by one. The Quality Manual must be re-signed and the Table of Contents updated whenever a Section is updated.

The Quality Manual is considered confidential within ENV and may not be altered in anyway except by approval of the Laboratory Manager and Quality Assurance Officer. If it is distributed to external users, it is for the purpose of reviewing the ENV management system and may not be used for any other purpose without written permission.

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2 ORGANIZATION

ENV is a legally identifiable organization as it resides in a department of state government established through Minn. Statutes 15.01. ENV is responsible for carrying out testing activities that meet the requirements of the ISO/IEC 17025:2005(E) Standard, USEPA regulations and that meet the needs of the client. Through application of the policies and procedures outlined in this Section and throughout the Quality Manual:

- ENV assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment.
- Management and technical personnel have the authority and resources to carry out their duties and have procedures to identify and correct departures from ENV's management system.
- Personnel understand the relevance and importance of their duties as related to the maintenance of ENV's management system.
- Ethics and data integrity procedures ensure personnel do not engage in activities that diminish confidence in ENV's capabilities.
- Confidentiality is maintained.

2.1 Organization

ENV is a division within a department of state government established in Minnesota Statutes, subdivision 15.01. The Commissioner of Health delegates authority to the Public Health Laboratory Division Director. The delegation notice is available upon request. ENV operates independent of other laboratories within the state's laboratory network. ENV does not operate outside its main facility or in any temporary locations, mobile facilities, or field stations.

ENV operates in St. Paul, Minnesota.

The department's organizational chart and its relation to ENV's organization chart can be found on the Minnesota Department of Health's intranet site, and is available upon request for those who do not have access to the intranet. Additional information regarding responsibilities, authority and interrelationship of personnel who manage, perform or verify testing is included in the Management and Personnel sections of the Quality Manual. These sections also include information on supervision, training, technical management, job descriptions, quality personnel, and appointment of deputies for key managerial personnel.

ENV has the resources and authority to operate a management system that is capable of identifying departures from that system and from procedures during testing, and initiates actions to minimize or prevent departures.

More information:

Organizational Charts

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2.2 Conflict of Interest and Undue Pressure

The organizational structure indicated above minimizes the potential for conflicting or undue interests that might influence the technical judgment of analytical personnel. In addition, procedures are in place to prevent outside pressures or involvement in activities that may affect competence, impartiality, judgment, operational integrity, or the quality of the work performed at ENV.

Employees must declare any real or perceived conflicts of interest to their immediate supervisor. Where possible, the supervisor must make accommodations to reduce or eliminate the situation creating a conflict of interest. Where the work cannot be reassigned, the employee and the supervisor will inform any affected parties and agree how to proceed with work while protecting the interests of the employee, the client and the State.

Arrangements, such as policies and procedures to prevent commercial, financial or other influences that may negatively affect the quality of the work or negatively reflect on the competence, impartiality, judgment or operational integrity are described in the state laws for Code of Ethics for Employees of the Executive Branch.

More information:

Code of Ethics for Employees in the Executive Branch

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3 MANAGEMENT

ENV maintains a management system that is appropriate to the scope of its activities.

3.1 Management Requirements

Top management includes the Division Director, Laboratory Manager, Unit Supervisors, and the Quality Assurance Officer.

Management's commitment to good professional practice and to the quality of its products is defined in the Quality Policy statement, Section 3.3.

Management has overall responsibility for the technical operations and the authority needed to generate the required quality of laboratory operations. Management ensures communication within the organization to maintain an effective management system and to communicate the importance of meeting client, statutory, and regulatory requirements. Management assures that the system documentation is known and available so that appropriate personnel can implement their part. When changes to the management system occur or are planned, management ensure that the integrity of the system is maintained.

Management is responsible for carrying out testing activities that meet the requirements of the ISO/IEC 17025:2005(E) Standard, the USEPA and that meet the needs of the client.

Management implements, maintains, and improves the management system, and identify noncompliance with the management system of procedures. Management initiates actions to prevent or minimize noncompliance.

Management ensures technical competence of personnel operating equipment, performing tests, evaluating results, or signing reports, and limits authority to perform laboratory functions to those appropriately trained and/or supervised. General requirements for training and experience are described in the Personnel section of the Quality Manual.

Management is responsible for defining the minimal level of education, qualifications, experience, and skills necessary for all positions in ENV and assuring that technical staff have demonstrated capabilities in their tasks.

Training is kept up to date as described in the Personnel section of the Quality Manual by periodic review of training records and through employee performance review.

Management bears specific responsibility for maintenance of the management system. This includes defining roles and responsibilities to personnel, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodically reviewing data, procedures, and documentation. The

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assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting the quality of environmental tests is documented in individual position descriptions for each person and job classification as indicated in the organization chart.

Management ensures that audit findings and corrective actions are completed within required time frames.

Designated deputies are appointed by management during the absence of the Laboratory Manager, Unit Supervisors or the Quality Assurance Officer, and always if the absence is more than 15 days.

More information:

Organizational Charts

3.2 Management Roles and Responsibilities

3.2.1 Laboratory Manager (Manager of the Environmental Laboratory Section)

The Laboratory Manager is responsible for the overall quality, safety, financial, technical, human resource and service performance of ENV. The Laboratory Manager provides the resources necessary to implement and maintain an effective quality and data integrity program.

The Laboratory Manager is responsible for:

- Ensuring that personnel are free from any commercial, financial and other undue pressures that might adversely affect the quality of their work.
- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the Quality Manual, laboratory SOPs or laboratory policies may be temporarily suspended by the Laboratory Manager.
- Reviews and approves all analytical and administrative ENV SOPs and policies prior to their implementation and ensures all approved SOPs and policies are provided to laboratory personnel and are adhered to.

3.2.2 Quality Assurance Officer (also known as the Quality Manager)

The Quality Assurance Officer (or designee) is responsible for the oversight and review of QC data, but is independent from laboratory operations. The Quality Assurance Officer reports to the Division Director and not to the Unit Supervisors or Laboratory Manager (see organization chart). The Quality Assurance Officer's training and proof of experience in QA/QC procedures, knowledge of analytical methods, and ENV's

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management system are available in the personnel records maintained by the MDH Human Resources Management office and personnel training files.

The Quality Assurance Officer is responsible for:

- serving as a focal point for QA/QC;
- arranging or conducting annual internal audits without outside (e.g., managerial) influence;
- notifying management of deficiencies, and monitoring corrective actions;
- oversight and review of QC data;
- ensuring staff are trained in the management system requirements;
- monitoring corrective actions;
- ensuring that the management system related to quality is implemented and followed at all times;
- monitoring and maintaining laboratory certifications; and
- keeping this Quality Manual current.

More information:

Organizational Charts

3.2.3 Technical Unit Supervisors

The Technical Unit Supervisor (or designee) is a full-time laboratory staff member and supervises laboratory operations for the respective Technical Unit. The Technical Units are Inorganics, Organics, and Biomonitoring and Emerging Contaminants. The Technical Unit Supervisor's proof of experience may be found in the personnel records maintained by the MDH Human Resources Management office and personnel training files.

If the Technical Unit Supervisor is absent for fifteen (15) consecutive calendar days or more, a deputy with appropriate qualifications will perform the Technical Unit Supervisor's duties.

The Technical Unit Supervisor is responsible for:

- meeting the general and education requirements and qualifications found the respective position description;
- monitoring performance data and the validity of the analyses for the respective Technical Unit within ENV;
- approving personnel have appropriate education and technical background to perform the tests for the respective Technical Unit.

3.2.4 Administrative Unit Supervisors

The Administrative Unit Supervisor (or designee) is a full-time laboratory staff member and supervises laboratory operations for the respective Administrative Unit. The Administrative Units are Operations and Environmental Sample Receiving. The Administrative Unit Supervisor's proof of experience may be found in the personnel records maintained by the MDH Human Resources Management office and personnel training files.

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If the Administrative Unit Supervisor is absent for fifteen (15) consecutive calendar days or more, a deputy with appropriate qualifications will perform the Administrative Unit Supervisor's duties.

The Administrative Unit Supervisor is responsible for:

- meeting the general and education requirements and qualifications found the respective position description;
- monitoring performance data and the quality of work for the respective Administrative Unit within ENV;
- approving personnel have appropriate education and technical background to perform the work for the respective Administrative Unit.

3.2.5 Laboratory Key Personnel Deputies

Other personnel meeting the requirements for key personnel positions may serve as deputy in the absence of the designee. In general, the deputies for key personnel are as follows:

- Laboratory Manager: the Division Director serves as deputy
- Unit Supervisor: the Laboratory Manager will appoint an ENV staff member to serve as deputy
- Quality Assurance Officer: the Division Director will appoint a PHL member staff to serve as deputy.

3.3 Quality Policy

Management's commitment to quality and to the management system is stated in the Quality Policy below, which is upheld through the application of related policies and procedures described in ENV's Quality Manual, SOPs and policies.

The objective of the management system and the commitment of management is to consistently provide our clients with data of known and documented quality that meets their requirements. ENV's policy is to use good professional practices, to maintain quality, to uphold the highest quality of service, and to comply with ISO/IEC 17025:2005(E), USEPA and any other applicable standards. ENV ensures that personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through the unequivocal commitment of management, at all levels, to the QA principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the ENV organization. Every ENV employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on ethical principles and procedures surrounding the data that is generated. ENV maintains a strict policy of client confidentiality.

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3.4 Ethics and Data Integrity System

ENV has an Ethics and Data Integrity policy and an Ethics and Data Integrity program, training and investigations which are discussed in the Ethics and Data Integrity policy for the Division.

More information:

Ethics and Data Integrity Policy

3.5 Documentation of Management/Quality System

The management system is defined through the policies and procedures provided in this Quality Manual and written laboratory SOPs and policies.

3.5.1 Quality Manual

The Quality Manual contains the following items:

3.5.1.1 document title;
3.5.1.2 ENV's full name, address and telephone number;
3.5.1.3 identification of all major organizational units which are to be covered by this Quality Manual and the effective date of the version;
3.5.1.4 the signed and dated concurrence (with appropriate names and titles), of all responsible parties including the Quality Assurance Officer(s), Unit Supervisor(s), Laboratory Manager, and Division Director;
3.5.1.5 the objectives of the management system and contain or reference ENV's policies and procedures;
3.5.1.6 ENV's official quality policy statement, which shall include management system objectives and management's commitment to ethical laboratory practices and to upholding the requirements of this Standard; and
3.5.1.7 a table of contents, and applicable lists of references, glossaries and appendices.

3.5.2 Standard Operating Procedures (SOPs)

SOPs represent all phases of current laboratory operations (they include an effective date, revision number, and signature of the approving authorities). The approving authorities for administrative, quality system and analytical documents are identified in the Document Control procedure. All laboratory documents are available to all ENV personnel. They contain sufficient detail such that someone with similar qualifications could perform the procedures. There are two types of SOPs used in ENV: 1) analytical SOPs, which have specific requirements as outlined below, and 2) general use SOPs which document general procedures (ENV, administrative or quality procedures).

Each analytical method has an SOP. ENV's analytical SOPs include the following topics, where applicable:

- identification of the method;
- applicable matrix or matrices;

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- limits of detection and quantitation;
- scope and application, including parameters to be analyzed;
- summary of the method;
- definitions;
- interferences;
- safety;
- equipment and supplies;
- reagents and standards;
- sample collection, preservation, shipment and storage;
- quality control;
- data assessment and acceptance criteria for quality control measures;
- corrective actions for out-of-control data;
- contingencies for handling out-of-control or unacceptable data;
- calibration and standardization;
- procedure;
- data analysis and calculations;
- method performance;
- pollution prevention;
- waste management;
- references; and
- any tables, diagrams, flowcharts and validation data.

3.5.3 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows unless otherwise noted:

1) Federal law or regulation
2) Statewide policy or procedure
3) Department policy or procedure
4) Division policy or procedure
5) Interagency Agreement, Memorandum of Understanding, or QAPP
6) Laboratory Quality Manual
7) SOPs and Policies
8) Other (Work Instructions (WI), memos, flow charts, etc.)

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4 DOCUMENT CONTROL

This Section describes how ENV establishes and maintains a process for document management. Procedures for document management include controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

Documents can be SOPs, policies, forms, worksheets, work instructions, reference methods, manuals, software, etc. These may be either hard copy or electronic documents.

ENV manages two types of documents, controlled and obsolete.

A controlled document is one that is uniquely identified, issued, tracked, and kept current as part of the management system. Internal controlled documents require final approval by management before use by laboratory personnel. External controlled documents are integrated and controlled into the quality system.

Obsolete documents are documents that have been superseded by more recent versions or are no longer needed.

4.1 Controlled Documents

Documents will be reviewed, revised (as appropriate) and approved for use prior to issue to ENV staff. Final review of Unit specific documents is performed by the Unit Supervisor and final approval is completed by the Laboratory Manager. Final review of quality system documents is performed by the Quality Assurance Officer and final approval is completed by the Division Director.

Documents are reviewed annually to ensure their contents are suitable and in compliance with the current management system requirements, regulatory requirements, and accurately describe current operations.

Copies of controlled documents are made available to staff at all locations within the laboratory where operations are essential to the effective functions of ENV. Printed and locally saved electronic copies of the documents may not be controlled and users must verify the accuracy of any printed or electronic convenience copies by comparing the revision number and content with the controlled document. A footnote appears on each page of the controlled document stating this information.

Controlled internal documents are uniquely identified with 1) a unique name or number identification, 2) date of issue, 3) revision identification, 4) page number, 5) the total number of pages (or a mark to indicate the end of the document), and 6) the signatures of the issuing authority (i.e. management).

A master list of controlled internal documents is maintained that includes title, revision, revision date and review date. A list of controlled copies is maintained that includes title, revision, and location. A master list of controlled external documents is maintained. The controlled document list is maintained and updated by the Quality Assurance Officer as needed.

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All employees must read and understand the content of documents that relate to the work that they perform. This compliance is documented in the employee's training file.

4.1.1 Document Changes to Controlled Documents

Document changes are approved by the role that performed the original final review. Suggested revisions to documents are presented to the approving authority for review and approval. Changes to documents are approved prior to editing of the document.

The document management process allows for handwritten modifications to documents. The modification(s), reason for modification, date of approval and approving authority are documented in the corrective action data system. The modifications are incorporated into the next scheduled revision (or sooner, if the change is critical or if the document must be distributed to external clients).

All document modifications must be approved. Changes that are not process modifications but clarifications may be performed without revision, however approval is still required. The modified document is then distributed, and obsolete documents are removed according to the master list of controlled documents.

Where practical, the altered text or new text in the draft is identified during the revision process to provide for easy identification of the modifications.

4.2 Obsolete Documents

All invalid or obsolete documents are removed from general distribution, or otherwise suitably marked to prevent unintended use.

Obsolete documents retained for legal use or historical knowledge preservation are appropriately marked and retained. At least one copy (either paper or electronic) of any obsolete document is kept by the Quality Assurance Officer for the time period allowed in the state-approved records retention schedule for ENV.

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5 REVIEW OF REQUESTS, TENDERS AND CONTRACTS

The review of all new work assures that oversight is provided so that requirements are clearly defined, ENV has adequate resources and capability, and the analytical method is applicable to the client's needs. This process assures that all work will be given adequate attention without shortcuts that may compromise data quality.

Contracts for new work may be formal bids, signed documents, verbal, or electronic. The client's requirements, including the methods to be used, must be clearly defined, documented and understood. Requirements might include target analyte lists, project specific RLs (if any), project specific QC requirements (if any), turnaround time, and requirements for data deliverables. The review must also cover any work that will be subcontracted by ENV.

5.1 Procedure for the Review of Work Requests

The Operations Unit ensures ENV has the necessary accreditations to meet the work request, if required. The Environmental Sample Receiving Supervisor and Technical Unit Supervisors determine if ENV has the resources, including schedule, equipment, deliverables, and personnel to meet the work request. The Operations Unit documents the review and approval via electronic mail, forms or notes of meetings with clients and laboratory management.

The Operations Supervisor informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the laboratory to the complete the work satisfactorily.

The client is informed of any deviation from the contract including the analytical method or sample handling processes. All differences between the request and the final contract are resolved and recorded before any work begins. It is necessary that the contract be acceptable to both ENV and the client. The Operations Supervisor records this information electronically.

The review process is repeated when there are amendments to the original contract by the client. The participating personnel are given copies of the amendments. The original contract and any amendments are maintained by the MDH Financial Management office and a convenience copy is retained by PHL for reference.

Note: For repetitive routine tasks, the review may be made only at the initial inquiry stage or on granting of a contract for ongoing routine work performed under a general agreement with the client, provided the client's requirements do not change.

ENV's Review of Requests, Tenders and Contracts procedure contains specific details about establishment and review of routine and large projects.

5.2 Documentation of Review

Records are maintained for every contract or work request, when appropriate. This includes pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

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Records of all project-related communication with the client (including e-mails, telephone conversation etc.) are kept electronically by the Operations Unit.

More information:

Requests, Tenders and Contracts

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6 SUBCONTRACTING OF ENVIRONMENTAL TESTS

A subcontract laboratory is defined as a laboratory external to this laboratory, or at a different location than the address indicated on the front cover of this manual, that performs analyses for this laboratory.

When subcontracting analytical services, ENV assures work requiring accreditation is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for performing the tests. ENV will ensure that the subcontract laboratory understands the requirements and will meet the same commitments made to the client by the primary laboratory.

6.1 Procedure for Subcontracting

The Operations Unit maintains a list of subcontractors in LIMS.

A copy of the certificate and analyte list from subcontractors is maintained as evidence of compliance. This information is maintained by the Operations Unit and is kept in LIMS.

The certificate and analyte list are reviewed by Operations Unit to ensure the subcontracting laboratory has the appropriate accreditation to do the work.

The Operations Unit notifies the client in writing of the intent to subcontract the work and the suggested laboratory that can perform the work. When possible, ENV gains the approval of the client to subcontract their work prior to implementation, preferably in writing.

The laboratory performing the subcontracted work is identified in the final report. ENV assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specified which subcontractor is to be used.

6.2 Approval of Subcontracting Laboratories

The Operations Unit or Technical Units may suggest a laboratory as a subcontractor based on need.

The Operations Unit must have supporting documentation on file prior to initiation of any work. A listing of all approved subcontracting laboratories and supporting documentation is available in the Operations Unit files and LIMS.

If a State of Minnesota-accredited laboratory is not available, the Operation Unit will note the reason for selection of a non-accredited laboratory and will request the following information (in addition to the items above):

- SOP for method. Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. A table of contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.

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- The most recent two sets of proficiency results and any associated corrective action. These should be updated annually.
- Example final report to confirm format is compliant and provides the necessary information.

The requested information is reviewed to ensure ENV and client needs are met.

More information:

Subcontracting Samples

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7 PURCHASING SERVICES AND SUPPLIES

ENV ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality, by using approved suppliers and products. General procedures and activities are described below.

PHL maintains more specific work instructions to follow the MDH Financial Management requirements, forms and electronic systems for purchasing, receiving, and storage of supplies.

More Information:

Purchasing Procedure

7.1 Procedure for Purchasing Services and Supplies

ENV staff review and approve the supplier of services and supplies and approve technical content of purchasing documents prior to ordering.

The electronic signature (i.e. username and password log-in) is included with the requests to purchase. By authorizing the request to purchase, the end user is indicating that the item(s) requested meet quality specification(s) (such as specific grades required in SOPs). The request is submitted for approval to a purchasing coordinator identified for ENV who ensures the division purchasing policies are followed.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. The purchasing documents contain the data that adequately describes the services and supplies ordered. The description may include type, class, grade, identification, specifications or other technical information.

The supplies received are inspected for breakage, leaks or any other damage. The supplies and chemicals are checked for expiration date, concentration, grade, storage conditions, and any other information indicated by ENV as appropriate to maintain the quality of a specific test. The supplies received are stored according to manufacturer's recommendations, laboratory SOPs or analytical method specifications. All standards, reagents and chemicals are tracked in the LIMS. The storage conditions for these items are identified in the ENV LIMS.

The vendor supplied Certificate of Analysis (CoA) received by ENV with standards, reagents and chemicals is scanned and attached to the consumable identified in LIMS. Other documents received by ENV with supplies and services including specifications, maintenance records, calibration records, etc. are retained by ENV. Documentation submitted to PHL purchasing agent(s) related to a purchase are filed with the purchase order in MDH Financial Management and archived accordingly.

The purchased supplies and reagents that affect the quality of the tests are not used until they are inspected or otherwise verified as complying with requirements defined in the analytical method.

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7.2 Approval of Suppliers

The MDH Financial Management office maintains a list of approved suppliers through SWIFT, a statewide centralized purchasing system.

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products, the overall quality of their services, their past history and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include CoAs, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all requests for purchases from specific vendors are evaluated and submitted by the end user and approved by the purchasing coordinator, as stated in the previous section.

Issues with vendors are reported to management by initiating a corrective action. Investigation of the issue and the number of issues from a vendor are evaluated to determine if a supplier should continue to be used.

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8 SERVICE TO THE CLIENT

ENV collaborates with clients and/or their representatives in clarifying their requests and in monitoring laboratory performance related to their work. Each request is reviewed to determine the nature of the request and ENV's ability to comply with the request within the confines of prevailing statutes and/or regulations without risk to the confidentiality of other clients.

8.1 Client Confidentiality

ENV's confidentiality policy is to not divulge or release any information to a third party without proper authorization. Third party requests for data and information are referred to the client. Data and records identified as private or confidential through the Minnesota Data Practices Act are protected from unintended distribution (i.e. public or unauthorized access).

All electronic data (storage or transmissions) are protected as required by client and regulation.

Specific data classifications for protection are in the PHL Data Inventory maintained by the state's Information Technology personnel (MN.IT) and available on the MDH Data Practices intranet site.

Unintended distribution is deterred by the addition of a Confidentiality Notice to the email signature lines of ENV personnel dealing with frequent client communications. The email notice below, or similar, is appropriate but may be modified based on the email contents, the clients contacted, or through directive from the Division Director or MDH Legal Unit.

Email notice: *This e-mail may contain private, confidential or trade secret information belonging to the sender who is legally privileged. If you are not the intended recipient: (1) you are prohibited from disclosing, copying, distributing, or taking any action in reliance on the contents of this message or attachment(s); (2) you should notify the sender immediately by reply e-mail or by calling the Public Health Laboratory by telephone (651.201.5300); and (3) you must delete the message and attachments received in error.*

More information:

Minnesota Data Practices Act
MDH Data Practices

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8.2 Client Support

Communication with the client, or their representative, is maintained to provide proper instruction and modification for testing. Technical staff is available to discuss any technical questions or concerns the client may have.

The client, or their representative, may be provided reasonable access to laboratory areas for witnessing testing.

Delays or major deviations to the testing are communicated to the client immediately. The Operations Unit is responsible for notifications to clients. The communications may be delivered by email, phone or in-person depending on the nature of the message and the timing required for delivery of the message and will be documented by the Operations Unit.

ENV will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

8.3 Client Feedback

ENV seeks both negative and positive feedback following the completion of projects and periodically for ongoing projects. Feedback provides acknowledgement, corrective actions where necessary, and opportunities for continuous improvement.

Negative client feedback is documented as a client complaint (see Section 9 – “Complaints”).

ENV receives client feedback in several ways. ENV staff meets regularly with clients in scheduled face-to-face meetings. The Operations Unit monitors phone requests and email requests to its direct line or email boxes (on test reports and client publications) as well as the phone calls and emails forwarded through the Public Health Laboratory’s general system.

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9 COMPLAINTS

The purpose of this Section is to assure that client complaints are addressed and corrected. This includes requests to verify results or analytical data. Complaints provide ENV an opportunity to improve laboratory operation and client satisfaction.

Complaints by clients or other parties are reviewed by management and an appropriate action is determined. All client complaints are documented by the person receiving the complaint and addressed to the Operations Unit.

If it is determined that the complaint has merit, the procedures outlined in Section 12 – Corrective Action are utilized. If it is determined that a complaint is without merit, it is documented, and the client is contacted by the Operations Unit.

A complaint such as a concern that data is repeatedly late should be reviewed for preventive action (see Section 13 – “Preventive Action”) to minimize a future occurrence.

Specific actions in logging and handling complaints are provided in ENV’s Complaints Resolution procedure.

More information:

Complaint Resolution

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10 CONTROL OF NONCONFORMING ENVIRONMENTAL TESTING WORK

Nonconforming work is work that does not meet acceptance criteria or requirements. Nonconformances can include departures from SOPs, analytical methods or unacceptable QC results (see Section 25 – "Quality Assurance for Environmental Testing"). Identification of nonconforming work can come through client complaints, QC, instrument calibration, evaluating consumable materials, staff observation, final report review, management reviews and internal and external audits.

10.1 Exceptionally Permitting Departures from Documented Policies and Procedures

Requests for departures from laboratory procedures are approved and documented by the Unit Supervisor, Quality Assurance Officer and Laboratory Manager. The approval is documented in the corrective action data system, on the bench sheets (i.e. log books) and in the LIMS, if applicable. If the permitted deviation will impact client data, the Operations Unit will contact the client and request approval for the deviation prior to the implementation of the departure. This communication will be documented by the Operations Unit. Planned departures from procedures or policies do not require audits or investigations.

10.2 Nonconforming Work

The responsibilities and authorities for the management of nonconforming work are detailed in the Nonconforming Work, Stop Work and Data Recalls SOP.

10.3 Stop Work Procedures

Stop work procedures are detailed in the Nonconforming Work, Stop Work and Data Recalls SOP.

More information:

Nonconforming Work, Stop Work and Data Recalls

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11 IMPROVEMENT

Improvement in the overall effectiveness of the laboratory management system is a result of the implementation of the various aspects of ENV's management system: quality policy and objectives (Section 3 – "Management"); internal auditing practices (Section 15 – "Internal Audits"); the review and analysis of data (Section 25 – "Quality Assurance for Environmental Testing"); the corrective action (Section 12 – "Corrective Action") and preventive action (Section 13 – "Preventive Action") process; and the annual management review of the quality management system (Section 16 – "Management Reviews") where the various aspects of the management/quality system are summarized, and evaluated and plans for improvement are developed.

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12 CORRECTIVE ACTION

Corrective action is the action taken to eliminate the cause(s) of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Deficiencies cited in external assessments, internal quality audits, data reviews, client feedback/complaints, control of nonconforming work or managerial reviews are documented and may require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk.

12.1 General Procedure

ENV uses a corrective action data system to document and track corrective actions. Specific work instructions for entry of nonconformances in the system are provided to staff in the corrective action data system document. Corrective actions are the responsibility of all laboratory personnel and can be initiated by any staff.

Upon discovering of a nonconforming event, the nonconforming event shall be documented within the corrective action data system within three business days. The initial documentation will record the known information pertaining to the nonconformance.

The initial response to the nonconforming event shall be completed within two weeks of documentation. The response will be documented in the corrective action data system. Based on the complexity of the nonconformance, the initial response can vary from resolution and closure, proposed plan for containment of event, assignment of root cause investigation, proposed corrective action, gathering of information, proposed timeline for completion or track and trend.

After the initial response has been documented and until such time that the event is resolved, monthly updates will be documented in the corrective action data system by the appropriate personnel, based on the nonconformance. This personnel may include the Quality Assurance Officer, Unit Supervisor, Laboratory Manager, lead analyst, and/or other laboratory staff. Based on the complexity of the nonconformance, the monthly updates can address items such as timeline for completion, containment of event, root cause investigation, track and trend, effectiveness of the corrective action or resolution and closure. Updates can occur more frequently than monthly, and if corrective actions are required, should occur as they are implemented and evaluated.

The resolution of each nonconformance event will be unique based on the circumstances of the event and the complexity of the resolution. This uniqueness also dictates that a set timeline for resolution of unique events is not feasible. Each event will have its own timeline and will end with successful resolution of the nonconformance and prevention its recurrence. Once complete, the event will be closed in the corrective action data system.

12.1.1 Root Cause Investigation

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The first step of the corrective action process starts with the initial investigation and determination of root cause(s) of the problem. Records are maintained in the corrective action data system for nonconformance requiring corrective action to show that the root cause(s) was investigated, and includes the results of the investigation.

Where there may be non-systematic errors and as such the initial cause is readily identifiable or expected random failures (e.g. failed QC), a formal root cause investigation may not be required and the process begins with selection and implementation of corrective action (also see Section 12.3 “Technical Corrective Actions”).

If guidance is needed for root cause investigation, consult with the Quality Assurance Officer or Unit Supervisor.

12.1.2 Selection and Implementation of Corrective Actions

Where corrective action is needed to resolve a nonconformance, potential corrective actions shall be identified. The action determined most likely to eliminate the nonconformance will be implemented. The corrective action should be appropriate to the magnitude and risk of the nonconformance.

Where uncertainty arises regarding which action to implement, appropriate personnel will recommend the corrective action most likely eliminate the nonconformance and prevent recurrence. This personnel may include the Quality Assurance Officer, Unit Supervisor, Laboratory Manager, lead analyst, and/or other laboratory staff.

The appropriate Unit Supervisor ensures that corrective actions are implemented within the agreed upon time frame.

12.1.3 Monitoring of Corrective Action

The Unit Supervisor and staff will monitor implementation and documentation of the corrective action to assure that the corrective actions were effective.

Routinely, issues logged into the corrective action data system are reviewed by management to monitor corrective action timelines and progress toward completion, determine appropriate action is taken, determine trends for escalation of issues, and assure the documentation is complete for corrective actions closed in the system.

12.2 Additional Audits

Where the identification of nonconformances or departures from normal laboratory procedures cast doubt on ENV's compliance with its own policies and procedures, ISO/IEC 17025:2005(E) requirements, USEPA regulations and/or client requirements, ENV ensures that the appropriate areas of activity are audited in accordance with Section 15 – “Internal Audits” as soon as possible.

The additional audits can be follow-ups after the corrective action has been implemented to ensure it is effective. These are done when a serious issue or risk to ENV have been identified.

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12.3 Technical Corrective Action

Sample data associated with a failed QC are evaluated for the need to be reanalyzed or qualified. Unacceptable QC results are documented, and if the evaluation requires root cause investigation, the cause and solution are recorded (also see Section 10 – "Control of Nonconforming Environmental Testing Work"*).*

Analysts routinely implement corrective actions for data with unacceptable QC measures. Initial correction may include reanalysis without further assessment. If the analytical SOP addresses the specific actions to take, they are followed. Otherwise, corrective actions start with investigation of the root cause of the problem.

Corrective actions for non-systematic errors or expected random failures are documented in the analytical logbooks, LIMS and the analytical data reports. Corrective actions for nonconformances that may recur (beyond expected random QC failures) or where there is concern that ENV is not in compliance with its own policies and procedures require documentation in the corrective action data system (see Section 12.1).

Technical Unit Supervisors review issues logged as corrective action items and suggest improvements, alternative approaches, and procedures where needed.

If the data reported are affected adversely by the nonconformance, the affected data is clearly identified in the report, the client is notified, and communication is documented.

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13 PREVENTIVE ACTION

Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of nonconformances or complaints.

Preventive action may include, but is not limited to: review of QC data to identify quality trends, regularly scheduled staff quality meetings to ensure staff is knowledgeable in quality procedures, review of client feedback to look for improvement opportunities, review of proficiency testing data to look for analytes that were nearly missed, annual managerial reviews, scheduled instrument maintenance, running a new LIMS in tandem with the old system to validate implementation and functionality, and other actions taken to prevent problems.

When improvement opportunities are identified or if preventive action is required, action plans are developed, implemented and monitored to reduce the likelihood of the occurrence of nonconformances.

Procedures for preventive actions include the initiation of such actions and subsequent monitoring to ensure that they are effective.

All personnel have the authority to offer suggestions for improvements and to recommend preventive actions, however management is responsible for implementing preventive action.

Preventive actions are documented in the corrective action data system and evaluated for effectiveness by management.

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14 CONTROL OF RECORDS

Records are a subset of documents and may be on any form of media, including electronic and hard copy. Records are objective evidence of activities that have been performed or results that have been achieved. Records allow for the historical reconstruction of laboratory activities related to sample handling and analysis.

ENV maintains a record system appropriate to its needs, records all laboratory activities, and complies with applicable standards or regulations as required. Records help establish factors affecting the uncertainty of the test and enable test repeatability under conditions as close as possible to the original.

14.1 Records Maintained

Records of all procedures to which a sample is subjected while in the possession of ENV are kept. ENV retains all original observations, calculations and derived data (with sufficient information to produce an audit trail), calibration records, personnel records and a copy of the test report in accordance with the records retention schedule and the Records and Information Management Policy. At a minimum, the following records are maintained by ENV to provide the information needed for historical reconstruction:

- all raw data, whether hard copy or electronic, for calibrations, samples and QC measures, including analysts' worksheets and data output records;
- all injections made during a sequence with sufficient documentation to account for any failures, reprocessing, or reinjection;
- a written description or reference to the analytical method(s) used, which includes a description of the calculations used to translate parametric observations into a reportable analytical value (a copy of all pertinent SOPs);
- laboratory sample ID code;
- date of analysis;
- time of analysis is required if the holding time is 72 hours or less, or when time critical steps are included in the analysis (e.g. extractions and incubations);
- instrument ID and instrument operating conditions/parameters (or reference to such data);
- all manual calculations (including manual integrations);
- analyst's initials/signature or electronic identification;
- sample preparation, including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results (including a copy of the final report);
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- QC protocols and assessment;
- software documentation, verification, and records of any changes to automated data entries;
- method performance criteria including expected QC requirements;

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- proficiency test results;
- records of demonstration of capability for each analyst;
- a record of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory record;
- correspondence relating to laboratory activities for a specific project;
- corrective action reports;
- preventive action records;
- copies of internal and external audits including audit responses;
- of all current and historical laboratory SOPs, policies and Quality Manuals;
- sample receiving records (including information on any interlaboratory transfers);
- sample storage records;
- data review and verification records;
- personnel qualification, experience and training records;
- archive records; and
- management reviews.

More information:

Records Retention Schedule
Records and Information Management Policy

14.2 Records Management and Storage

The PHL Division maintains a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

Data is recorded immediately and legibly in permanent ink. Data generated by automated data collections systems are recorded electronically. A single line strikeout is used to make corrections so that the original record is not obliterated, and the correction is initialed and dated. Corrections to electronic records are tracked using several different mechanisms depending on the electronic system used. If an audit trail is available in the software system, the audit trail is enabled. The audit may be reviewed during internal assessments or periodically by the Unit Supervisors or Data Reviewer as a function of data review. If an audit trail is not available (such as with spreadsheets used for calculations), corrections are noted by the person making the correction. Records are backed up nightly by MN.IT on a separate server from the initial storage location. Weekly, the backup server is archived on tape and moved offsite. Backup tapes are retained for one month, then purged.

Records, including electronic records, are easy to retrieve, legible, and protected from deterioration or damage; held secure and in confidence; and are available to accrediting bodies based on retention schedule or as required by regulation or contract. When possible, records that are stored only on electronic media are supported by the hardware and software necessary for their retrieval. Access to protected records is

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limited to laboratory management or their designees to prevent unauthorized access or amendment.

Each Unit has designated storage locations on the PHL network or in the LIMS. Records are stored by calendar year and managed by the ENV records retention schedule. When records are stored in both hardcopy and electronic format, unless otherwise noted, the electronic copy will be the primary record.

Records may be subject to litigation holds. To determine if a record is under litigation hold or if questions arise regarding litigation hold, consult with the PHL Records Coordinator, the MDH Records Management Office and/or the MDH Legal Unit.

In the event that ENV transfers ownership or goes out of business, records are maintained or transferred according to state laws and approved retention schedules. Records for this laboratory are considered the property of the State of Minnesota, regardless of the name or location of the facility producing the record. Appropriate regulatory and state legal requirements concerning laboratory records are followed.

14.3 Legal Chain of Custody Records

Evidentiary sample data are used as legal evidence. Evidentiary records are maintained in the same manner as all other records. ENV maintains a separate, secure location for the storage of evidentiary samples. The location is controlled access to enter the room and key access to access the storage refrigerator where samples are stored before and after analysis. Receipt and handling procedures of evidentiary samples are provided in the Civil and Criminal Chain of Custody document.

More information:

Civil and Criminal Chain of Custody

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15 ASSESSMENTS

Assessments (i.e. audits) measure laboratory performance and verify compliance with accreditation/certification and project requirements. Assessments specifically provide management with an ongoing assessment of the management system. They are also instrumental in identifying areas where improvement in the management/quality system will increase the reliability of data. Assessments are of four main types: internal, external, performance, and system. Section 15.5 discusses the handling of assessment findings.

15.1 Internal Assessments

Annually, ENV prepares a schedule of internal assessments to be performed during the year. These assessments verify compliance with the requirements of the management/quality system, including analytical methods, SOPs, the Quality Manual, ethics policies, data integrity, other laboratory policies, USEPA requirements and the ISO/IEC 17025:2005(E) Standard. ENV uses method-specific checklists, USEPA checklists and/or its own analytical SOPs to cover the elements for compliance.

It is the responsibility of the Quality Assurance Officer to plan and organize assessments as required by the schedule and requested by management. These assessments are carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be assessed.

In addition to the scheduled internal assessments, it may sometimes be necessary to conduct special assessments as a follow-up to corrective actions, PT results, complaints, regulatory assessments or alleged data integrity issues. These assessments address specific issues. The special assessments are conducted by ENV's Quality Assurance Officer or designee.

The area assessed, the assessment findings, and corrective actions are recorded. Assessments are reviewed after completion to assure that corrective actions were implemented and effective.

More information:

Manual for the Certification of Laboratories Analyzing Drinking Water
Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water

15.2 External Assessments

It is ENV's policy to cooperate and assist with all external assessments, whether performed by clients or an accrediting body. Management ensures that all areas of ENV are accessible to assessors as applicable and that appropriate personnel are available to assist in conducting the assessment.

ENV participates in external assessments from Region 5 of the USEPA for the review of analysis in compliance with the Safe Drinking Water Act, external assessments for accreditation to the ISO/IEC 17025:2005(E) standard, and compliance assessments by

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clients, data users or regulatory bodies for various other laboratory activities (e.g. Centers for Disease Control and Prevention, Ramsey County Hazardous Waste, Environmental Health – Radiation Control).

15.2.1 Confidential Business Information (CBI) Considerations/Trade Secret

During on-site assessments, on-site assessors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment."

The classification of data in the State of Minnesota is defined in Data Practices Act. There are no provisions in state law identifying data as Confidential Business Information; however, ENV protects data meeting the classification of trade secret under the Minnesota Uniform Trade Secrets Act.

Trade secret portions of each document must be clearly marked with the words "trade secret". Trade secret information may be redacted of references to client identity by the responsible laboratory official prior to removing the documents from ENV. Sample identifiers may not be obscured from the information.

When information is claimed as trade secret, ENV must place on (or attach to) the information at the time it is submitted to the assessor, a cover sheet, stamped or typed legend or other suitable form of notice, employing the term "trade secret".

More information:

Minnesota Data Practices Act
Minnesota Uniform Trade Secrets Act

15.3 Performance Assessments

Performance assessments may be Proficiency Test Samples, internal single-blind samples, double-blind samples through a provider or client, or anything that tests the performance of the analyst and method.

Proficiency Test Samples are discussed in Section 25 – "Quality Assurance for Environmental Testing".

15.4 System Assessments

ENV's management system is assessed though annual management reviews. Refer to Section 16 – "Management Reviews" for further discussion of management reviews.

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15.5 Handling Assessment Findings

Internal or external assessment findings are responded to within the time frame established by the assessment, typically 30 calendar days. A completion date is established by management for each action item and included in the response. The response to external assessments is led by the Quality Assurance Officer. The response to internal assessments is led by the Unit Supervisor for the assessed area.

The responsibility for developing and implementing corrective actions in response to findings is the responsibility of top management. Corrective actions are documented through the corrective action process described in Section 12 – “Corrective Actions”.

Assessment findings that cast doubt on the effectiveness of the laboratory operation to produce data of known and documented quality or that question the correctness or validity of sample results must be investigated. Corrective action procedures described in Section 12 – “Corrective Action” must be followed. Clients must be notified in writing if the investigation shows the laboratory results have been negatively affected and the clients’ requirements have not been met. The client must be notified within 5 business days after ENV discovers the issue. Laboratory management will ensure that this notification is carried out within the specified time frame and documented.

Findings of inappropriate activity are handled following the procedures outlined in Section 17 (Data Integrity Investigation).

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16 MANAGEMENT REVIEWS

Top management reviews the management system on an annual basis and maintains records of review findings and actions.

16.1 Management Review Topics

The following are reviewed to ensure their suitability and effectiveness:

- the suitability of policies and procedures;
- reports from managerial and supervisory personnel;
- the outcome of recent internal audits;
- corrective and preventive actions;
- assessments by external bodies;
- the results of interlaboratory comparisons or proficiency tests;
- changes in the volume and type of the work;
- client feedback;
- complaints;
- recommendations for improvement;
- other relevant factors, such as QC activities, resources, and staff training.

16.2 Procedure

The management review topics are discussed throughout the year during ad hoc and formal meetings with top management. At least annually, the Quality Assurance Officer assures the topics above are summarized in a formal report to the Division Director.

The report contains observations, recommendations and findings. The Quality Assurance Officer enters findings into the corrective actions system for investigation and resolution. When appropriate, the Quality Assurance Officer may enter recommendations into the corrective action system as quality improvement initiatives or preventive action opportunities.

Management will determine appropriate completion dates for action items and ensure they are completed within the agreed upon time frame.

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17 DATA INTEGRITY INVESTIGATIONS

In addition to covering data integrity investigations, this Section covers all topics related to ethics and data integrity policies, procedures and training.

The Public Health Laboratory is committed to ensuring the integrity of its data and providing valid data of known and documented quality to its clients. The elements in PHL's Ethics and Data Integrity program include:

- Documented data integrity procedures signed and dated by top management.
- An Ethics and Data Integrity Policy is signed, dated and distributed by the PHL Division Director.
- An Ethics and Data Integrity Policy is reviewed by all management and staff and acknowledgement is signed during initial employee training and annual review.
- Annual data integrity training.
- Procedures for confidential reporting of alleged data integrity issues (see Section 17.3).
- An audit program that monitors data integrity (see Section 15 – "Assessments") and procedures for handling data integrity investigations and client notifications.

17.1 Ethics and Data Integrity Procedures

The Ethics and Data Integrity Policy provides an over view of the program. Written procedures that are considered part of the Ethics and Data Integrity program include:

- Ethics and Data Integrity Policy
- Manual integration procedures
- Corrective action procedures (see Section 12 – "Corrective Action")
- Procedures for Data Integrity Investigations
- Records Management procedures
- Data Integrity training procedures

Management reviews data integrity procedures yearly and updates these procedures as needed.

More information:

Ethics and Data Integrity Policy
Manual Integration

17.2 Training

Data integrity training is provided as a formal part of new employee orientation and a refresher course is given annually for all employees. Employees are required to understand that any infractions of the laboratory data integrity procedures shall result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. This is discussed in the Ethics and Data Integrity Policy that every employee is required to sign annually.

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Attendance for required training is monitored through a signature attendance sheet or an electronic training management system.

The following topics and activities are covered:

- organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting;
- how and when to report data integrity issues;
- record keeping;
- training, including discussion regarding all data integrity procedures;
- data integrity training documentation;
- in-depth data monitoring and data integrity procedure documentation; and
- specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

When contracted technical or support personnel are used, the Laboratory Manager or designee is responsible for ensuring that they are trained to the laboratory's management system and data integrity procedures, competent to perform the assigned tasks, and appropriately supervised.

Topics covered are provided in writing and provided to all trainees.

17.3 Confidential Reporting of Ethics and Data Integrity Issues

Confidential reporting of data integrity issues is assured through the following procedures:

- direct reporting to an immediate supervisor, manager, or the Director's office; or
- anonymous reporting through a suggestion box (monitored by the Assistant Division Director).

The laboratory management assures the confidentiality of the employees involved. Employees may privately discuss ethical issues with the Director's Office or management team at any time. In all cases, the Director's Office must be informed of the need for any further detailed investigation.

More information:

Ethics and Data Integrity Policy
Disclosure of Information by Employees (Whistleblower Protection)

17.4 Investigations

All investigations resulting from data integrity issues are conducted confidentially. They are documented and notifications are made to clients who received any negatively affected data that did not meet the client's data quality requirements. Procedures for investigation are included in the Ethics and Data Integrity Policy.

More information:

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Ethics and Data Integrity Policy

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18 PERSONNEL

ENV employs competent personnel based on education, training, experience and demonstrated skills as required. ENV's organization chart can be found on the MDH intranet site.

More information:

Organizational Charts

18.1 Overview

All personnel are responsible for complying with all quality and data integrity policies and procedures that are relevant to their area of responsibility.

All personnel who are involved in activities related to sample analysis, evaluation of results or who sign test reports, must demonstrate competence in their area of responsibility. Appropriate supervision is given to any personnel in training and the trainer is accountable for the quality of the trainees work. Personnel are qualified to perform the tasks they are responsible for based on education, training, experience and demonstrated skills as required for their area of responsibility.

The laboratory provides goals with respect to education, training and skills of laboratory staff. These goals are outlined in employee performance reviews. Training needs are identified at the time of employment, when personnel are moved to a new position, during performance review or new responsibilities are added to their job responsibilities. Ongoing training, as needed, is also provided to personnel in their current jobs. The effectiveness of the training must be evaluated before the training is considered complete.

Contracted personnel, when used, must meet the same competency standards and follow the same policies and procedures that laboratory employees must meet.

18.2 Position Descriptions

Position descriptions are available for all positions that manage, perform, or verify work affecting data quality, and are located in the employee position description on the MDH HRM intranet site. An overview of top management's responsibilities is included in Section 3 – "Management".

Position descriptions include the specific tasks, minimum education and qualifications, skills, and experience required for each position.

More Information

Position Description and Position Data

18.3 Training

Training requirements of the Division are followed, as documented in the Training Records Policy.

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All personnel are appropriately trained and competent in their assigned tasks before they contribute to functions that can affect data quality. Work performed by the trainee shall be evaluated by the trainer performing one-on-one mentoring and observing the work. The employee's work will be reviewed, until the trainer and trainee determine that the trainee is able to complete the work independently. The trainee will demonstrate competency by independently performing the work and obtaining acceptable results. It is management's responsibility to assure personnel are trained. Training records are used to document management's approval of personnel competency. The date on which authorization and/or competence is confirmed is included.

Staff are required to continuously update their training record to maintain an accurate transcript of activities. Staff must maintain their training record in the format that is required by the Division. Supervisors or designees are required to review their staff training records on an annual basis. Training records include required employee orientation training, required job-specific training that includes laboratory-specific analytical methods, required Division and Section specific training, laboratory-specific safety training and all optional training that employees take for job development and continued education.

18.3.1 Training for New Staff

New staff members are provided training as outlined on the MDH HRM New MDH Employees intranet site. In addition, new employees are provided the following training, as appropriate:

Ethics and Data Integrity
Right-To-Know
Bloodborne Pathogens
MasterControl Basic User Training
Quality Manual review and acknowledgement
Job specific SOPs
Initial Demonstration of Capabilities
Laboratory-specific safety training

The Unit supervisor ensures new employees for the Unit receive the necessary training.

18.3.2 Ongoing Training

Staff members shall perform ongoing training and meet annual requirements. Staff members are given the following ongoing training, as appropriate:

Ethics and Data Integrity
Right-To-Know Training
Bloodborne Pathogens
Ongoing Demonstration of Capability
Annual Review of SOPs
Laboratory-specific safety training

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Attending training related to job function as applicable

More information:

Training Records Policy
New MDH Employees
Initial and Ongoing Demonstration of Capability

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19 ACCOMODATIONS AND ENVIRONMENTAL CONDITIONS

19.1 Environmental

The laboratory facility is designed and organized to facilitate testing of environmental samples in a safe manner. Environmental conditions are monitored to ensure that conditions do not invalidate results or adversely affect the required quality of any measurement.

The MDA/MDH Laboratory Building was designed specifically for the purposes of analytical testing and support services. Some of the key features of the building include:

The building ventilation system includes a heat-recovery wheel. The building has 100% outside air running through the labs with no recirculation. In addition, the air in the metals clean room area is HEPA-filtered.

Appropriate ventilation throughout the laboratory to ensure the work being performed is safe. The laboratory has fume hoods as well as snorkel ventilation for open bench areas.

The metals clean room, the routine metals area, and the radiation chemistry area are equipped with polypropylene hoods to protect the integrity of the hood surfaces and reduce risk of contamination from corrosion.

Backup generators provide power for all electrical functions within the laboratory.

Temperature is continually monitored through a wireless temperature monitoring system in the metals clean room, general chemistry laboratory and organics laboratory.

Environmental tests are stopped when the environmental conditions jeopardize the results.

19.2 Work Areas

Work areas may include access and entryways to the laboratory, sample receipt area, sample storage area, sample process area, instrumental analysis area, chemical and waste storage area and data handling and storage area.

Access to, and use of, areas affecting the quality of the environmental tests is controlled by restriction of areas to authorized personnel only. See Section 19.4 below.

The laboratory work spaces are adequate for their use, and appropriately clean to support environmental testing and ensure an unencumbered work area.

Laboratory space is arranged to minimize cross-contamination between incompatible areas of the laboratory. Volatiles analysis is conducted in a room separate from the rest of the laboratory. Electronic balances are located away from drafts and doorways, and may be located on marble balance tables to minimize the effects of vibrations. Biological work areas are cleaned and disinfected between uses.

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19.3 Floor Plan

A floor plan for the laboratory and administrative areas is published and maintained current by the MDH Facilities Management Section.

More information:

Building Layout- MDA/MDH Laboratory

19.4 Building Security

The MDA/MDH Laboratory building is a locked, secure area, and it is not open to the public. Badges activate card readers on doors to grant access to secure areas based on employee responsibilities and duties. Individuals must register at the Orville Freeman Office Building reception desk and receive one of three types of security badges:

"Lab Visitor" badges provide access to the front door and the atrium's turnstiles during regular business hours. These visitors then have access to the elevators and conference rooms on the 2nd and 3rd floors.

"Lab Staff" badges provide access to the general laboratory spaces throughout the building and general spaces in the Freeman Building during regular business hours.

"Contractor" badges provide access to all of the general laboratory spaces throughout the building and general spaces in the Freeman Building. These badges also provide access to the engineering spaces in both buildings.

Visitors must be escorted by an authorized employee while in the laboratory facility.

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20 ENVIRONMENTAL METHODS AND METHOD VALIDATION

Methods and/or procedures are available for all activities associated with the analysis of the sample including preparation and testing. For purposes of this Section, “method” refers to both the sample preparation and determinative methods.

Before being put into use, an analytical method is confirmed by a demonstration of capability or method validation process.

All methods are published or documented. Deviations from the methods are allowed only if the deviation is documented, technically justified, allowable by the reference method, authorized by management and accepted by the client.

Methods are listed in ENV’s LIMS and document control system.

20.1 Method Selection

A reference method is a method issued by an organization generally recognized as competent to do so. When a laboratory is required to analyze a parameter by a specified method due to a regulatory requirement, the parameter/method combination is recognized as a reference method.

ENV will use methods that meet both regulatory requirements and are appropriate to the needs of the client. Such methods will be based on the latest edition of the method unless it does not meet the needs of the client. When the regulatory authority mandates or promulgates methods for a specific purpose, only those methods will be used.

If a method proposed by a client is considered to be inappropriate or out-of-date, the client is informed and the issue resolved and documented before proceeding with analysis of any samples.

When a method is not specified by the client, or the proposed method is inappropriate, ENV will recommend a method that is appropriate to the end use of the data.

- If the data are to be submitted to a regulatory authority, the method(s) specified by the regulatory authority will be used.
- For drinking water compliance, a method will be selected from those specified in 40 CFR Part 141, or the applicable state regulations.
- For NPDES permits, the method will be selected from those specified in 40 CFR Part 136 or SW-846, as appropriate.
- If the end use of the data is not regulatory or if the regulatory authority does not specify a method, ENV will determine the client needs in terms of RL, bias (e.g., screening versus quantitative) and ENV’s capabilities and capacity. Based on these criteria, ENV will recommend an appropriate method based on the following hierarchy and obtain confirmation from the client:

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- Methods published in regional, national or international standards
- Methods published by other technical organizations such as ASTM, Standard Methods or AOAC
- Methods developed by the instrument manufacturer
- Laboratory-developed methods.

All communications between ENV and the client are documented.

20.2 Laboratory-Developed Methods

If ENV develops a method, the process of designing and validating the method is carefully planned and documented. All personnel involved in the method design, development and implementation will be in constant communication during all stages of development.

20.3 Method Validation

Validation is the confirmation, by examination and objective evidence, that the particular requirements for a specific intended use are fulfilled.

At a minimum, reference methods are validated by performing an initial demonstration of capability as described in the analytical SOP. At a minimum, the following procedure will be used to validate a method.

For each analyte of interest:

- Calibrate the instrument.
- If applicable, validate the linear dynamic range of instrument.
- Verify the calibration with a SCV.
- Perform a valid MDL study.
- Determine the RL.
- Verify the RL with an RLV standard. Evaluate the recovery based on analytical SOP; if unspecified in the analytical SOP, the criteria will be ±40%.
- Evaluate the precision and bias by analyze a minimum of four LCS, at a concentration specified by the analytical SOP, or if unspecified, at a level 1-4X the RL. Evaluate for precision and bias based on criteria established in the analytical SOP.

These procedures must be repeated whenever there is an implementation of a new method, a change in the method affecting how the analysis is performed, or change in the instrument that affects the sensitivity of analysis.

All methods that are not reference methods are validated before use. The validation is designed so that ENV can demonstrate that the method is appropriate for its intended use. All records (e.g., planning, method procedure, raw data and data analysis) are retained while the method is in use and to the records retention schedule. Based on the validation process, ENV will make a statement in the analytical SOP of the intended use requirements. The validity of the method for the intended use requirements will be documented in either the validation section of the analytical SOP or a separate

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acquisition details document. At a minimum, the procedures outlined above will be used to validate these methods.

Method validation and Demonstration of Capability procedures can be found in the ENV analytical SOPs and Initial and Ongoing Demonstration of Capability SOP.

More information:

Initial and Ongoing Demonstration of Capability
Demonstration of Capability Chemistry Form
Demonstration of Capability Microbiology Form

20.4 Estimation of Analytical Uncertainty

When requested, ENV will provide an estimate of the analytical uncertainty.

ENV will use in-house, statistically-derived LCS control limits based on historical LCS recovery data as an estimate of the minimum laboratory contribution to analytical uncertainty at a 99% confidence level. For methods for which LCS data is not available, ENV will identify all components of analytical uncertainty and make a reasonable estimation. The estimation shall be based on knowledge of method performance and previous experience. When estimating uncertainty, all components that are of importance for the given method will be taken into account.

20.5 Control of Data

To ensure that data are protected from inadvertent changes or unintentional destruction, ENV uses procedures to check calculations and data transfers (both manual and automated).

20.5.1 Computer and Electronic Data Requirements

ENV assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

- documented in sufficient detail and validated as being adequate for use;
- protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
- maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and
- held secure including the prevention of unauthorized access to, and the unauthorized amendment of, computer records. Data archive security is addressed in Section 14 – “Control of Records” and building security is addressed in Section 19- “Accommodations and Environmental Conditions”.

ENV resides in a security controlled building which prevents unauthorized access to laboratory spaces. ENV controls access to all programs that are used to acquire, process,

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record or report data. Computers and LIMS are password protected. Each employee is granted access only to those programs that he or she uses. The password is unique to the individual, and cannot be shared.

Access to and capabilities within ENV's LIMS are customizable and are dependent on an employee's responsibilities and job description (e.g. administrator, supervisor, reviewer, and analyst). An administrator is given access to change the settings, but may not modify any data entry.

Analysts enter data, add data qualifiers, and have an opportunity to verify data entries before permanently saving them. After the data has been saved, any modifications will be automatically documented in the LIMS audit trail. During review of the data, a reviewer may notify the applicable supervisor and analyst of any errors. The reviewer may update data qualifiers and determine which value will be reported to the client, in the event that repeat analysis occurred due to QC failure or sample dilutions. Any other changes are performed by the applicable supervisor or analyst. These modifications are captured in the LIMS audit trail. Once the data have been reviewed, it is authorized for release to the client.

ENV uses spreadsheets to calculate final results from the raw data for select tests. Before reporting any results derived from these programs, ENV shall validate the underlying calculations through manual calculation verification and peer review.

After the spreadsheet is validated, the calculations are protected from inadvertent manipulations.

20.5.2 Data Reduction

The analyst calculates final results from raw data or appropriate computer programs provide the results in a reportable format. The analytical methods provide required concentration units, calculation formulas and any other information required to obtain final analytical results.

ENV has manual integration procedures that must be followed when integrating peaks during data reduction. Significant figures are established for methods and maintained in the LIMS.

All raw data must be retained in the instrument data system, laboratory file server, analytical logbooks and/or LIMS and it is maintained as described in Section 14 – "Control of Records".

More information:

Manual Integration

20.5.3 Data Review Procedures

Data review procedures are located in Section 25.4 – "Data Review".

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21 CALIBRATION REQUIREMENTS

21.1 General Equipment Requirements

ENV provides all the necessary equipment required for the correct performance of the scope of environmental testing performed by ENV.

All equipment and software used for testing and sampling are capable of achieving the accuracy required for complying with the specifications of the environmental analytical methods as specified in ENV SOPs.

Equipment is operated only by authorized and trained personnel (see Section 18 – "Personnel"*)*.

ENV has procedures for the use, maintenance, handling and storage of equipment and they are readily available to laboratory personnel. This information is contained in manuals provided by the manufacturer of the equipment and individual analytical SOPs. They provide information on use, maintenance, handling and storage of the equipment. ENV maintains an equipment inventory that includes additional information on storage location.

All equipment is calibrated or verified before being placed in use to ensure that it meets ENV specifications and relevant standard specifications.

Test equipment, including hardware and software, are safeguarded from adjustments that would invalidate the test result measurements by limiting access to the equipment and using password protection where possible (see Section 20.5 – "Control of Data"*)*.

Equipment that has been subject to overloading, mishandling, given suspect results, or shown to be defective or outside specifications is taken out of service. The equipment is isolated to prevent its use or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for nonconforming work are followed and results are documented (see Section 10 – "Control of Nonconforming Environmental Testing Work" and Section 12 – "Corrective Action").

When equipment is needed for a test that is outside of permanent control of ENV, the laboratory ensures the equipment meets the requirements of this manual prior to its use by inspecting, calibrating, and/or verifying it.

Each item of equipment and software used for testing and significant to the results is uniquely identified. Records of equipment and software are maintained. This information includes the following:

- identity of the equipment and its software;
- manufacturer's name, type identification, serial number or other unique identifier;
- checks that equipment complies with specifications of applicable tests;
- current location;
- manufacturer's instructions, if available, or a reference to their location;

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- dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;
- maintenance plan where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications;
- any damage, malfunction, modification or repair to the equipment;

More information:

Equipment Inventory
Individual ENV Analytical SOPs

21.2 Support Equipment

Support Equipment includes, but is not limited to balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, volumetric dispensing devices, and sample preparation devices.

All support equipment is maintained in proper working order. Records are kept for all repair and maintenance activities, including service calls.

All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files.

Information regarding support equipment maintenance and calibration can be found in the Support Equipment SOP.

More information:

Support Equipment

21.3 Analytical Equipment

21.3.1 Maintenance for Analytical Equipment

All equipment is properly maintained, inspected, and cleaned following procedures outlined in instrument manuals and individual analytical SOPs.

Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis. Instrument malfunction is documented in instrument maintenance logs which become part of ENV's permanent records. A description of what was done to repair the malfunction and proof of return to control are also documented in the LIMS.

21.3.2 Instrument Calibration

Initial instrument calibration and continuing instrument calibration verification are an important part of ensuring data of known and documented quality. Procedures and criteria regarding instrument calibrations are provided within the Calibration and Standardization section of each analytical method. At a minimum, the instrument calibration procedures meet the requirements of the approved method.

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Unless otherwise specified in the individual analytical method, upon generation of a calibration curve and prior to the analysis of samples, both the calibration curve and the RL must be verified. This is accomplished through the analysis and a SCV and a RLV standard.

More information:

Individual ENV Analytical SOPs

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22 MEASUREMENT TRACEABILITY

Measurement quality assurance comes in part from traceability of standards to certified materials.

All equipment used affecting the quality of test results are calibrated prior to being put into service and on a continuing basis (see Section 21 – "Calibration Requirements"). These calibrations are traceable to national standards of measurement where available.

If traceability of measurements to national standards is not possible or not relevant, evidence for correlation of results through interlaboratory comparisons, proficiency testing, or independent analysis is provided.

22.1 Reference Standards

Reference standards are standards of the highest quality available at a given location, from which measurements are derived.

Reference Standards, such as ASTM Class 1 weights and reference thermometers, are used for calibration only and for no other purpose. Reference standards are calibrated by an entity that can provide traceability to national or international standards. The following reference standards are calibrated by an external body to a national standard as indicated in Section 21 – "Calibration Requirements" and per the Support Equipment SOP.

- Class 1 weights
- Precision Handheld Reference Thermometer with High Precision Platinum Resistance Probe

More information:

Support Equipment

22.2 Reference Materials

Reference materials are substances that have concentrations that are sufficiently well established to use for calibration or as a frame of reference.

Reference materials, where commercially available, are traceable to national standards of measurement, or to Certified Reference Materials, usually by a CoA. The CoAs are attached to the associated standard in the LIMS.

Purchased reference materials require a CoA where available. If a reference material cannot be purchased with a CoA, it is verified by analysis and comparison to a certified reference material and/or demonstration of capability for characterization.

Working standards, intermediate stock solutions or other reference materials are checked as far as is technically and economically practical. Working standards or intermediate stock solutions are checked against a second source during initial calibration. When a second source is not available, a separate lot from the same vendor is accepted as a second source. Working standards and intermediate stock solutions are

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given expiration dates when they are prepared based on method or regulatory requirements. These standards are used or removed from service by the expiration date.

Additional working standards such as working class weights or internal thermometers are checked using procedures found in the Support Equipment SOP.

More information:

Support Equipment

22.3 Transport and Storage of Reference Standards and Materials

ENV handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials.

Reference standards and materials are stored according to manufacturer's recommendations, analytical SOP requirements and separately from samples. Storage conditions for reference materials are documented in the LIMS.

22.4 Labeling of Reference Standards, Reagents, and Reference Materials

ENV has procedures for purchase, receipt and storage of standards, reagents and reference materials. Purchase procedures are described in Section 7 – "Purchasing Services and Supplies".

Expiration dates can be extended if the reference standard or material's integrity is verified. The extended date may not be beyond the expiration date of the referenced standards used to re-verify. In cases where the reference materials are limited or are expensive to replace, the expired standards are recertified by the vendor (e.g. PFC mass-labeled standards) and the new CoA and reference material expiration date is updated in the LIMS.

Reagent quality is verified during routine blank analyses.

22.4.1 Stock Standards, Reagents, Reference Materials and Media

Records for all stock (purchased) standards, reagents, reference materials, and media are maintained in the LIMS and include:

- the manufacturer/vendor name (or traceability to purchased stocks or neat compounds)
- the manufacturer's CoA or purity (if supplied)
- the date of receipt
- recommended storage conditions

All stock standards, reagents, reference materials or media received are entered into the LIMS. Upon entry, a unique ID is automatically generated. Required fields for entry include, but are not limited to, vendor, vendor lot, date of receipt, date of expiration

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and recommended storage conditions. An optional field exists for attaching manufacturer supplied CoA.

The CoA may contain both a certified value and nominal concentration for a standard. If the certified value is within four percent of the expected concentration, the nominal value may be used. If the deviation is greater than four percent, the certified value must be entered into LIMS.

If the original container does not have an expiration date provided by the manufacturer, it is assigned by ENV. Solid compounds, strong acids, strong bases and solvents are given an expiration date of five years from the date of receipt by ENV. All other solutions are given an expiration date of two years from the date of receipt by ENV. The laboratory generated label must include the expiration date.

In methods where the purity of reagents is not specified, analytical reagent grade is used. If the purity is specified, that is the minimum acceptable grade. Purity is verified and documented according to Section 7 – "Purchasing Services and Supplies".

22.4.2 Prepared Standards, Reagents, Reference Materials and Media

Records for prepared standards, reagents, reference materials, and media preparation include:

- traceability to purchased stock or neat compounds
- date of preparation
- an expiration date after which the material shall not be used (unless its reliability is verified by ENV)
- preparer's initials (if prepared)

All prepared standard, reagent, reference material or media are prepared following the procedures outlined in the analytical SOP for which they will be used and must be entered into the LIMS. Upon entry, a unique ID is automatically generated. Required fields for entry include, but are not limited to materials used to prepare, amount of material used, total volume, date of preparation, date of expiration, preparer's name and recommended storage conditions. Traceability is established by selection of materials used in the preparation and a link is established between the parent compound(s) and the progeny.

All containers of prepared standards, reagents, or materials must be labeled. The label will include, at a minimum, a unique ID and an expiration date.

Prepared reagents are verified to meet the requirements of the analytical method through internal QC measures and blank analysis.

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23 COLLECTION OF SAMPLES

ENV does not provide sampling services. ENV's responsibility in the sample collection process lies in supplying the sampler with the necessary shipping containers, reagent water, sample containers, preservatives, sample labels, COC forms, ice packs, and packing materials required to properly preserve, pack, and ship samples to ENV.

23.1 Sampling Containers

ENV offers clean sampling containers for use by clients. ENV obtains the containers per the procedures outlined in Section 7 – "Purchasing Supplies and Services" to ensure the containers are purchased from container manufacturers that meet required specifications. Prior to release, each lot of sample containers must pass the quality assurance process for each analysis/analyte that the container will be used for.

More information:

Bottle Blank Quality Assurance

23.1.1 Preparing Container Orders

Containers (containing or supplied with any required preservatives) are provided to the client upon request. Requests are submitted through the Bottle Order Form and Environmental Sample Receiving Unit procedures are followed to fulfill the request.

More information:

Sample Container and Supplies Request
Container Labeling
Sample Collection Kit Request
Bottle Order Form

23.1.2 Sampling Containers, Preservation Requirements, Holding Times

Sampling container, chemical preservation and holding time requirements can be found in the Environmental Laboratory Sampling and Analysis Guide. Further information regarding both chemical and thermal preservation is contained in the LIMS.

If preservation or holding time requirements are not met, the procedures in Section 10 – "Control of Nonconforming Environmental Testing Work" are followed.

More information:

Environmental Laboratory Sampling and Analysis Guide

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24 HANDLING SAMPLES AND TEST ITEMS

24.1 Sample Receipt

When client samples are received at ENV, the COC is reviewed, condition is documented, samples are given unique identifiers, and COC and container information is logged into the LIMS prior to the onset of analysis. In the interest of public health, the exact order of these procedures may not be followed to expedite the analysis of emergency samples received for noncompliance purposes. When possible, efforts will be made to follow these procedures, however, emergency samples may be received outside of normal business hours. ENV maintains the ability to respond to an emergency situation at any hour with a select group of staff. Typically, this group does not include members of the Environmental Sample Receiving Unit. In the event this happens, the samples typically will be logged in to the LIMS system prior to data being reported to the client, depending on the nature of the samples.

24.1.1 Chain of Custody

The COC or sample submission sheets from the field are reviewed. This documentation is completed in the field and provides a written record of the handling of the samples from the time of collection until they are received at ENV. ENV's Sample Acceptance Policy outlines what information is needed on this record. The COC also provides information on what type of testing is being requested and can act as an order for laboratory services in the absence of a formal contract. COC and any additional records received at the time of sample submission are maintained by ENV through scanned images maintained in the LIMS.

More information:

Sample Acceptance Policy
Electronic Scanning
MDH Chain-of-Custody

24.1.1.1 Legal Chain of Custody

ENV has procedures for legal chain of custody services. If samples are noted as being used for legal/evidentiary purposes, special chain of custody procedures are put into place by ENV. Shipping records are maintained with the COC, internal chain of custody is initiated that provides additional documentation of internal handling by analysts and a disposal record or record of return is provided.

More information:

Custody Procedures for Sample Shipment and Delivery
Civil and Criminal Chain of Custody

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24.2 Sample Acceptance

Procedures for opening shipping containers, examining and triaging samples are provided in Environmental Sample Receiving Unit procedures and login guides.

ENV has a sample acceptance policy and trip blank policy that are made available to sample collection personnel. The sample acceptance policy emphasizes the need for use of water resistant ink, providing proper documentation (to include sample ID and/or location, date and time of collection, collector's name, preservation type, sample type and any special remarks about the sample), labeling of sample containers to include a unique sample ID, use of appropriate containers, adherence to holding times, and sample volume requirements. The trip blank policy emphasizes the need for submitting trip blanks in the same manner as routine samples, following the unique identification requirements discussed in the sample acceptance policy. In addition, ENV has nonconformance/corrective action procedures to handle samples that do not meet the requirements above or show signs of damage, contamination or inadequate preservation. Data will be appropriately qualified where samples are reported that do not meet sample acceptance requirements.

ENV checks samples for the conditions above, by evaluating items from the Sample Condition Upon Receipt form (SCURF) as appropriate to evaluate sample acceptance. Criteria regarding preservation, holding time and sample volume requirements can be found in the Environmental Laboratory Sampling and Analysis Guide and the LIMS. If these conditions are not met, the nonconforming condition is noted in the LIMS and the client is contacted by a member of the Operations Unit prior to any further processing. The decision by the client to reject or proceed with analysis of the sample is documented, and the data are qualified in the report as necessary and appropriate.

More information:

Sample Acceptance Policy
Trip Blank Policy
Sample Entry
Triage
Sample Condition Upon Receipt
Environmental Laboratory Sampling and Analysis Guide

24.2.1 Preservation Checks

The following preservation checks are performed and documented upon receipt or at the analytical bench:

24.2.1.1 *Thermal preservation:*

- For thermal preservation, the temperature must be within 0 - 6°C unless otherwise stated.
- Samples that are delivered to the laboratory the same day as they are collected are likely not to have reached a fully chilled temperature. This is

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acceptable if the samples were received on ice and the chilling process has begun.
- Information regarding the presence of ice and representative sample temperature is recorded on the SCURF.

Chlorine checks:

- Samples received from potable water supplies (including source water) and requiring chlorine checks are tested for chlorine upon initiation of analysis.

pH checks:

- The pH of samples requiring acid/base preservation is checked upon transfer of the sample to the laboratory from the Environmental Sample Receiving Unit, initiation of analysis or completion of analysis.

24.3 Sample Identification

Samples are uniquely identified in a permanent chronological record within the LIMS to prevent mix-up and to document receipt of all sample containers. Samples are assigned sequential numbers that reference more detailed information kept in the LIMS. Subsamples, extracts and digestates are labeled with the parent sample identification.

The following information is documented in the LIMS system:

- Unique laboratory identification number
- Client or project name
- Date and time of receipt at laboratory
- Identification of person making the entries
- Date and time of sampling
- Unique field identification number
- Analyses requested (including applicable approved method numbers)
- Comments regarding rejection (if any)

All documentation received regarding the sample, such as memos or chain of custody, are retained in the LIMS and associated to the unique laboratory identification number.

24.4 Sample Aliquots / Subsampling

In order for analysis results to be representative of the sample collected in the field, ENV has subsampling procedures. Samples are thoroughly mixed prior to subsampling and a representative aliquot is removed, unless otherwise specific in the analytical SOP and/or reference method.

24.5 Sample Turnaround Time

Turnaround times are derived to meet client needs and to provide sufficient time to receive, prepare, analyze, process, review, and report data. All activities need to occur prior to the stated turnaround time for each sample submission, as indicated in LIMS. If

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there are challenges in meeting these times, or competing priorities, staff must consult with their Unit Supervisor for assistance in handling the challenges. If issues arise that will affect timely reporting of results such as instrument problems, reagent shortages, QC failures, etc., the issues must be communicate to the Unit Supervisor and the Operations Unit. A typical workflow for samples is listed in Section 27.2.

24.6 Sample Storage

Samples that require thermal preservation are stored under refrigeration that is +/-2°C of the specified preservation temperature unless regulatory or method specific criteria require something different. Temperature storage conditions are wirelessly monitored for required criteria, verified, and the verification is documented in Isensix ARMS.

Samples are held secure, as required. Samples are accessible only to laboratory personnel.

Samples are stored apart from standards, reagents, food or potentially contaminating sources to minimize cross-contamination. All portions of samples, including extracts, digestates, leachates, or any product of the sample is maintained according to the required conditions.

24.7 Sample Disposal

Samples are retained a minimum of 30 calendar days after the data has reported unless other arrangements have been made with the client.

Samples are disposed of according to Federal, State and local regulations. ENV procedures describe the disposal of samples, digestates, leachates, and extracts.

More information:

Sample Disposal Procedure

24.8 Sample Transport

Samples that are transported under the responsibility of ENV, where necessary, are done safely and according to storage conditions. This includes moving bottles within ENV. Specific safety operations are addressed outside of this document.

Sample shipping procedures for routine environmental samples are described in the Subcontracting Samples SOP.

More information:

Subcontracting Samples

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25 QUALITY ASSURANCE FOR ENVIRONMENTAL TESTING

ENV has procedures for monitoring the validity of the testing it performs. The quality of test results is recorded in such a way that trends are detectable, and where practicable, are statistically evaluated. To evaluate the quality of test results, ENV utilizes a combination of certified reference materials, control charting, proficiency testing samples, QC samples, replicate sample, confirmation analysis and at times comparison to historical data.

In addition to procedures for calibration, ENV monitors QC measurements such as blanks, laboratory control samples (LCS), matrix spikes (MS), duplicates, surrogates and internal standards to assess precision and accuracy. Proficiency Testing samples are also analyzed to assess laboratory performance.

QC data are analyzed and, when found to be outside predefined criteria, action is taken to correct the problem and to prevent incorrect results from being reported. The analytical SOPs outline permissible actions to take for common QC failures that may be encountered. If the necessary corrective action is not outlined within the analytical SOP, the actions taken shall be documented in the LIMS, instrument maintenance logs or bench sheets. Data associated with QC data outside of criteria and still deemed reportable will be qualified so the end user of the data may make a determination of the usability of the data – see Section 26 – "Reporting of Results".

25.1 Essential Quality Control Procedures

The QC procedures specified in analytical SOPs are followed by laboratory personnel. When regulations and analytical methods have multiple QC requirements, the most stringent of control procedures is used. If it is not clear which is the most stringent, consult with the Quality Assurance Officer for guidance.

For analytical methods that do not provide acceptance criteria for an essential QC element or where no regulatory criteria exist, acceptance criteria are developed. The criteria are described in individual analytical SOPs and if applicable, are listed as limits in the LIMS. In some specialized projects, the client may set criteria. These limits can be found in the Interagency Agreements, Quality Assurance Project Plans, or similar contract documents.

Procedures to monitor routine QC are located in the analytical SOPs and include such procedures as:

- use of laboratory control samples and blanks to serve as positive and negative controls for chemistry methods;
- use of laboratory control samples to monitor test variability of laboratory results;
- use of calibrations, continuing calibrations, certified reference materials and/or PT samples to monitor accuracy of the analytical method;
- measures to monitor analytical method capability, such as MDL, RL, and/or range of test applicability, such as linearity;
- use of regression analysis, internal/external standards, or statistical analysis to reduce raw data to final results;
- use of matching stable isotopes as internal standards, when possible;

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- use of reagents and standards of appropriate quality and use of second source materials as appropriate;
- procedures to ensure the selectivity of the analytical method for its intended use;
- measures to assure constant and consistent test conditions, such as temperature, humidity, rotation speed, etc., when required by analytical method;
- use of sterility checks for equipment, media and dilution water for microbiology;
- use of positive and negative culture controls for microbiology; and
- measures to monitor analytical method capability, such as Minimum Detectable Activity for radiochemistry.

25.2 Internal Quality Control Practices

Analytical data generated with QC samples that fall within all prescribed acceptance limits indicate the analytical method is deemed to be in control.

QC samples that fall outside QC acceptance limits indicate the analytical method is deemed to be out of control (nonconforming) and procedures listed in the analytical SOP shall be followed, corrective action may be required and/or that the data are qualified (see Section 10 – "Control of Nonconforming Environmental Testing Work" and Section 12 - "Corrective Actions").

Detailed QC procedures and QC limits are included in analytical SOPs, or where unspecified in the SOPs, are listed as limits within the LIMS. All QC measures are assessed and evaluated on an ongoing basis, and control charts can be generated in LIMS so that trends are detected.

25.2.1 General Controls

The following general controls are used:

25.2.1.1 Positive and Negative Controls such as:

- Blanks (negative)
- Laboratory control sample (positive)
- Sterility checks and control cultures (positive and negative).

25.2.1.2 Selectivity is assured through:

- absolute and relative retention times in chromatographic analyses;
- two column confirmation when using non-specific detectors;
- use of acceptance criteria for mass-spectral tuning (found in analytical SOPs);
- use of the correct method according to its scope assessed during method validation; and
- use of reference cultures (positive and negative) from a recognized manufacturer (where applicable).

25.2.1.3 Consistency, Variability, Repeatability, and Accuracy are assured through:

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- proper installation and operation of instruments according to manufacturer's recommendations or according to the processes used during method validation;
- monitoring and controlling environmental conditions (temperature, access, proximity to potential contaminants);
- selection and use of reagents and standards of appropriate quality;
- use of Class A glassware for critical applications (i.e. preparation of standards, reagents, measurement of sample volume, etc.);
- cleaning glassware appropriate to the level required by the analysis as demonstrated with method blanks and laboratory control samples and as described in the Glassware Processing SOP;
- following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;
- testing to define the variability and/or repeatability of ENV results, such as replicates; and/or
- use of measures to assure the accuracy of the analytical method, including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures.

More information:

Glassware Processing

25.2.1.4 Analytical Method Capability (also see Section 20 – "Environmental Methods and Method Validation") is assured through:

- establishment of the MDL or minimum detectable activity (radiochemistry) where appropriate;
- establishment of the RL; and/or
- establishment of the range of applicability such as linearity.

25.2.1.5 Data reduction is assured to be accurate by:

- selection of appropriate formulae to reduce raw data to final results such as regression;
- following specific procedures for data reduction such as manual integration procedures;
- periodic review of data reduction processes to assure applicability;
- microbiological calculations, data reduction, and statistical interpretations specified by each analytical method; and
- radiochemistry results reported with its counting uncertainty.

25.2.1.6 Sample specific controls are used to evaluate the effect of sample matrix on the performance of the selected analytical method (not a measure of laboratory performance):

Examples:

- Matrix Spike and Matrix Spike Duplicate (MS/MSD)

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- Surrogate Spikes
- Sample Duplicates

25.2.1.7 ENV describes the essential QC elements for chemistry, radiochemistry and microbiology within each individual analytical SOP. The procedure describes the element, frequency, acceptance criteria and the corrective action or where unspecified in the SOPs, are acceptance criteria listed as limits within the LIMS.

25.2.2 Specific Controls

25.2.2.1 Method Blanks

Method blanks are processed along with and under the same conditions as the associated samples to include all steps in the method. A method blank must be analyzed at a minimum of one per preparation batch. When no separate preparation method is used the batch is defined as the environmental samples that are analyzed with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples, not including method blanks, LCS, matrix spikes and matrix duplicates. The matrix of the method blank must be similar to the associated samples and be free from any analytes of interest. Method blanks are not required for some analyses such as: pH, conductivity, flash point, turbidity and total coliform presence/absence.

Contaminated blanks are identified according to the acceptance limits in the analytical SOPs or LIMS. When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem.

The contaminated blank must be qualified. Data that are unaffected by the blank contamination (non-detects or other analytes) are reported unqualified. Sample data that are suspect due to the presence of a contaminated blank are reanalyzed, qualified, or canceled.

25.2.2.2 Laboratory Control Samples

Laboratory Control Samples (LCS) (i.e. laboratory fortified blank) are prepared from analyte free water or other clean matrix, and spiked with verified and known amounts of analytes for the purpose of establishing precision or bias measurements.

LCS are prepared and analyzed at a frequency mandated by method, regulation, or client request, whichever is most stringent. The standard frequency is one per analytical batch or as otherwise stated in the analytical SOP. Exceptions would be for those analytes where no spiking solution is available, such as: solids methods, pH, color, temperature, dissolved oxygen or turbidity. When no separate preparation method is used the batch is defined as the environmental samples that are analyzed with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20

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environmental samples, not including method blanks, LCS, matrix spikes and matrix duplicates.

The analytes to be spiked in the LCS are specified in the analytical SOP. In some cases, a client may specify a list of analytes for spiking and the request is handled using ENV's nonconformance procedures, as a permitted departure.

The results of LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The calculation for percent recovery can be found in the glossary of this Quality Manual in Section 1.3.1.

The individual LCS is compared to the acceptance criteria in the analytical SOP, or where unspecified in the SOP, the acceptance criteria are listed as limits within the LIMS.

25.2.2.3 Matrix Spikes and Matrix Spike Duplicates

Matrix Spikes and Matrix Spike Duplicates (MS/MSD) are environmental samples fortified with a known amount of analyte to help assess the effect of the matrix on method performance.

ENV procedure for MS/MSD includes spiking appropriate analytes at appropriate concentrations, calculating percent recoveries and relative percent difference (RPD), and evaluating and reporting the results. The calculations for percent recovery and relative percent difference can be found in the glossary of this Quality Manual in Section 1.3.1.

Where there are no established criteria, ENV uses the client mandated control limits for MS/MSD. Acceptance criteria are documented in the analytical SOPs or LIMS. For MS/MSD results outside established criteria, a corrective action is documented or the MS/MSD and source sample data are reported with appropriate data qualifying codes. If a qualifier is to be used, evaluate the LCS for acceptance and qualify the data indicating that there may be matrix interference present.

25.2.2.4 Surrogate Spikes

Surrogate spikes are substances with chemical properties and behaviors similar to the analytes of interest used to assess method performance in individual samples. Surrogates are added to all samples (in analytical methods where surrogate use is appropriate) prior to sample preparation or extraction.

Surrogate recovery results are compared to the acceptance criteria as published in the mandated analytical method. Where there are no established criteria, ENV uses the criteria listed in the analytical SOPs or LIMS as surrogate control limits.

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For surrogate results outside established criteria, data are evaluated to determine the impact. Corrective actions include reanalysis, qualifying the data, or client discussions, as appropriate.

25.2.2.5 Reporting Limit Verification

The RL must be verified each time the instrument is calibrated, or monthly at a minimum. The concentration for all analytes of the verification standard must be less than or equal to the RL for the method. RL verifications are not required for radiochemistry, microbiology and some inorganic analyses including, but not limited to pH, conductivity, turbidity and dissolved oxygen.

The RLV standard may be a separate standard injection, or at the time of calibration, an appropriate standard from the calibration curve may be reprocessed against the calibration response.

The percent recovery for the RLV standard must meet the acceptance criteria as documented in the analytical SOP or LIMS. For results outside the criteria, the RLV standard must be reanalyzed prior to sample analysis or the RL must be raised to a level that meets this criteria. If the limit is raised, sample values may not be reported below the elevated limit.

25.3 Proficiency Test Samples or Interlaboratory Comparisons

25.3.1 Compliance to Accreditation Requirements

The Quality Assurance Officer and the Technical Unit Supervisors review the customer needs and accreditation requirements to determine the PT sample schedule. ENV analyzes at least two PT samples per calendar year for each analysis/matrix combination for which ENV is accredited to the ISO/IEC 17025:2005(E). For methods certified through the USEPA, ENV analyzes at least one PT sample per calendar year. An exception is made for analytes where there is no PT available from any approved PT provider at least twice per year. In these cases, the laboratory will run the PTs in the minimum time frame the PTs are available or not at all if they are not available.

The successive PTs are analyzed at least five months apart and no more than 7 months apart unless the PT is being used for corrective action to maintain or reinstate accreditation, in which case the dates of successive PT samples for the same accreditation analysis/matrix combination is at least fifteen days apart.

25.3.2 PT Sample Handling, Analysis and Reporting

PT samples are treated as typical samples in the normal production process where possible, including the same analysts, preparation, calibration, QC and acceptance criteria, sequence of analytical steps, number of replicates, and sample login. Preparation of PT samples, if required, must follow the instructions provided by the vendor. PT samples are not analyzed multiple times unless routine environmental samples are analyzed multiple times or dilutions are necessary. Where PT samples

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present special problems in the analysis process, they will be treated as laboratory samples where clients have special requests.

The type, composition, concentration and frequency of QC samples analyzed with the PT samples are the same as with typical samples.

Prior to the closing date of a study, laboratory personnel do not:

- Subcontract analysis of a PT sample to another laboratory being run for accreditation purposes.
- Knowingly receive and analyze a PT for another laboratory being run for accreditation purposes.
- Communicate with an individual from another laboratory concerning the analysis of the PT sample.
- Attempt to find out the assigned value of a PT from the PT Provider.

The Technical Unit Supervisors will ensure the staff have the resources necessary to complete the analysis and will ensure the analysis is completed prior to the closing date of the study. The Technical Unit Supervisors or Data Reviewer will review results prior to data being reporting and will coordinate with analytical staff and the Quality Assurance Officer if remedial PT samples are required.

The Quality Assurance Officer will maintain the schedule and coordinate ordering for PT samples. The Quality Assurance Officer or designee will ensure the data has been reviewed prior to submission and will submit the data for evaluation to the vendor or program operating the PT study. The Quality Assurance Officer will maintain a copy of the final report from the vendor or program and will monitor evaluation results for acceptability and trends. The Quality Assurance Officer will coordinate with the Technical Unit Supervisors when remedial PT samples may be required.

Results are reported within the range of the method applicable for the PT sample. If a result is below the range, the result will be reported as less than the reporting limit for the PT sample.

ENV institutes corrective action procedures for failed PT samples following the guidelines in Section 12 – “Corrective Action”.

Retention of PT records is similar to that maintained for regular environmental samples. In addition, the laboratory maintains a copy of the online data entry summary when the PT results are submitted online.

25.4 Data Review

ENV reviews all data generated in the laboratory for compliance with method, laboratory and, where appropriate, client requirements. ENV utilizes the LIMS in support of the data review process for all environmental data and a significant proportion of biomonitoring data. The LIMS serves as a repository for final results, raw data packages, client records, final reports, and other records associated with each sample. The LIMS provides ENV the ability to incorporate regulatory and method specific criteria for hold

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time, QC parameters, maximum contaminant levels and other items. These values are reviewed by the Quality Assurance Officer or Unit Supervisor for accuracy prior to sample analysis and may be reviewed during internal audits.

Upon entry of results by the analyst, typically through electronic data transfer, LIMS automatically evaluates whether data is in compliance with these parameters and will identify all data outside of the established acceptance limits. Examples of data that may be flagged by LIMS include but are not limited to blank spikes, matrix spikes, duplicates, surrogates, continuing calibration verification standards, RLV standards, second source calibration verification standards and hold time exceedances. If data has been identified as falling outside the established limits, the analyst qualifies any flagged data with the appropriate data qualifier and marks data as reportable or non-reportable. Once the analyst has completed their review process and imported the complete raw data package(s) into LIMS, they mark the data as analyzed. The secondary reviewer is a designated Data Reviewer, Unit Supervisor, or lead analyst that review the data after it has been marked as analyzed. Both the analyst and secondary data reviewer perform similar functions in reviewing the data's acceptability of QC measures and accuracy of each final result utilizing LIMS and associated uploaded raw data. Criteria commonly reviewed include but are not limited to the following, if applicable.

- the bench sheet and sequence for the standards used;
- any method specific information that is recorded on the bench sheet;
- the analyst initials and instrument name;
- calibration curves and second source standard(s);
- initial daily requirements before running samples; such as continuing calibration standards, tune standards, instrument blanks, etc.;
- the frequency of QC run during the analytical sequence, verifying that QC is being run after the required number of samples or the samples are analyzed within the required time;
- internal standard responses and retention times;
- instrument printouts and chromatograms;
- the data in the LIMS matches the instrument data by confirming all the samples and QC were electronically transferred and spot checks the data;
- all data that is manually changed, calculated and/or entered into LIMS;
- qualifiers are added correctly and are appropriate;
- samples are correctly marked as reportable or non-reportable
- posts data to the repository, if required.

Final reports are compared to raw data either directly or through several reviewed steps.

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26 REPORTING THE RESULTS

The result of each test performed is reported accurately, clearly, unambiguously, and objectively and complies with all specific instructions contained in the analytical method.

Laboratory results are reported in a test report that includes all the information requested by the client and necessary for the interpretation of the test results and all information required by the method used.

Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or method integrity.

26.1 Test Reports

The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse.

Unless there is a written agreement with the client, each test report generated contains the following information:

- a title, such as Final Report;
- the name and address of the laboratory, and the phone number and name of a contact person;
- unique identification of the test report on each page and a pagination system that ensures that each page is recognized as part of the test report and a clear identification of the end of the report, such as 3 of 10;
- the name and address of the client;
- the identification of the method used;
- a description of, the condition of, and unambiguous identification of the sample(s) tested, including the client identification code;
- the date of sample receipt when it is critical to the validity and application of the results, date and time of sample collection, dates the tests were performed, the time of sample preparation and analysis if the required holding time for either activity is less than or equal to 72 hours;
- the test results, units of measurement, an indication of when results are reported on any basis other than as received (e.g. dry weight), failures identified (ENV maintains a list of data qualifiers in the LIMS);
- the name, function, and signature or an equivalent electronic identification of the person authorizing the test report, and the date of issue;
- where relevant, a statement to the effect that the results relate only to the samples;
- Any non-accredited tests or parameters shall be clearly identified as such to the client when claims of accreditation to the ISO/IEC 17025:2005(E) Standard are made in the analytical report or in the supporting electronic or hardcopy deliverables; and
- A statement that the report shall not be reproduced except in full without written approval of the laboratory.

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26.2 Supplemental Test Report Information

When necessary for interpretation of the results or when requested by the client, test reports include the following additional information:

- deviations from, additions to, or exclusions from the analytical method, information on specific test conditions, such as environmental conditions, and any nonstandard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers;
- a statement of compliance/noncompliance when requirements of the management system are not met, including identification of test results that did not meet the laboratory and regulatory sample acceptance requirements, such as holding time, preservation, etc.;
- where applicable and when requested by the client, a statement on the estimated uncertainty of the measurement;
- where appropriate and needed, opinions and interpretations. When opinions and interpretations are included, the basis upon which the opinions and interpretations are documented. Opinions and interpretations are clearly marked as such in the test report.
- additional information which may be required by specific methods or client;
- qualification of results with values outside the calibration range as appropriate.

26.3 Environmental Testing Obtained from Subcontractors

Test results obtained from tests performed by subcontractors are clearly identified on the test report by subcontractor name and/or accreditation number.

The subcontractors report their results in writing or electronically. A copy of the subcontractor's original report is supplied with the test results.

26.4 Electronic Transmission of Results

All test results transmitted by telephone, e-mail, or other electronic means comply with the requirements of the ISO/IEC 17025:2005(E) Standard and associated procedures to protect the confidentiality and proprietary rights of the client (see Section 20 - "Environmental Methods and Method Validation").

26.4.1 Electronic Data Deliverables

EDDs are generated through the LIMS in a format that is acceptable by each requesting client.

26.5 Amendments to Test Reports

Amendments to information (i.e. sample information, qualifiers, results, etc.) on a test report after it has been issued are made only in the form of another document or data transfer. All supplemental reports meet all the requirements for the initial report and the requirements of this Quality Manual.

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Amended test reports include the statement, ”Amended Report,” or an equivalent form of wording to assure they can be differentiated from other test reports.

When it is necessary to issue a complete new report, the new report is uniquely identified and contains a reference to the original that it replaces.

More information:

Reporting

26.6 Exceptions

Additional reporting requirements as requested by the client may be provided.

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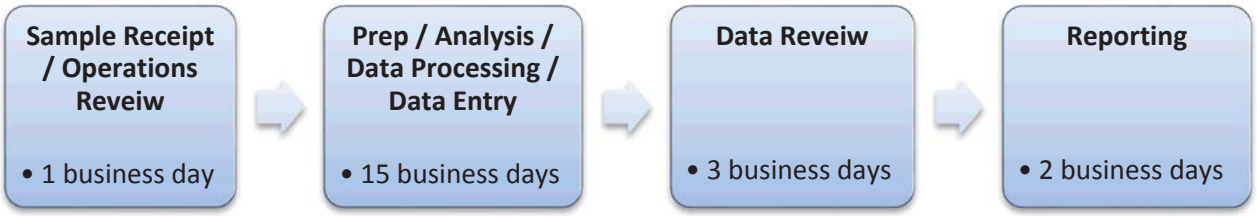
27 APPENDICES

27.1 Laboratory Accreditation/Certification/Recognition

USEPA Safe Drinking Water Act Scope of Certification

27.2 Typical Workflow

A typical workflow for samples with a 21 business day turnaround time.



Signature Manifest

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PROCEDURE FOR THE DETERMINATION OF:

VOLATILE ORGANIC COMPOUNDS (VOCs) IN WATER BY PURGE AND TRAP GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOLLOWING EPA METHOD 8260B

VOCs in Water by GC/MS - 498

Table of Contents

This procedure has been prepared for the sole use of the Minnesota Department of Health (MDH) Environmental Laboratory and may not be specifically applicable to the activities of other organizations.

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1.0 SCOPE AND APPLICATION

1.1 The following volatile organic compounds are the Target Analytes for this procedure:

Analyte	CAS	Analyte	CAS
Acetone	67-64-1	2,2-Dichloropropane	590-20-7
Allyl Chloride	107-05-1	1,1-Dichloropropene	563-58-6
Benzene	71-43-2	cis-1,3-Dichloropropene	10061-01-5
Bromobenzene	108-86-1	trans-1,3-Dichloropropene	10061-02-6
Bromochloromethane	74-97-5	Ethylbenzene	100-41-4
Bromodichloromethane	75-27-4	Ethyl Ether	60-29-7
Bromoform	75-25-2	Hexachlorobutadiene	87-68-3
Bromomethane	74-83-9	Isopropylbenzene	98-82-8
n-Butylbenzene	104-51-8	p-Isopropyltoluene	99-87-6
sec-Butylbenzene	135-98-8	Methylene Chloride	75-09-2
tert-Butylbenzene	98-06-6	Methyl Ethyl Ketone (MEK)	78-93-3
Carbon Tetrachloride	56-23-5	Methyl Isobutyl Ketone (MIBK)	108-10-1
Chlorobenzene	108-90-7	Methyl tert-Butyl Ether (MTBE)	1634-04-4
Chlorodibromomethane	124-48-1	Naphthalene	91-20-3
Chloroethane	75-00-3	n-Propylbenzene	103-65-1
Chloroform	67-66-3	Styrene	100-42-5
Chloromethane	74-87-3	1,1,1,2-Tetrachloroethane	630-20-6
2-Chlorotoluene	95-49-8	1,1,2,2-Tetrachloroethane	79-34-5
4-Chlorotoluene	106-43-4	Tetrachloroethene	127-18-4
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	Tetrahydrofuran (THF)	109-99-9
1,2-Dibromoethane (EDB)	106-93-4	Toluene	108-88-3
Dibromomethane	74-95-3	1,2,3-Trichlorobenzene	87-61-6
1,2-Dichlorobenzene	95-50-1	1,2,4-Trichlorobenzene	120-82-1
1,3-Dichlorobenzene	541-73-1	1,1,1-Trichloroethane	71-55-6
1,4-Dichlorobenzene	106-46-7	1,1,2-Trichloroethane	79-00-5
Dichlorodifluoromethane	75-71-8	Trichloroethene (TCE)	79-01-6
1,1-Dichloroethane	75-34-3	Trichlorofluoromethane	75-69-4
1,2-Dichloroethane	107-06-2	1,2,3-Trichloropropane	96-18-4
1,1-Dichloroethene	75-35-4	1,1,2-Trichlorotrifluoroethane	76-13-1
cis-1,2-Dichloroethene	156-59-2	1,2,4-Trimethylbenzene	95-63-6
trans-1,2-Dichloroethene	156-60-5	1,3,5-Trimethylbenzene	108-67-8
Dichlorofluoromethane	75-43-4	Vinyl Chloride	75-01-4
1,2-Dichloropropane	78-87-5	o-Xylene	95-47-6
1,3-Dichloropropane	142-28-9	p&m-Xylene 108-38-3	106-42-3

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1.2 This Standard Operating Procedure (SOP) is for a purge and trap, gas chromatography/mass spectrometry (GC/MS) method used for the determination of the volatile organic compounds (VOCs) listed above in drinking water, surface water, ground water and waste water.

1.3 This SOP is compliant with EPA Method 8260B, Revision 2. For introduction of the sample into the GC/MS system EPA Method 5030B is used. The preparation reference method is EPA 5000. Additional quality control guidance is from EPA 8000B and EPA Chapter 1, Chapter 2 and Chapter 4.

1.4 The procedure is restricted to use by or under the supervision of an analyst that is experienced in the use of GC/MS and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results by performing an initial and ongoing demonstration of capability.

2.0 SUMMARY OF METHOD

2.1 Samples and QC are delivered to the concentrator by an autosampler. The tray in the autosampler holds the samples at room temperature. The laboratory's extraction technique uses helium to purge the target analytes out of a 5-mL aliquot of water sample. The sample is contained in a specially designed purging chamber that is at near ambient temperature during this extraction. Volatiles are transferred from the aqueous phase to the vapor phase along with the purge helium. The vapor is swept through a sorbent tube, the trap, where the volatiles are held. After the purge cycle, the trap is heated and back flushed with helium to desorb the volatiles into a gas chromatographic (GC) system equipped with a capillary GC column.

2.2 A GC program uses a constant flow of helium and a temperature program to aid in the separation of the compounds within the column, and subsequently, the compounds are detected with a mass spectrometer (MS). Compounds are identified and quantified by comparing their retention times, mass spectra and responses to reference retention times, mass spectra and responses that are in the mass spectral libraries.

2.3 Reference retention times, spectra and calibration are obtained by the measurement of calibration standards analyzed under the same conditions used for the samples. The internal standard form of calibration is used. This means that the concentration of each identified compound is measured by relating the mass spectrometer response of the quantitation ion produced by that compound to the mass spectrometer response of the quantitation ion produced by a compound that is used as an Internal Standard. Surrogate analytes, with concentrations that are known in every sample, are measured with the same internal standard calibration

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procedure.

3.0 DEFINITIONS

3.1 Definitions that are common to all areas of the laboratory appear in Section 2.0 of the QA Manual and in Section 3.0 of the Organics SOP (most recent edition org006).

4.0 INTERFERENCE

4.1 Interferences that are common to all areas of the organic laboratory appear in Section 4.0 of the Organics SOP (most recent edition org006).

4.2 Field sampling site conditions, procedures and transportation procedures may have contaminated the samples. A Field Blank may determine if this contamination is present.

5.0 SAFETY

5.1 Safety precautions that are common to the Organics area of the laboratory appear in Section 5.0 of the Organics SOP (most recent edition org006).

5.2 The following analytes may be purged and have been classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, vinyl chloride and 1,4-dioxane. Standard materials and stock standard solutions should be handled in a hood. Exhaust from gas chromatograph split vents and mass spectrometer vacuum pumps should be properly trapped and/or vented.

6.0 EQUIPMENT AND SUPPLIES

6.1 Sample Containers:

6.1.1 Sample containers for use with non-chlorinated water supplies: Vials, 40 mL, screw cap with Teflon-faced silicone septa and containing 0.5 mL of HCl preservative added by the vial manufacturer. These vials are Precleaned Certified from Environmental Sampling Supply, Oakland, CA, PC Class Stock# 4050-W300-PC (or equivalent). After use these vials and caps are not to be washed, they are disposable.

6.1.2 Each lot of vials must be tested for quality. Refer to the Organics Sample Container SOP.

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6.2 Micro Syringes: Various sizes.

6.3 Glassware: Class A volumetric flasks and pipets, as required.

6.4 Autosampler: EST Centurion WS.

6.5 Purge and Trap System: EST Encon Concentrator that uses a 5-mL fritted disc sparge (or equivalent).

6.6 Gas Chromatograph/Mass Spectrometer: Hewlett Packard 6890/Agilent 5973 (or equivalent).

6.7 Data System: Enviroquant software product# G1701DA version D.01.00 (or equivalent).

6.8 LIMS Data System: Promium – Element Version 6.06:2013 (or equivalent).

7.0 REAGENTS AND STANDARDS

7.1 Reagent Water: Prepare by boiling distilled, deionized water for 20 to 30 minutes, cool and cover.

7.2 Methanol: Purge and trap grade, demonstrated to be free of analytes (Burdick & Jackson # 232-235 or equivalent).

7.3 Stock Standards: Solutions are purchased as certified standards from commercial suppliers:

7.3.1 Target Analytes: Single Custom Mix VOCs by Ultra Scientific, Part # CUS-12678. Dilute to make the Intermediate Standard solution, refer to Section 7.4.1.

7.3.1.1 At 2000-µg/mL for most analytes (p-Xylene and m-Xylene are at 1000 µg/mL, MEK and MIBK 10,000, Acetone and THF 20,000) in Methanol.

7.3.1.2 Store at < -10ºC and away from light.

7.3.1.3 Expiration date of stock is set by the manufacturer at one year from preparation of the mix.

7.3.2 Quality Control Sample (QCS): Four parts from Absolute Standards; EPA 524.2 Volatiles part #33003 – 78 compounds, EPA 502/524 High Conc. VOC Mix #1 part #30058 – 6 compounds, Dichlorofluoromethane part #61211 and Freon 113 part #90523.

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7.3.2.1 At 2000-µg/mL each analyte, in Methanol.

7.3.2.2 Store at < -10ºC and away from light.

7.3.2.3 Expiration date of stock is set by the manufacturer.

7.3.3 Internal Standard & Surrogate Standard Mixture [8 compounds] (ULTRA – STM-541 or equivalent): fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, dibromofluoromethane, 1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene and 1,2-dichlorobenzene-d4.

7.3.3.1 At 5000-µg/mL each analyte, in Methanol.

7.3.3.2 Store at < -10ºC and away from light.

7.3.3.3 Expiration date of stock is set by the manufacturer.

7.4 Intermediate Analyte Standards:

7.4.1 Target Analyte Intermediate Standard Solution (ISS) from Stock Standard (2000-ug/mL for most, refer to Section 7.3.1).

7.4.1.1 For calibration, RLV, CVS/LCS and MS spiking.

7.4.1.2 1000 µL Stock into 20 mL methanol = ISS at 100 ug/mL

7.4.1.3 Store at < -10ºC and away from light.

7.4.1.4 Expiration Date is 2 months from the preparation date and not beyond the date set by the manufacturer of the Stock Standards.

7.4.2 QCS Intermediate Standard Solution (QCS ISS) from Stock Standard (2000-ug/mL).

7.4.2.1 For calibration verification after an initial calibration. From a source that is separate from that used for calibration.

7.4.2.2 250 µL of each Stock into 5 mL methanol = QCS ISS at 100 ug/mL

7.4.2.3 Store at < -10ºC and away from light.

7.4.2.4 Expiration Date is 2 months from the preparation date and not beyond the date set by the manufacturer of the Stock Standard.

7.5 Calibration Standards (CAL):

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Level	**µg/L**	**µL *of Intermediate Standard***	***into***	***mL Reagent Water***	***Use***
1	0.5	0.5		100	MDL
2	1	1		100	RLV & MDL
3	5	5		100	MDL
4	10	10		100	CVS, QCS
5	20	20		100	
6	50	50		100	
7	75	75		100	
8	100	100		100	
9	200	200		100	

7.5.1 Fill a volumetric flask to the line with reagent water.

7.5.2 Inject the intermediate dilution standard rapidly with the needle of the syringe submerged in the reagent water, well into the expanded area of the flask.

7.5.3 Remove the needle as quickly as possible after the injection, bring the water volume to the line and stopper the flask.

7.5.4 Mix aqueous standards by inverting and righting the flask three times, only three times.

7.5.5 Fill a 40-mL preserved VOC vial with the prepared standard solution. Cap the vial and shake it vigorously to mix the standard with the preservative. Invert each vial to check for air bubbles that may have been trapped with the water. If an air bubble of 3 mm to 4 mm diameter or larger is trapped inside the vial, remake the standard.

7.6 Intermediate of Internal Standards and Surrogates:

7.6.1 10 µL of the IS/Surr Stock into 10 mL methanol = ISS at 5 ug/mL.

7.6.2 Store at the ambient room temperature, within the auto-sampler.

7.6.3 Expiration Date: 6 months from the preparation date and not beyond the date set by the manufacturer of the stock standard.

7.7 Surrogate Calibration: Using separate methods on the Centurion autosampler, the instrument injects appropriate amounts of ISS mixture for each desired calibration level as outlined in the following table:

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Level	**~µg/L**	**Injection Time (µL)**	***into***	***mL Reagent Water***
1	2	2		5
2	5	5		5
3	10	10		5
4	15	15		5
5	25	25		5

7.7.1 Average of Response Factors should be used for the each analyte's 5-point calibration curve fit. Response Factor Relative Standard deviation should be <15% for all surrogate calibrations. A Quadratic curve fit, correlation coefficient of =>0.99, may be used if the Average of Response is beyond 15%. The results (response) for the Surrogate Cal are entered into the Enviroquant calibration table by hand.

7.8 Laboratory Control Samples (LCS/LCSD): LCS are analyzed in the same manner as a sample. Spike at the L3 or L4 from the CAL level chart in Section 7.5 above. LCS are prepared on the day that they are analyzed.

7.9 Matrix Spikes (MS): Prepare an MS by injecting 4.4 µL of the intermediate dilution standard through the septa of the 40 mL (actual sample volume is 43 mL) vial that the field sample was collected in, shake the MS vigorously. This results in a spike concentration of 10 µg/L for each analyte in the sample (a few analytes are higher). A single MS or an MS/MSD pair can be prepared, as needed. MS are prepared on the day that they are run. Prepare and analyze the MS in the same manner as the samples, throughout the analytical procedure.

7.10 Method Blank (MB): MBs are prepared in and by the laboratory. Add reagent water to VOC vial containing 0.5 mL of 1:1 HCl, just to overflowing. Cap the vial and shake the MB vigorously to mix the sample with the preservative. MBs are prepared on the day that they are run.

7.11 Calibration Verification Standard (CVS): CVS are the same as LCS; procedural standards.

7.12 Trip Blanks: Prepared in and by the laboratory, in the same manner as MBs, refer to Section 7.9. Trip blanks are prepared in a set, one set contains 3 vials. TBs are preserved in the same manner as the samples that they are sent out with and from the same vial and preservative lot number. They must be chilled to 4ºC $\pm$ 2ºC on the day that they were prepared and maintained at that temperature until analysis. TBs are marked with an expiration date of 60 days. They are sent out with each set or group (a group is many sampling points on one site or day) of empty sample

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vials to the sampling site and returned to the laboratory with the filled sample vials for analysis.

7.13 Field Blanks: Prepared in the field by the sampler, in the same manner as a sample, but with reagent water. This water is brought to the field and poured into preserved VOC vials. Field Blanks are prepared in a set, one set contains 3 vials. They must be chilled to 4ºC $\pm$ 2ºC on the day that they were prepared and maintained at that temperature until analysis. Analyze FBs within 14 days of preparation. They are sent out, by request, with empty sample vials to the sampling site and returned to the laboratory with the filled sample vials for analysis.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Collection:

8.1.1 Remove the sampling container cap. Be careful not to touch the inside of the sampling container or cap with your fingers. Set the cap on a clean surface.

8.1.2 Sample Containers: Three (3) clear 40 ml vials per site. Each vial contains 0.5 mL of a 1:1 HCl liquid preservative. DO NOT RINSE OR SEVERLY OVERFILL THE VIALS. Position the vial at a slight angle while it is under the water flow (6-8 inches below the faucet). Do not allow the lip of the vial to touch the distribution vessel. The objective is to collect the water with as little agitation as possible. When the flow hits the inside wall of the vial (with the vial at a slight angle), it is agitated less than if the initial flow was to hit the flat bottom of the vial. Add water until the vial is just beginning to overflow.

8.1.3 If needed, add a little more water to the point where the vial is just overflowing, forming a meniscus (the curved upper surface of a liquid formed by surface tension).

8.1.4 Screw the cap on the vial so that the Teflon® side of the septum (shiny, smooth side) is in contact with the water. Do not touch the septum and do not over tighten the cap.

8.1.5 Check for air bubbles by inverting the vial and tapping it lightly. This tapping will dislodge air bubbles from the sides of the vial and from under the cap. The air bubbles must not be larger than a pea. Smaller pinhead sized air bubbles are at times unpreventable. If bubbles are present then discard this vial and pour a fresh aliquot of sample into another vial. Check for air bubbles. Air bubbles result in lower recoveries during analysis.

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8.1.6 Shake the sampling container well.

8.2 Sample Storage and Holding:

8.2.1 The samples must be chilled to 4°C ± 2°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be 4°C upon arrival at the laboratory. The sample storage area must be free of organic solvent vapors and direct or intense light.

8.2.2 The expiration date on the vials must be checked by the collector. If an expiration date is exceeded and noted by the collector (on the C of C) then a comment is required on the report: Results Suspect. This sample was collected in a vial that expired. The expiration date was (insert date here) and the sample was collected on (insert date here).

8.2.3 Analyze all samples and field blanks within 14 days of collection. Samples not analyzed within this period must be discarded and re-sampled.

8.2.4 Trip blanks must be used and analyzed within 60 days of preparation. The Trip Blank is given the same collection date as the sample(s) that it is associated with, and analyzed with the samples that it is associated with. An expired trip blank (older than 60 days from preparation.) can be analyzed, but a comment is required on the report: Results Suspect. This trip blank expired as of (insert date here).

9.0 QUALITY CONTROL

9.1 Corrective Action: When it has been determined that a corrective action should be initiated follow the procedures in the Corrective Action SOP (most recent edition qao011).

9.2 Method Validation: The Organics SOP Method Validation steps and procedures must be performed prior to routine analysis. Below is a list of the limits that are specific for this SOP.

9.2.1 External Verification of Calibration:

9.2.1.1 QCS deviation ≤ 30% from the true value.

9.2.2 Initial Demonstration of Capability:

9.2.2.1 Recovery must be 80-120% of the true value, in each replicate.

9.2.2.2 Mean accuracy must be 80-120%.

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9.2.2.3 %RSD must be ≤ 20%.

9.2.3 If the criteria cannot be met take corrective action and repeat the procedure for that analyte until satisfactory performance is achieved. The study is on file in the MDH Environmental Laboratory.

9.3 Ongoing Demonstration of Acceptable Performance: To demonstrate acceptable routine performance the following are required; an instrument BFB tune check, a RLV, beginning and bracketing mid-level CVS/LCS and a MB. Internal Standards & Surrogates are added to all QC and samples; the response and recovery must be monitored. One Matrix Spike/Matrix Spike Duplicate set is required per 20 samples run. Field Duplicate samples are not required, but recommended.

9.3.1 BFB Tune Check: A tune check verifies that the associated data is collected with a detector that is measuring the proper masses in the proper ratios, as established during the initial tune. The initial tune is normally performed prior to the Target Analyte calibration and then used during the daily analytical runs. A re-tune (initial tune) of the detector can be performed at any time, recalibration of Target Analytes is not necessary after a tune.

9.3.1.1 Preparation of a tune check is not needed. Perform the check on the Surrogate 4-bromofluorobenzene (BFB) in the Method Blank. This Surrogate is added into the GC at 25-ng or less and a mass spectrum is acquired at m/z 35-260 at 70eV (nominal) or SIM masses are collected for each of the tune components.

9.3.1.2 Include the BFB tune check as part of the examination of the analytical batch, after it has been run. Perform this check on each CVS/LCS or MB. No more than 12 hours of run time is allowed between tune checks.

9.3.1.3 Acceptable tune checks will meet the ratio requirements found in Table 2 of this SOP. Use the Autofind function to perform the tune check:

9.3.1.3.1 All criteria must pass. No failures are allowed.

9.3.1.4 If these criteria are not met the problem must be resolved.

9.3.1.4.1 When the Autofind tune check does not meet the criteria a manually integrated average spectrum, of at least three scans across the 4-BFB peak, can be

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used to evaluate the tune. A passing manual check is a substitute for the Autofind tune check.

9.3.1.4.2 If an analytical batch is completed with a failing BFB tune, the data cannot not be used.

9.3.1.4.3 The mass spectrometer must be adjusted, retuned or cleaned, to meet the BFB tune criteria before proceeding with the analyses.

9.3.2 Report Limit Verification (RLV):

9.3.2.1 Prepare the RLV as instructed and at the level that is specified in Section 7.

9.3.2.2 Include a RLV after the beginning CVS and prior to the samples. One RLV must be run monthly.

9.3.2.3 Acceptable RLV are a measurement & confirmation of the instrument's calibration accuracy at the Report Limit. The following criterion must be met for each analyte:

9.3.2.3.1 Deviation ≤ 40% of true value.

9.3.3 Calibration Verification Standard (CVS) & Laboratory Control Sample (LCS/LCSD): The CVS and LCS are combined as a procedural standard.

9.3.3.1 Prepare the CVS/LCS as instructed and at the levels that are specified in Section 7.5; a 10 µg/L standard.

9.3.3.2 Include a beginning CVS/LCS and bracketing CVS/LCS every 12 hours with a maximum run of 20 samples between them. Generally 10 samples are run in an analytical batch. For this procedure the analytical and preparation batches are the same.

9.3.3.3 Acceptable recoveries for the CVS/LCS are a measurement & confirmation of the accuracy while the relative percent difference (RPD) between the LCS and LCSD pair is a measurement of precision (Zero is used as the background for the needed calculations). The following criteria must be met for each analyte (CCC & SPCC have unique criteria):

9.3.3.3.1 Recovery must be between 70-130% of the true value.

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9.3.3.3.1.1 No more than 10 of the Target Analytes (15% of the total if fewer than 68 target analytes are requested for analysis) are allowed to exceed this limit.

9.3.3.3.2 Deviation ≤ 20% of true value (80 - 120%) for Calibration Check Compounds (CCCs): 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene and vinyl chloride.

9.3.3.3.3 RPD must be ≤ 30%.

9.3.3.3.4 System Performance Check Compounds (SPCCs) must meet the minimum average response factor requirements. SPCCs are: chloromethane 0.10, 1,1-dichloroethane 0.10, bromoform 0.10, chlorobenzene 0.3 and 1,1,2,2-tetrachlroethane 0.3.

9.3.3.4 If a CVS/LCS analyte is outside of the acceptance limit and the analyte is found in an associated sample, then the sample should be re-analyzed with a passing CVS/LCS pair.

9.3.3.5 If the 10 limit, 15% of total, is exceeded, then the associated analytical batch should be re-analyzed with a passing CVS/LCS pair.

9.3.3.6 If a limit is exceeded then reanalyze with these suggested corrections:

9.3.3.6.1 CVS/LCS prepared from a fresh vial of Intermediate Standard solution.

9.3.3.6.2 Prepare a fresh batch of Intermediate Standard solution for CVS/LCS preparation.

9.3.3.6.3 Prepare a fresh Internal Standard /Surrogate Solution for within the Autosampler.

9.3.3.6.4 Retune the detector.

9.3.3.6.5 Instrument maintenance as found in Section 10.2.

9.3.3.6.6 Recalibrate.

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9.3.3.7 If CVS/LCS fail to pass the required criteria with repeat runs, recalibration and within the specified holding time of the related samples, then the associated samples will require a qualifier.

9.3.4 Matrix Spikes (MS/MSD):

9.3.4.1 Prepare the MS as instructed and at the levels that are specified in Section 7. The concentration spiked into the MS sample should exceed the background concentration of the analyte in the corresponding unspiked sample (source) by a factor of five.

9.3.4.2 Include one of the following per 20 samples:

9.3.4.2.1 An MS/MSD pair. Extra sample volume/vials should be supplied by the client or collector (for a total of five vials).

9.3.4.2.2 A duplicate field sample and a single matrix spike. Refer to Section 9.2.5 for duplicate sample preparation and acceptance criteria.

9.3.4.3 Acceptable recoveries for the MS/MSD are a measurement of accuracy while the relative percent difference (RPD) between the MS and MSD pair is a measurement of precision (the un-spiked sample is used as the background for the needed calculations). Refer to Section 12.7 of the Organics SOP for the calculations. The following criteria must be met for each analyte:

9.3.4.3.1 Recovery must be between 70-130% of the true value.

9.3.4.3.2 RPD must be $\leq$ 30%.

9.3.4.4 If MS fail to pass the required criteria the sample must contain a qualifier for each failure. The source sample will require the qualifier and the MS results are to be reported with the batch QC.

9.3.5 Field Duplicates:

9.3.5.1 Preparation of duplicates is the same as for samples.

9.3.5.2 Include the duplicate pair as part of the analytical batch.

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9.3.5.2.1 Duplicate pair are normally blind, meaning there is a number of samples from a site and a site duplicate. This site duplicate is analyzed and reported as a normal sample. The data user receives the report and examines the results.

9.3.5.2.2 If a field duplicate is to be run and examined at the laboratory level, then mark and enter the duplicate in the LIMS system as a duplicate sample within the Bench Sheet and transfer as a QC item. It will appear in the QC portion of the report as a duplicate sample with RPD calculations.

9.3.5.3 Acceptable relative percent difference (RPD) between the sample and duplicate (DUP1) pair is a measurement of precision. The following criterion should be met for each analyte:

9.3.5.3.1 RPD must be ≤ 30%.

9.3.5.4 If failures are found, the sample must contain a qualifier for each failure.

9.3.6 Method Blank (MB):

9.3.6.1 Prepare the MB as instructed in Section 7.

9.3.6.2 Include a MB after the beginning CVS/LCS and prior to the samples. Every analytical batch must have a MB.

9.3.6.3 Acceptable MB results, ideally, will not have the Target Analyte(s) present. The following criterion must be met for each analyte:

9.3.6.3.1 Results must be <RL.

9.3.6.4 If a passing MB result is not possible, with repeat runs, recalibration and within the specified holding time of the related samples, then a qualifier will be required.

9.3.7 Internal Standard:

9.3.7.1 Prepare the Internal Standards as instructed in Section 7.

9.3.7.2 Include the addition of the Internal Standard(s) as part of the preparation procedure. Add a consistent/constant amount of the

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Internal Standard(s) to all samples, blanks and standards in an analytical run. This amount is the same as the amount added to the Calibration Standards.

9.3.7.3 Acceptable Internal Standards must meet the following criteria for the quantitation ion (absolute) area:

9.3.7.3.1 Response of the IS in the CVS must also remain above 50% and not beyond 200% (-50% to +100%) of the mean area of the IS response measured during the initial calibration.

9.3.7.3.2 Response of the IS in the samples should not vary by more than 50% from that in the beginning CVS/LCS.

9.3.7.3.3 Response should remain relatively constant for everything that is run in an analytical batch. An abrupt change may indicate a matrix effect or an instrument problem.

9.3.7.4 If IS responses are found to be unacceptable in either the QC component or in a sample with repeat runs, recalibration and within the specified holding time, then a qualifier will be required.

9.3.8 Surrogates:

9.3.8.1 Prepare the Surrogates as instructed in Section 7.6.

9.3.8.2 Include the Surrogate(s) as part of the preparation procedure. Add a consistent/constant amount of the Surrogate(s) to all samples, blanks and standards in an analytical run. This amount is the same as the amount added to the Calibration Standards.

9.3.8.3 Acceptable Surrogates must meet the following criteria:

9.3.8.3.1 Recovery must be 70-130% of the true value.

9.3.8.3.2 Responses, quantitation ion (absolute) area responses, should remain relatively constant for everything that is run in an analytical batch. An abrupt change may indicate a matrix effect or an instrument problem.

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9.3.8.4 If Surrogates are found to be unacceptable with repeat runs, recalibration and within the specified holding time, then a qualifier will be required.

9.3.9 External Verification of Laboratory Performance: Analyze performance (PE) samples twice per year and when available. Refer to Section 9.1.10 of the Organics SOP for details.

9.3.10 MDL Study: Repeat the MDL study once every year. Also repeat the study when changes in instrumentation and/or method occur. Refer to Section 9.1.6 of the Organics SOP for details.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Method Validation & Initial Demonstration of Capability (IDC): The Validation & IDC list of steps and procedures must be performed prior to the calibration that is used for routine analysis.

10.1.1 Prepare the required studies as indicated in Section 9.1.

10.1.2 Include these studies as part of the SOP development, verification and as directed in Section 9.1.

10.1.2.1 Changes in instrumentation or method procedures will require partial or full validation & IDC, prior to routine work.

10.1.3 Acceptable results will meet the criteria for each area as found in Section 9.1.

10.1.4 If the Validation & IDC is not performed as outlined, then routine analytical data are considered to be invalid.

10.2 Instrument Set-up and Tuning: Verify that the instruments are configured as indicated in Section 17. Routine maintenance is required for proper operation. There are some areas of the instrumentation that are known to drift, requiring re-tuning (detector settings or gas flows), cleaning or replacement. These areas are the focus of this segment. Use the instrument log to record and schedule maintenance.

10.2.1 Prepare the instruments by examining the following areas:

10.2.1.1 Fresh Internal Standard and Surrogate intermediate solution (within the autosampler) should be prepared.

10.2.1.2 Rinse water level should be full, prior to a run.

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10.2.1.3 Sample aliquot volume of 5 mL must be confirmed to be delivered by the autosampler. Measure this volume at the autosampler to sparge vessel intake line or reference the previously mark level line on the sparge tube. Cleaning may be necessary if the level is inaccurate. Replace the components when they are shown to be worn.

10.2.1.4 The purge vessel interior and the sample delivery needle must be examined for film or build up. Clean or replace them, as needed.

10.2.1.5 Concentrator flow is set to approximately 40 mL/min, with the system pressure at about 20 PSI.

10.2.1.6 Trap replacement is recommended when chromatography has changed or response has dropped, especially for late eluting compounds. Reference maintenance records and replace the trap as needed. Condition a fresh trap by purging it for 11+ minutes to remove any oxygen, do not desorb the trap into the GC, step it to bake for 2-12 hours at 260 ºC.

10.2.1.7 Transfer line replacement is periodically required. This is the heated line that the desorbed sample transfers through from the concentrator to the GC. Reference maintenance records and replace the line as needed.

10.2.1.8 Gas Chromatograph maintenance may be necessary. Reference maintenance records and Sections 10.3 for a chromatographic check, perform the maintenance as needed. Clean the injection port with a wire brush and then swab it with methanol. Replace the liner and O-ring. Cut 2-3 feet off of the column head. Replace the ferrule. The column will need to be replaced every one to three years.

10.2.1.9 Mass Spectrometer maintenance may be necessary. Reference maintenance records and Section 10.3, perform the maintenance as needed. Clean the source as instructed by the manufacturer. Sand the source components and scrub them with an abrasive methanol powder mix. Wash down the components with hot water to remove the residue. Solvent sonication rinses are required, as described in the instructions found in the maintenance kit. Components must be air dried before being installed.

10.2.1.10 Mass Spectrometer vacuum chamber integrity must be monitored. Perform a daily air and water check using the

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Enviroquant instrument control software. The relative abundances for water, nitrogen, oxygen and carbon dioxide must be below 5%. Read the vacuum level on the Ionization Gauge Controller. The vacuum must be greater than 1 x 10^{-5} TORR.

10.2.1.10.1 If the vacuum chamber integrity is found to be unacceptable there is most likely a leak present. Tighten the transfer line (GC oven to MS) nut or vent the system and replace the transfer line's ferrule and clean or replace the chamber's door seal.

10.2.1.11 Tune the Instrument: Perform a fresh detector tune by calibrating the mass and abundance scales of the mass spectrometer with calibration compounds and procedures prescribed by the manufacturer. A modified ATune program is used instead of the normal BFB tune. This procedure requires a manual adjustment to be made on the Ion Focus Lens after the completion of the automatic ATune.

10.2.1.11.1 Preparation is not needed. The Agilent 5973 uses perfluorotributylamine (PFTBA) as the tuning compound. A lifetime supply of PFTBA is contained within a vial that is attached on the detector housing. A small amount is automatically injected during a tune.

10.2.1.11.2 Include a tune as part of each calibration. After calibration a re-tune will be necessary when the sensitivity has dropped below the levels indicated by the Internal Standard or when a tune check fails.

10.2.1.11.3 Acceptable tunes will meet the ratio requirements of the BFB Tune Check, the procedure is found in Section 9.2.1 and the criteria in Table 2. The abundances of each mass ion must also be met.

10.2.1.11.4 If a tune results in an Electron Multiplier (EM) voltage near or at 3000 the source requires cleaning or the EM is in need of replacement.

10.2.2 Include the (above) instrument set-up steps with each calibration run and also with each daily run. Maintenance steps will not be a daily item, but

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they will be required if chromatography or response problems are found. Recalibration will be necessary after a source cleaning (or major maintenance).

10.2.3 Acceptable set-up will result in a stable calibration run and stable daily runs. Some criteria for the set-up are located in the preparation section.

10.3 Instrument Check: The instrument's performance needs to be examined prior to the calibration run. This involves chromatography quality, sensitivity and the verification of the detector's tune. Chromatographic and tune changes will influence the identification, the precision, and the accuracy of the analysis. A BFB tune check verifies that the associated data is collected with a detector that is measuring the proper masses in the proper ratios, as established during the initial tune. Follow the procedure as outline in Section 9.2.1.

10.3.1 Prepare and analyze a midlevel CVS/LCS (10-µg/L level).

10.3.2 Include this test prior to the calibration and continue to examine the integrity of the instrument during daily operations.

10.3.3 Acceptable peaks should be symmetrical with minimum tailing for most compounds. The data system must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make correct tentative identifications using the qualifiers (secondary ions).

10.3.4 Acceptable tune checks will meet the ratio requirements found in Table 2 of this SOP. Use the Autofind function to perform the tune check:

10.3.4.1 All criteria must pass. No failures are allowed.

10.3.5 If peaks are unusually broad, there is poor resolution between peaks, misidentification, sensitivity issues, failing tunes or inconsistent qualifiers, remedial action (maintenance) may be necessary. Refer to Section 10.2.1 for some of the possible maintenance action.

10.3.6 If the BFB tune check does not pass with Autofind the detector is out of calibration. Retune, rerun a test standard and perform another tune check.

10.3.7 If a detector retune does not correct the problem then the detector source or the GC may need to be cleaned. After a cleaning, run a tune check. The tune criteria must be met before proceeding with the calibration of analytes.

10.4 Calibration Set-up: When all performance criteria are met, prepare the appropriate number of Calibration Standards at the required concentrations using certified

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volumetric flasks, syringes, reagent water and standards.

10.4.1 Prepare Calibration Standards (CAL), QCS and Surrogate CAL as described in Section 7.

10.4.1.1 A fresh Intermediate Standard solution should be prepared for each calibration (Target Analyte), refer to Section 7.4. A fresh IS/Surr and QCS Intermediate Standard solution is not necessary but recommended. Expiration dates may not be exceeded for stock or Intermediate Standard solutions.

10.4.1.2 The CAL must be transferred into the same style of vial and be preserved in the same manner as the samples.

10.4.1.3 One CAL level must be at or below the Report Limit. The lowest level standard must be above the MDL to be a valid CAL level.

10.4.1.4 A minimum of five concentration levels must be used. Six levels will be required for a quadratic curve fit. A normal range for this analytical procedure is 0.5 µg/L to 50 µg/L, up to 200 µg/L for some compounds.

10.4.1.5 Calibration Standards define the working range. The working range is between (and including) the lowest and highest Calibration Standards, from which the value of unknown samples can be determined. Quantification is not allowed below the lowest standard or above the highest.

10.4.2 Include the CAL standard preparation as part of each calibration.

10.4.3 Acceptable Calibration Standards must be prepared as instructed in Section 7:

10.4.3.1 No more than 12 hours should pass from preparation to analysis.

10.5 Calibration Run: Purge and analyze each of the CAL levels using the procedure as outlined in Section 11.0. Evaluate the quality of the integration, response and identification of the analytes in each level.

10.5.1 Prepared CAL standards are loaded into the autosampler in a sequential manner, lowest to highest with blanks between the higher standards.

10.5.1.1 Surrogate CALs do not require blanks to be run between the standards. Surrogates are run as instructed in Section 7, by the autosampler.

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10.5.2 Include the Target Analyte CAL levels, the Surrogate CAL levels, a MB and a QCS with each calibration run that is performed. The Surrogate CAL should be run prior to the calibration for the Target Analyte while the QCS and the MB must be run after the calibration of the Target Analyte(s).

10.5.2.1 Calibration runs must be placed onto the autosampler and analyzed as soon as possible to minimize analyte degradation.

10.5.2.2 They are prepared on the day that they are run (analyzed within 12 hours of preparation) and they are run in a sequential manner.

10.5.2.3 A level or multiple levels of the calibration may not be prepared and run at a later date and then added to the previously built calibration.

10.5.2.4 Surrogate calibration may not be prepared and analyzed at a later date from the Target Analyte(s). If a Surrogate re-cal is necessary then a Target Analyte re-cal is also required.

10.5.2.5 The lowest CAL must produce an area that is responsive enough for the integrator and it must be above the background that is found in the Method Blank.

10.6 Curve Fit: Table 1 lists the quantitation ions for each compound, Internal Standard, Surrogate, and Target Analyte, as well as the Internal Standard that is used for each Target Analyte. The GC/MS data system software will calculate a Relative Response Factor (RF) for each calibration level of an analyte.

10.6.1 Prepare an average of response factors fit for each analyte, from the CAL levels in the calibration run.

10.6.1.1 Calculate the relative standard deviation (RSD) from the standard deviation of the RF (SD) and the mean of the RF (M): RSD = 100 (SD/M). The Agilent software will perform these calculations automatically when the Avg of Response Factors is chosen as the Curve Fit. Average of Response Factors uses the average of the Relative Response Factors for all calibration levels as the response/concentration ratio for quantification.

10.6.2 Include the Average fit as a starting point for establishing the calibration. This choice will determine the linearity of the response produced by the calibration levels as a whole working tool for calculating the concentration of unknowns.

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10.6.3 Acceptable Average fit RSD must be 15% or less. This indicates a calibration range that produces ratios that are relatively consistent. The concentration of the unknown samples should be determined by using the Average fit if the 15% rule is met.

10.6.3.1 Visually examine the curve plot for CAL level points that deviate unusually far from the line that is drawn. These levels are producing a response that is not linear or consistent with the other levels. This situation may not be acceptable. Refer to Section 10.7 for a test of this situation.

10.6.3.2 If the RSD of any analyte or Surrogate mean RF exceeds 15% there may be instrumental shortcomings. Analyze additional aliquots of appropriate CAL to obtain an acceptable RSD or to identify instrumental problems.

10.6.3.2.1 Take action to achieve the performance traditionally found by the procedure and instrumentation, maintenance may be required.

10.6.3.3 If a CAL level is found to produce non-linear responses, eliminate that level (if possible). Low and/or high concentration levels commonly have this problem. Re-examine the fit after removal. Removal of a level at the lowest or highest level will affect the working range of the instrument. One level must be at or below the RL.

10.6.4 If an Average of Response Factor fit is found to be unacceptable a second order regression (quadratic) calibration curve must be used. Use the data system to generate a curve. Curve fit is determined via the coefficient of determination (COD). The analyst may choose to weight the curve as needed. Six or more standard levels are required when a quadratic fit is used.

10.6.4.1 The correlation coefficient must be ≥ 0.99.

10.6.4.2 Visually examine the calibration curve for each compound. Curve plots that have an inversion, a plateau or other oddities are not in control. If CAL level points are found to deviate unusually far from the line that is drawn they are producing a response that is not linear or consistent with the other levels. This situation may not be acceptable. Refer to Section 10.7 for a test of this situation.

10.6.4.3 Forced through zero curve fits are not allowed.

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10.7 Verification of Calibration: This procedure tests the quality of the calibration, throughout the working range.

10.7.1 Prepared and analyzed Calibration Standards (CALs) are processed with the calibration method that they produced.

10.7.2 Include this procedure as part of each calibration. The processing must be performed after the calibration is complete, as a test of the whole process.

10.7.3 Acceptable results are a measurement of the calibration's accuracy.

10.7.3.1 Deviation must be ≤ 40% for RL and below RL levels and ≤ 30% for all other levels, when compared to the true value.

10.7.4 If results are found to be unacceptable there are several corrective options. Retry the calibration verification test after attempting any of the following options.

10.7.4.1 Prepare and analyze a new CAL at the level of the failing one. Replace the current data in the calibration table with the new CAL data.

10.7.4.2 Analyze more CAL levels around the problematic area to better define that zone with more curve data points. Update the calibration with these new levels.

10.7.4.3 Use a different curve fit or weight.

10.7.4.4 Remove levels. The low and/or high concentration levels commonly distort the curve. Removal will affect the working range of the instrument. Removal of other working range CAL levels should be avoided (those in the middle of the CAL range, not at the ends of the CAL range). If these levels must be removed there must be a comment in the calibration packet explaining the reason for this removal.

10.8 External Verification of Calibration: A CVS is prepared and analyzed to verify the accuracy of the initial calibration. This particular CVS is made from standards that have been obtained from a source (vendor/company) that is different from the source of the Calibration Standards. This particular CVS is known as a quality control sample (QCS).

10.8.1 Prepare a QCS as instructed and at the level specified in Section 7.

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10.8.2 Include a QCS as part of each calibration. The QCS is run after the curve and prior to any routine samples. If a calibration is used for more than 90 days (quarterly basis) a QCS must again be analyzed.

10.8.3 Acceptable QCS are a measurement of the instruments calibration and the calibration stock standard's accuracy. The following criterion must be met for each analyte:

10.8.3.1 Deviation must be ≤ 30% of true value.

10.9 Calibration Packet - Records: Instrument Control and data analysis methods must be saved for future reference (5 years).

10.10 Re-calibration: An established analytical procedure will require periodic calibration up-dating due to the wear of everyday use. This up-dating is known as the "re-calibration", to "re-calibrate" or simply as the "calibration".

10.10.1 Prepare and perform the re-calibration procedure as outlined in Section 10.2 through Section 10.8

10.10.2 Include this procedure as a correction for the following situations:

10.10.2.1 CVS/LCS acceptance criteria cannot be met.

10.10.2.2 Re-calibration is recommended quarterly (every 90 days).

10.10.2.3 Major maintenance such as cleaning the ion source, cleaning quadrupole rods or major component replacement requires a full recalibration.

10.10.3 Acceptable re-calibration will meet the criteria as listed in Sections 10.7 and 10.8.

11.0 PROCEDURE

11.1 Sample preparation:

11.1.1 Invert the vials to check for air bubbles. It is unacceptable to have an air bubble of 3 mm in diameter or larger trapped inside the vial.

11.1.1.1 If an air bubble is found, examine the other vials that were collected from that site (samples are collected in triplicate). Run the vial that has the smallest air bubble or no air bubble (if possible).

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11.1.1.2 If all of the vials from a site contain unacceptable air bubble/headspace, contact the Operations Unit and await further instructions prior to analysis. A photo of the vials may be requested by Operations, as an aide in the Client's decision on how to handle the samples.

11.1.1.3 When reporting results for samples that have been run with unacceptable bubbles, include a comment: Air bubble in vial; may have lost volatiles.

11.1.2 Inspect the vials for soil, sediment or sample discoloration. A ¼ inch or more of solids will interfere with the sample delivery system (autosampler).

11.1.2.1 If found to be unacceptable, use past experience as a guide, dilute the sample (2:1 or 5:1 for solids, as required for coloration). Do not mix the vial prior to performing the dilution.

11.1.3 Past results for the sample site(s) should be referenced if available. Carry-over from one sample to the next is common when analytes are found at a high concentration. Some samples may also be destructive to the instrumentation if they are not diluted prior to analysis.

11.1.3.1 Dilute the samples if sample history indicates high concentration or other factors that may affect the analysis, refer to Section 12.2.5 for dilution instructions.

11.2 Batch requirements:

11.2.1 Water Blank - This is made the same as an MB, but the data is not used. The water blank is run to prime the system and remove any contamination that may have settled during the time that the system was not being used.

11.2.2 Calibration Verification Standard - An initial CVS/LCS is analyzed in order to verify the instrument calibration that was established during the last calibration.

11.2.3 Matrix Spike and Matrix Spike Duplicate- One pair per 20 samples.

11.2.4 Report Limit Verification - A RLV is analyzed to verify that the instrument is capable of detecting the Target Analyte(s) at the RL.

11.2.5 Method Blank - Analysis of the MB will verify that the laboratory procedure is free of Target Analytes or other interferences that may give positive results that are not from the actual sample(s).

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11.2.6 Batch of Samples - A sequence of no more than 20 environmental samples. It is also required that there is at maximum an analysis injection time of 12 hours between the initial CVS/LCS and the final bracketing CVS/LCS. The 12 hour time restriction limits the number of samples to less than 20 in a batch (10 samples are typically run in a batch).

11.2.7 CVS/LCS - A final or bracketing CVS/LCS is analyzed in order to verify that the instrument is still in calibration, as verified during the run of the initial CVS/LCS and originally established during the last initial calibration run.

11.2.8 Next Analytical Batch - Load another MB, a batch of samples, a final CVS/LCS and continue this pattern as needed. A Water Blank and RLV are not required.

11.3 Autosampler:

11.3.1 A 5 mL aliquot of sample is delivered to the concentrator.

11.3.2 An aliquot of the Internal Standard/Surrogate mix is automatically introduced into the sample by the auto-sampler as it is delivered to the concentrator.

11.3.3 After sample delivery a start signal is sent from the autosampler to the concentrator.

11.3.4 A reagent water rinse of the sample loop is performed after the delivery of the sample to the concentrator.

11.3.5 A reagent water rinse of the sparge tube is applied to the purge vessel after the purging and removal of the sample.

11.4 Concentrator:

11.4.1 An 11 minute purge is performed, to extract the analytes.

11.4.2 During the purge the sample temperature is held at 45°C, to assist in the removal of the analytes.

11.4.3 A 2 minute dry purge is performed on the trap, without heating the trap, to remove water.

11.4.4 A trap preheat of 255°C with no gas flow prepares the trap for desorption.

11.4.5 A 260°C trap temperature is maintained for the 0.5 minutes as a flow of desorption gas (He) passes through the trap, removing the analytes.

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11.4.6 At the start of the desorption cycle, a start signal is sent from the concentrator to the gas chromatograph (GC) and mass spectrometer (MS). This is the beginning of the GC temperature program and the start of data acquisition.

11.4.7 260ºC trap and moisture control module temperature is maintained while the trap and vessel is flushed with desorption gas (this gas is vented). This is the bake-out cycle. It reconditions the concentrator for the next sample.

11.4.8 After 8 minutes the trap and Moisture Control System heaters are turned off. When the trap is cool, the next sample can be analyzed.

11.5 Gas Chromatograph (GC):

11.5.1 Sample analytes are introduced to the GC during the concentrators 0.5 minute desorb mode.

11.5.2 An injector split distributes 1 part of the desorb volume, from the concentrator, onto the analytical column and 50 parts out of the split vent. The split vent volume is waste. The purpose of this split mode is to regulate the amount of water that is sent over to the column.

11.5.3 A GC oven program begins at the start of the desorb mode (Data acquisition also begins at this point). The oven program regulates, holds and raises the column temperature in a manner that separates the analytes that were introduced onto the column.

11.5.4 A constant column carrier gas (He) flow rate of 1.2 mL/min is maintained during the temperature program.

11.5.5 After the 0.5 minute injector split an increase in the injector flow rate is used to flush out the injector. This extra flow is vented.

11.5.6 After the elution of the Target Analytes, the GC oven is ramped up to a temperature that is beyond what is necessary, 220ºC. This increase in temperature bakes out the column to prepare it for the next run.

11.5.7 At the completion of the oven ramp the MS data acquisition ends and the oven is cooled to the initial hold temperature of 40ºC. At this point the GC is ready for the next sample to desorb.

11.6 Mass Spectrometer (MS):

11.6.1 Full scan setting with a collection mass range of 35-300 AMU.

11.6.2 An electron multiplier boost of 306eV.

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11.6.3 The cycle time must be adjusted to measure five or more spectra during the elution of each peak.

11.7 Data acquisition:

11.7.1 Integrator RTE is used for this mass spectrometer work.

11.7.2 Quantify using initial Cal RFs or calibration curve, NOT using Continuing CAL RFs. This option is under Quant (in the Enviroquant Software).

11.7.3 Samples and QC components must be run under the same instrument control method and processed under the same calibration method (data analysis method). High and low curves are used for some analysis work, due to linearity issues. The curve used on samples must also be the curve used on QC.

11.7.4 A Summary report is printed out for each sample and all of the QC components are printed out and compiled into a QC packet. The reports and the packet must be saved (stored) and accessible for 5 years.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative Analysis: To identify analytes the GC/MS software uses the retention time of the quantitation ion, the detection of the qualifier (secondary) ions, a ratio comparison of these ions, and a spectral match.

12.1.1 Record a retention time for each single analyte and Surrogate to three decimal places (e.g., 0.007) using a mid-level CAL, that was used for calibration, as the reference.

12.1.2 Establish time windows should be 0.05 - 0.2 minutes from the assigned retention time. These windows are allowed to be set at widths that aid the software in identification and to prevent misidentification.

12.1.3 During daily operations the retention time of each analyte must be within +/- 0.06 minutes of the assigned retention time for that analyte. This rule applies to samples, QC samples, and standards.

12.1.4 The Internal Standard(s) RT may not deviate by more than 30 seconds from the mid-CAL time of the Internal Standard used for the most recent calibration. Re-calibration will be necessary if this condition exists. Column cutting may result in retention times that shift by this degree.

12.1.5 Reference spectra ratio values are updated during the calibration process, using a mid-level CAL for the update. The quantitation ion is assigned a value of 100% and the qualifying (secondary) ions are assigned a

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percentage of the quantitation ion's value. Qualifying ions are defined as the 3 ions of greatest intensity or any ions over 30% relative intensity. During the qualitative-identification editing process the relative intensity ratios of the characteristic (qualifying) ions should agree within +/- 20% of the relative intensities of those ion ratios found in the reference spectrum.

NOTE: Spectral ion ratios do not change with varying concentrations.

12.1.6 When performing a full spectral examination, in general, all ions that are present above 10% relative abundance in the library's mass spectrum should be present in the mass spectrum of the sample. A quality match above 75% (rated by the software) is considered to be an acceptable match, depending on the concentration. Lower concentration detects will have some ions missing and this will lower the match quality. Saturated detects will have some of the ions cut off short, this will also lower the match quality.

12.1.7 Complete chromatographic resolution is not necessary for accurate and precise identification of an analyte if unique ions with adequate intensities are available. When analytes co-elute the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound. These ions must not interfere with the quantitation ion or the qualifying ion(s). Results are suspect if there is interference.

12.1.8 Structural isomers that produce very similar spectra can be identified only if they have sufficiently different retention times. Acceptable resolution is achieved if the height of the valley between the two peaks is less than 25% of the sum of the two peak heights. If unresolved, these isomers must be reported as isomeric pairs.

12.1.9 Identification requires expert judgment when sample components are not resolved chromatographically and the produced mass spectra contains ions from more than one analyte; interfering Target Analytes overlapping or non-Target Analytes overlapping Target Analytes. Further identification of such sample components is achieved by a comparison of a background subtracted mass spectrum to a reference spectrum in the user-created database. Spectral ion ratios may be outside of the normal 20% range at such times.

12.1.10 Tentatively Identified Compounds (TICs) are required to be identified and quantified at the client's request. TICs are any significant peaks/compounds that are not among the list of target analytes. Significant peaks are those that are approximately 50% as high as the height of the closest Internal Standard. A Library Search Compound Report will

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identify the unknown by a full spectra library match and it will estimate the quantity by comparing the response of the unknown peak to the response produced by the Internal Standard that is closest to the unknown. Note these results in the final report using the add analyte function in Element.

12.2 Quantitative Analysis: To quantify the amount of identified analyte, the GC/MS software uses the Internal Standard form of calibration to compare the intensity (abundance) of the quantitation ion from the detect in the sample to the varied intensities of the same ion that was used during the multipoint calibration development for that analyte.

12.2.1 Abundance is measured by the area of the peak, not the height of the peak.

12.2.2 Quantification for each analyte is based on a measurement of a single mass ion. This ion is either the primary ion (most responsive) or the EPA recommended ion. If interference is found with the primary or recommended ion then the secondary ion or a unique ion may be used for quantification.

12.2.3 Integration should be performed automatically by the integrator that is part of the software; this will maintain the highest possible duplication quality.

12.2.3.1 Manual integration can be performed if necessary. It must be performed in a manner that replicates the integration performed during calibration.

12.2.3.2 The act of manual integration must be noted on the printed report (the Agilent software performs this task).

12.2.3.3 Refer to the Manual Integration SOP for further details: http://fyi.health.state.mn.us/phl/environmental/policies.html.

12.2.4 Internal Standard calibration is used for quantification:

12.2.4.1 The Internal Standards are added to all samples, blanks and standards at a constant amount, should not be present in the original test samples in interfering amounts, and should behave similarly to the Target Analyte(s).

12.2.4.2 Refer to Table 1 for a list of Target Analytes and the Internal Standard that is assigned to each analyte.

12.2.4.3 A 10 µL aliquot of intermediate Internal Standard/Surrogate solution is added by the Centurion Autosampler to each 5-mL

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blank, standard and sample that is analyzed. This results in a final concentration of approximately 10-µg/L for each.

12.2.5 Dilutions are performed to produce results that are within the quantification limit or to remove matrix interference.

12.2.5.1 Remove a fresh sample vial from the refrigerator and allow it to warm up to room temperature. Shake the sample vial well.

12.2.5.2 Use the table below as a dilution guide:

Dilution	***Dilution Factor***	***Flask Volume mL***	***Amount of Sample***
1:2	X2	50	25 mL
1:5	X5	50	10 mL
1:10	X10	50	5 mL
1:25	X25	100	4 mL
1:100	X100	100	1mL
1:1,000	X1,000	100	100 µL
1:10,000	X10,000	100	10 µL

12.2.5.3 A dilution is generally made in 50 mL or 100 mL flask. Select the appropriate flask and partially fill the flask with reagent water, allowing room for the sample aliquot.

12.2.5.4 Perform the dilution, using a micro syringe or a volumetric pipette to distribute the sample aliquot to the volumetric flask.

12.2.5.5 Fill the flask to the volume line with reagent water. Stopper the flask and slowly invert and right the flask three times to mix the reagent water with the sample aliquot.

12.2.5.6 With as little disturbance as possible, slowly pour the diluted sample to a VOC vial that contains the proper preservative. Immediately cap the vial and invert and right the vial three times to mix the preservative with the sample. The sample is now ready to analyze.

12.2.6 Diluted sample results (concentration without the multiplier) should be in the upper half of the calibration range. Results just above the RL are suspect of being too dilute and possibly inaccurate. Mid-level to upper level results are more appropriate and an indication of the correct dilution choice.

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12.2.7 The established RL is based on a non-diluted sample. Diluted sample results must have a RL that is adjusted (increased) to reflect the amount of dilution applied to the sample.

12.2.8 Accurate analysis dates are required when reporting analyte results that are from a dilution and added to (reported out with) the results from an initial undiluted run.

12.3 Reporting Rules: These rules apply for all of the routine data that is reported. The Client may request data to be reported in a manner that suits their needs for a specific project. Usually these specific needs involve less or more QC to be included in the final report.

12.3.1 Results are in µg/L.

12.3.2 The number of significant figures is two.

12.3.3 Preparation and analysis date are required.

12.3.4 Report only those values that fall between the lowest and highest Calibration Standards (within the working range).

12.3.5 Values above ½ of the RL and the RL are J flagged. The concentration is listed with a J flag. The explanation of the flag can be found on the last page of the report.

12.3.6 Values below ½ the RL or not detected are expressed as < the RL (RL is a concentration).

12.3.7 Dilutions will require an RL increase that reflects the diluted amount. Example: RL of 2 µg/L, diluted x10, new RL of 20 µg/L. A comment is also added for samples or single analytes that were diluted: Report Limit was changed due to sample dilution.

12.3.8 Diluted results must have the correct analysis date (it may be different then the initial or straight/non-diluted run).

12.3.9 Qualifiers must mark analytes that have (associated) QC that is unacceptable.

12.3.10 Data transfer, QC calculations, qualifiers and reporting functions must be handled by the LIMS system; Promium - Element.

12.3.11 Include a LIMS developed QC report that contains the holding time, MB results, LCS/LCSD and the MS/MSD recoveries and RPD calculations, and Field Duplicates RPD calculations.

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12.3.12 Tentatively Identified Compounds (TICs) must be listed as added analytes with concentration and the TIC qualifier attached (at the Client's request).

12.3.13 Preliminary reports must be reviewed by the Unit Supervisor or designee prior to transmittal to the client.

13.0 PERFORMANCE

13.1 Information pertinent to our laboratory's performance can be found in the MDH Environmental Laboratory Quality Assurance Manual, Section 9 and the Organics SOP (most recent edition org006).

14.0 POLLUTION PREVENTION

14.1 The Public Health Laboratory's pollution prevention mission is in the Organics SOP (most recent edition org006).

15.0 WASTE MANAGEMENT

15.1 The Public Health Laboratory's waste management guidelines are in the Organics SOP (most recent edition org006).

15.2 All purged water samples and water standards, from this automated procedure, can be disposed of through the laboratory's sanitary sewer. VOC vials, samples and standards, that contain water at a pH<2 are disposed of as bulk hazardous waste (in a 55 gallon drum). These VOC vials could be opened and dumped down our sanitary sewer system but we choose to refrain from excess handling and exposure by our staff. All solvent based standards are considered hazardous since they contain chlorinated compounds at fairly high concentrations (generally <2000 µg/mL). These hazardous standards are combined with the laboratory's chlorinated solvent waste. The chlorinated solvent waste and the VOC vials pH<2 are removed from the site and treated by an approved Hazardous Waste Specialist.

16.0 BIBLIOGRAPHY

16.1 USEPA Methods 8260B, 8000B, 5030B, 5000, Chapter 1, Chapter 2 and Chapter 4.

16.2 Appendix B to Part 136 - Definition and Procedure for the Determination of Method Detection Limit - Rev 1.1.1, Federal Register Vol. 49, No. 209 Oct 26,

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1984, pp. 198-204.

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17.0 TABLES, FLOWCHARTS, VALIDATION DATA

Table 1: Primary & Secondary Ions, Internal Standards, Report Limits (pg 1 of 3).

Internal Standards	Primary Ion	Secondary Ion(s)	Internal Standard#	Report Limit (µg/L)
Fluorobenzene (IS1)	96	77, 70	**1**	-
Chlorobenzene-d_5 (IS2)	117	54	**2**	-
1,4-Dichlorobenzene-d_4 (IS3)	152	115, 150	**3**	-
Surrogates				
Dibromofluoromethane (Surr 1)	113	190	1	-
1,2-Dichloroethane-d_4 (Surr 2)	102	104	1	-
Toluene-d_8 (Surr 3)	98	100	2	-
4-Bromofluorobenzene (Surr 4)	95	174, 176	3	-
1,2-Dichlorobenzene-d_4 (Surr 5)	152	115, 150	3	-
Target Analytes				
Acetone	58	43	1	20
Allyl Chloride	76	41, 39, 78	1	1.0
Benzene	78	77	1	1.0
Bromobenzene	156	77, 158	3	1.0
Bromochloromethane	128	49, 130	1	1.0
Bromodichloromethane	83	85, 127	1	1.0
Bromoform	173	175, 254, 252	3	1.0
Bromomethane	94	96	1	2.0
n-Butylbenzene	91	92, 134	3	1.0
sec-Butylbenzene	105	134	3	1.0
tert-Butylbenzene	119	91, 134	3	1.0
Carbon Tetrachloride	117	119	1	1.0
Chlorobenzene	112	77, 114	2	1.0
Chlorodibromomethane	129	208, 206, 127	2	1.0
Chloroethane	64	66	1	1.0
Chloroform	83	85	1	1.0
Chloromethane	50	52	1	1.0
2-Chlorotoluene	91	126	3	1.0

Table 1: Primary & Secondary Ions, Internal Standards, Report Limits (pg 2 of 3).

Target Analytes	Primary	Secondary	Internal	Report

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	Ion	Ion(s)	Standard#	Limit (µg/L)
4-Chlorotoluene	91	126	3	1.0
1,2-Dibromo-3-Chloropropane	75	155, 157	3	5.0
1,2-Dibromoethane	107	109, 188	2	1.0
Dibromomethane	93	95, 174	1	1.0
1,2-Dichlorobenzene	146	111, 148	3	1.0
1,3-Dichlorobenzene	146	111, 148	3	1.0
1,4-Dichlorobenzene	146	111, 148	3	1.0
Dichlorodifluoromethane	85	87, 101	1	1.0
1,1-Dichloroethane	63	65, 83	1	1.0
1,2-Dichloroethane	62	98	1	1.0
1,1-Dichloroethene	96	61, 63	1	1.0
cis-1,2 Dichloroethene	96	61, 98	1	1.0
trans-1,2-Dichloroethene	96	61, 98	1	1.0
Dichlorofluoromethane	67	69, 47	1	1.0
1,2-Dichloropropane	63	112, 76	1	1.0
1,3-Dichloropropane	76	78, 41	2	1.0
2,2-Dichloropropane	77	97	1	1.0
1,1-Dichloropropene	75	110, 77	1	1.0
cis-1,3-Dichloropropene	75	77, 39, 110	2	1.0
trans-1,3-Dichloropropene	75	77, 39, 110	2	1.0
Ethylbenzene	91	106	2	1.0
Ethyl Ether	74	45, 59, 73	1	1.0
Hexachlorobutadiene	225	223, 227, 260	3	1.0
Isopropylbenzene	105	120	3	1.0
p-Isopropyltoluene	119	134, 91	3	1.0
Methylene Chloride	84	86, 49	1	2.0
Methyl Ethyl Ketone	72	43, 57	1	10.0
Methyl Isobutyl Ketone	100	43, 58, 85	2	5.0
Methyl tert-Butyl Ether	73	57	1	2.0

Table 1: Primary & Secondary Ions, Internal Standards, Report Limits (pg 3 of 3).

Target Analytes	Primary Ion	Secondary Ion(s)	Internal Standard#	Report Limit (µg/L)
Naphthalene	128	127	3	1.0
n-Propylbenzene	91	120	3	1.0

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Styrene	104	78	2	1.0
1,1,1,2-Tetrachloroethane	131	133, 119	2	1.0
1,1,2,2-Tetrachloroethane	83	131, 85	3	1.0
Tetrachloroethene	164	129, 131, 166	2	1.0
Tetrahydrofuran	42	72, 71	1	10.0
Toluene	92	91	2	1.0
1,2,3-Trichlorobenzene	180	182, 145	3	1.0
1,2,4-Trichlorobenzene	180	182, 145	3	1.0
1,1,1-Trichloroethane	97	99, 61	1	1.0
1,1,2-Trichloroethane	83	97, 85	2	1.0
Trichloroethene	95	97, 130, 132	1	1.0
Trichlorofluoromethane	151	101, 153	1	1.0
1,2,3-Trichloropropane	75	77, 110	3	1.0
1,1,2-Trichlorotrifluoroethane	151	101, 85	1	1.0
1,2,4-Trimethylbenzene	105	120	3	1.0
1,3,5-Trimethylbenzene	105	120	3	1.0
Vinyl chloride	62	64	1	1.0
o-Xylene	106	91	2	1.0
p&m-Xylene	106	91	2	1.0

Table 2: Ion Abundance Criteria for 4-Bromofluorobenzene Tune.

Mass (m/z)	Relative Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95

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175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

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Table 3: Quality Control Acceptance Criteria (page 1 of 3).

Target Analytes	**MS %R**	**MS/ MSD RPD**	**LCS % R**	**LCS/ LCSD RPD**	**Method Blank**
Acetone	70-130%	<30%	70-130%	<30%	<RL
Allyl Chloride	70-130%	<30%	70-130%	<30%	<RL
Benzene	70-130%	<30%	70-130%	<30%	<RL
Bromobenzene	70-130%	<30%	70-130%	<30%	<RL
Bromochloromethane	70-130%	<30%	70-130%	<30%	<RL
Bromodichloromethane	70-130%	<30%	70-130%	<30%	<RL
Bromoform	70-130%	<30%	70-130%	<30%	<RL
Bromomethane	70-130%	<30%	70-130%	<30%	<RL
n-Butylbenzene	70-130%	<30%	70-130%	<30%	<RL
sec-Butylbenzene	70-130%	<30%	70-130%	<30%	<RL
tert-Butylbenzene	70-130%	<30%	70-130%	<30%	<RL
Carbon Tetrachloride	70-130%	<30%	70-130%	<30%	<RL
Chlorobenzene	70-130%	<30%	70-130%	<30%	<RL
Chlorodibromomethane	70-130%	<30%	70-130%	<30%	<RL
Chloroethane	70-130%	<30%	70-130%	<30%	<RL
Chloroform	70-130%	<30%	**80-120%**	<30%	<RL
Chloromethane	70-130%	<30%	70-130%	<30%	<RL
2-Chlorotoluene	70-130%	<30%	70-130%	<30%	<RL
4-Chlorotoluene	70-130%	<30%	70-130%	<30%	<RL
1,2-Dibromo-3-chloropropane (DBCP)	70-130%	<30%	70-130%	<30%	<RL
1,2-Dibromoethane (EDB)	70-130%	<30%	70-130%	<30%	<RL
Dibromomethane	70-130%	<30%	70-130%	<30%	<RL
1,2-Dichlorobenzene	70-130%	<30%	70-130%	<30%	<RL
1,3-Dichlorobenzene	70-130%	<30%	70-130%	<30%	<RL
1,4-Dichlorobenzene	70-130%	<30%	70-130%	<30%	<RL
Dichlorodifluoromethane	70-130%	<30%	70-130%	<30%	<RL
1,1-Dichloroethane	70-130%	<30%	70-130%	<30%	<RL
1,2-Dichloroethane	70-130%	<30%	70-130%	<30%	<RL
1,1-Dichloroethene	70-130%	<30%	**80-120%**	<30%	<RL
cis-1,2-Dichloroethene	70-130%	<30%	70-130%	<30%	<RL

Table 3: Quality Control Acceptance Criteria (page 2 of 3).

Target Analytes	**MS %R**	**MS/ MSD RPD**	**LCS % R**	**LCS/ LCSD RPD**	**Method Blank**

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trans-1,2-Dichloroethene	70-130%	<30%	70-130%	<30%	<RL
Dichlorofluoromethane	70-130%	<30%	70-130%	<30%	<RL
1,2-Dichloropropane	70-130%	<30%	**80-120%**	<30%	<RL
1,3-Dichloropropane	70-130%	<30%	70-130%	<30%	<RL
2,2-Dichloropropane	70-130%	<30%	70-130%	<30%	<RL
1,1-Dichloropropene	70-130%	<30%	70-130%	<30%	<RL
cis-1,3-Dichloropropene	70-130%	<30%	70-130%	<30%	<RL
trans-1,3-Dichloropropene	70-130%	<30%	70-130%	<30%	<RL
Ethylbenzene	70-130%	<30%	**80-120%**	<30%	<RL
Ethyl Ether	70-130%	<30%	70-130%	<30%	<RL
Hexachlorobutadiene	70-130%	<30%	70-130%	<30%	<RL
Isopropylbenzene	70-130%	<30%	70-130%	<30%	<RL
p-Isopropyltoluene	70-130%	<30%	70-130%	<30%	<RL
Methylene Chloride	70-130%	<30%	70-130%	<30%	<2XRL
Methyl Ethyl Ketone (MEK)	70-130%	<30%	70-130%	<30%	<RL
Methyl Isobutyl Ketone (MIBK)	70-130%	<30%	70-130%	<30%	<RL
Methyl tert-Butyl Ether (MTBE)	70-130%	<30%	70-130%	<30%	<RL
Naphthalene	70-130%	<30%	70-130%	<30%	<RL
n-Propylbenzene	70-130%	<30%	70-130%	<30%	<RL
Styrene	70-130%	<30%	70-130%	<30%	<RL
1,1,1,2-Tetrachloroethane	70-130%	<30%	70-130%	<30%	<RL
1,1,2,2-Tetrachloroethane	70-130%	<30%	70-130%	<30%	<RL
Tetrachloroethene	70-130%	<30%	70-130%	<30%	<RL
Tetrahydrofuran (THF)	70-130%	<30%	70-130%	<30%	<RL
Toluene	70-130%	<30%	**80-120%**	<30%	<RL
1,2,3-Trichlorobenzene	70-130%	<30%	70-130%	<30%	<RL
1,2,4-Trichlorobenzene	70-130%	<30%	70-130%	<30%	<RL
1,1,1-Trichloroethane	70-130%	<30%	70-130%	<30%	<RL
1,1,2-Trichloroethane	70-130%	<30%	70-130%	<30%	<RL
Trichloroethene (TCE)	70-130%	<30%	70-130%	<30%	<RL
Trichlorofluoromethane	70-130%	<30%	70-130%	<30%	<RL

Table 3: Quality Control Acceptance Criteria (page 3 of 3).

Target Analytes	**MS %R**	**MS/ MSD RPD**	**LCS % R**	**LCS/ LCSD RPD**	**Method Blank**
1,2,3-Trichloropropane	70-130%	<30%	70-130%	<30%	<RL
1,1,2-Trichlorotrifluoroethane	70-130%	<30%	70-130%	<30%	<RL
1,2,4-Trimethylbenzene	70-130%	<30%	70-130%	<30%	<RL

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1,3,5-Trimethylbenzene	70-130%	<30%	70-130%	<30%	<RL
Vinyl Chloride	70-130%	<30%	**80-120%**	<30%	<RL
o-Xylene	70-130%	<30%	70-130%	<30%	<RL
p&m-Xylene	70-130%	<30%	70-130%	<30%	<RL
Surrogates	%R		%R		%R
Dibromofluoromethane (Surr 1)	70-130%	NA	70-130%	NA	70-130%
1,2-Dichloroethane-d4 (Surr 2)	70-130%	NA	70-130%	NA	70-130%
Toluene-d8 (Surr 3)	70-130%	NA	70-130%	NA	70-130%
4-Bromofluorobenzene (Surr 4)	70-130%	NA	70-130%	NA	70-130%
1,2-Dichlorobenzene-d4 (Surr 5)	70-130%	NA	70-130%	NA	70-130%

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Table 4: Requirement Summary (page 1 of 2).

Parameter	MDH SOP 498 VOCs in Water EPA Method 8260B
Applicability	• Drinking water, surface water, waste water and ground water.
Analytes	• 68. • Report Limits are 1.0 µg/L for most, 5-20 µg/L for ketones, high Quantitation limit 50-200 µg/L. • Report Limit is found in Table 1.
Field Sample Amount Required	• 40 mL VOA vial in triplicate without headspace. • Glass container. • Teflon lined septa.
Preservation/Storage Conditions	• pH < 2 with 0.5 mL HCl. • Store at 4 +/-2°C.
Holding Time	• 14 days from collection.
Extraction Amount	• 5-mL.
Internal Standards	• Fluorobenzene, Chlorobenzene-d5 and 1,4-Dichlorobenzene-d4, each at 10-µg/L. • Absolute areas of Quantitation ions for Internal Standards may not deviate by more 50% - 100% from the response produced during initial calibration.
Surrogate Standard	• Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8, 4-Bromofluorobenzene and 1,2-Dichlorobenzene-d4, each at 10-µg/L. • 5-level curve prepared at 2.5, 5, 10, 15 and 25-µg/L. • %Recovery = 70-130; for all samples and related QC.
Standard Solution Expiration	• Store Intermediate Dilution at < -10°C for a maximum of 2 months.
BFB Tune	• Refer to Table 2. This is a modified ATune, as recommended by Agilent.
Initial Demonstration of Precision & Accuracy	• Analyze 4-7 replicate LCSs at a concentration of 10-µg/L. • Mean accuracy 20% of true value. • %Deviation from true ≤ 20%.
Detection Limit	• Analyze a minimum of 7 (recommend 12) low concentration LCSs, peaks yielding a 3-5 signal to noise response.

Table 4 Requirement Summary (Continued page 2 of 2).

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Initial Calibration	• Minimum of 5 levels, 9 levels are run at a range of 0.5 to 200-µg/L. • One level must be at or below the Report Limit. • High level represents the high Quantitation Limit. • RSD < 15% for linearity to be assumed, and to use Average RRF fit. • Linear Regression and Quadratic curve fits may be used, but COD ≥ 0.99 and the curve may not be forced through the origin.
Blanks	• Analyze 1 Method Blank every 12 hours. • Analytes must be below the Report Limit. • One Trip Blank per batch of field samples is recommended. • Field Blanks are a recommended practice.
BFB Tune Check	• Tuning: 25 ng Bromofluorobenzene (BFB) at the beginning of each 12 hour shift. • Tune Check performed on MB; tune check must pass in order to produce usable data.
QC Calibration Verification Standard	• Analyze 1 Report Limit Verification (1.0-µg/L) and then a mid-level CVS/LCS (10-µg/L). Analyze a second mid-level CVS/LCS after the samples to bracket the run, every 12 hours with a maximum of 10 samples & QC between CVS/LCS. • Deviation ≤ 40% for RLV and ≤ 30% for CVS/LCS.
Samples	• Absolute areas of Quantitation ions for Internal Standards may not deviate by more than 50% from the beginning CVS/LCS. • Surrogate %R = 70-130 for acceptance.
Accuracy/Precision	• One LCS/LCSD pair must be analyzed per analysis batch. CVS are the LCS for this analytical procedure (procedural standards). • One Matrix Spike/Matrix Spikes Duplicate pair per 20 samples. • LCS/LCSD % R 70-130, %R CCC 80-120%; RPD ≤ 30. MS/MSD 70-130; RPD ≤ 30. Refer to Table 3 for details.

Table 5: Autosampler Settings – EST Centurion:

Sample Type	Water
Sample Volume	5-mL
Standard Cycle	1
Sample Loop Fill	18 seconds

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Loop Equilibrium	5 seconds
Sample Transfer	18 seconds
Needle Rinse	18 seconds
Needle Sweep	18 seconds
Sample Loop Rinse	18 seconds
Sample Loop Sweep	18 seconds
Sample Drain	30 seconds
Sparge Rinse	18 seconds
Sparge Rinse Transfer	18 seconds
Conc 1 Cycle Timer (min)	0
Conc 2 Cycle Timer (min)	0
Rinse Cycles	3
Concentrators	Single Concentrator
Tray Configuration	Single Calibration Curve
Start Sequence With	Concentrator 1
Error Setup	Stop On No Vial: NO
Auto Correct Configure	Enable Auto Correct: NO
Internal Standard Vial	IS #1
Rinse Water Temperature	90ºC

Table 6: Concentrator Settings – EST Enchon:

Trap	VOCARB 3000
Moisture Control System	50ºC
Valve Temperature	140ºC
Transfer Line Temperature	140ºC
Purge	11 minutes (40 cc/minute helium)
Purge Temperature	45ºC (actual temp just above ambient)

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Dry Purge	2 minutes
Desorb Preheat	255ºC
Desorb	0.5 minutes at 260ºC
Bake Trap	8 minutes at 260ºC
Moisture Control System Bake	260ºC

Table 7: Gas Chromatograph Settings – Hewlett Packard 6890:

Carrier Gas	Helium
Total Flow	43.5-mL/minute
Injector Temperature	200ºC
MS Transfer Line Temp	150ºC
Column Head Pressure	23.9 psi @ 40ºC
Column Flow	Constant flow of 1.2-mL/minute & 49-cm/second linear velocity
Split Ratio	50:1
Liner	Restek PT#21111 SPME 0.75 mm ID, deactivated.
Column	Restek Rxi-624Sil MS, 20 meter, 0.18 mmID, 1.0 um df
Initial Temperature	40ºC, hold 2.0 minutes
Rate	12ºC/minute to 175ºC
Rate	30ºC/minute to 220ºC, hold 2.0 minutes
Final Temperature	220ºC
Run Time	16.75 minutes

Table 8: Mass Spectrometer – Agilent 5973:

Scan Setting	Full Scan
Scan Range	35-300 AMU
Scan Rate	2.78 scan/second
Electron Ionization	70 eV
MS Source Temperature	230°C
MS Quad Temperature	150°C
EM Boost	306 eV initial
Threshold	700

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Signature Manifest

Document Number: DOC-458 **Revision:** 0

Title: VOCs in Water by Purge and Trap GC/MS

All dates and times are in Central Time.

Organic Unit Document-Inital Upload

Review

Name/Signature	Title	Date	Meaning/Reason
Stephanie Drier (DRIERS1)	Organic Chemistry Supervisor	03 Feb 2016, 11:49:40 AM	Approved

Final Approval

Name/Signature	Title	Date	Meaning/Reason
Paul Moyer (MOYERP1)	PUBLIC HEALTH LAB MGR	12 Apr 2016, 09:48:51 AM	Approved

Review: DOC-458 0

Review

Name/Signature	Title	Date	Meaning/Reason
Katie Rinker (RINKEK1)	ENVIRONMENTAL ANALYST 2	28 Feb 2017, 01:11:39 PM	Reviewed - Under Revision

Review: DOC-458 0 VOCs in Water by Purge and Trap GC/MS

Review

Name/Signature	Title	Date	Meaning/Reason
Betsy Edhlund (EDHLUB1)	RESEARCH SCIENTIST 2	04 Jun 2018, 10:46:30 AM	Reviewed

http://fyi.health.state.mn.us/phl/environmental/organics/index.html

REVISION NUMBER	AUTHOR /REVISOR	REVISION DATE	DESCRIPTION OF CHANGE
A	Martin Bevan, Andrew Mittendorff	02-14-2008	• Initial Release
1	Andrew Mittendorff	08-08-2011	• Updated MDL • Lowered RL and curve range • Updated secondary standard solution preparations • Added mass-labeled PFPeA as internal standard • Updated internal standard preparations • Nine-point calibration curve • QCS acceptable range 30% • Calibration curve point acceptable range 25%, lowest point 30% • Updated the daily run requirements to include QCS • Updated bibliography • Updated Table 1, analyte structures and formula, PFHxS and PFOS are now obtained as sodium salts instead of potassium salts • Updated Tables to reflect most recent MDL/IDC, standard solution preparation, instrument and method parameters • Updated signature page
2	Andrew Mittendorff	08-03-2013	• Updated MDLs • Updated sample hold time • Updated standards tables • Updated MS/MS parameters

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			• Added concentration units to tables • Corrected collision gas to Argon • Stated MDL frequency • Added Corrective Action statement • Added Organics SOP revision number • Added dilution rules and table • Added reference to manual integration • Clarified secondary fragment ion transition and ion ratio conditions • Updated standards tables

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PROCEDURE FOR THE DETERMINATION OF:

PERFLUORINATED CHEMICALS IN WATER SAMPLES BY HPLC-MS/MS

PFC – 555

Table of Contents

1.0 SCOPE AND APPLICATION

1.1 This method provides analytical procedures for the determination of seven perfluorinated chemicals in drinking water and clean monitoring well water using HPLC-MS/MS.

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1.2 This method is applicable for the determination of perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluorooctanoic acid (PFOA), perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHxS) and perfluorooctane sulfonate (PFOS), as listed below, in clean water matrix only. The analyte structures are listed in Table 1.

Method	Analyte	Formula	CAS	MDL (ppb)	RL (ppb)
555	Perfluorobutanic acid	$C_4HF_7O_2$	375-22-4	0.004	0.05
555	Perfluoropentanoic acid	$C_5HF_9O_2$	2706-90-3	0.003	0.05
555	Perfluorohexanoic acid	$C_6HF_{11}O_2$	307-24-4	0.004	0.05
555	Perfluorooctanoic acid	$C_8HF_{15}O_2$	335-67-1	0.004	0.05
555	Perfluorobutane sulfonate	$C_4F_9SO_3^-$	29420-49-3	0.006	0.05
555	Perfluorohexane sulfonate	$C_6F_{13}SO_3^-$	3871-99-6	0.003	0.05
555	Perfluorooctane sulfonate	$C_8F_{17}SO_3^-$	2795-39-3	0.004	0.05

1.3 The working range is 0.01 to 10 ng/mL. Dilutions are prepared for concentrations greater than 10 ng/mL.

1.4 Perfluorochemicals (PFCs) are a class of compounds characterized by a fluorinated alkyl chain and a polar head group. There are two classes of PFCs included in this method, the perfluorinated carboxylates: PFBA, PFPeA, PFHxA, PFOA and the perfluorinated sulfonates: PFBS, PFHxS, and PFOS.

1.5 While the PFCs are persistent and bioaccumulative, their toxicity is not well understood. They are ubiquitous in environmental and human samples; they have been detected in biota from remote as well as urban locations, and are well distributed in environmental samples at all latitudes.

2.0 SUMMARY OF METHOD

2.1 This method is a quantitative analysis for PFCs in water using high performance liquid chromatography and tandem mass spectrometry (HPLC-MS/MS) with negative electrospray ionization. Aqueous samples are diluted with acetonitrile (3:1 sample:ACN by volume), and then injected onto the

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liquid chromatography mass spectrometry system where the analytes of interest are separated, and identified by retention times, molecular ions, primary and secondary fragment ions, and primary/secondary fragment ion ratio, and then quantitated by comparing with an internal standard and plotted against calibration curves.

3.0 DEFINITIONS

3.1 Definitions that are common to all areas of the laboratory appear in Section 2.0 of the QA Manual and in Section 3.0 of the Organics SOP (most recent edition org006).

3.2 Definitions that are applicable only to this SOP:

3.2.1 Instrument Blank (IB): A blank sample that is run with each set of samples and at the end of the analytical run. Prepared from solvents, the IB indicates carryover, contamination or other changes in the instrument occurring during the course of the analytical run. At the beginning of the analytical run, the IB can be used to demonstrate that the system is free of contaminants at the start of the process.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in reagent water, solvents, reagents, glassware, columns, HPLC tubing, and other sample processing apparatus that can lead to discrete artifacts, elevated baselines or that may otherwise bias analyte response. All of these materials must be shown to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems.

4.2 Teflon® containing materials (e.g. caps, liners, wash bottles) contain fluoro-compounds which may cause interferences, and should not be used during collection, storage, extraction, or analysis of the samples.

4.3 Contamination may occur due to carryover from samples with high concentrations of compounds. If carryover is suspected an IB should be analyzed after a high level sample, high calibration standard, or QC sample to ensure that carryover has not occurred.

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4.4 Glassware must be scrupulously cleaned. Wash with hot water and detergent and rinse with tap water, followed by reagent water. Loosely cap washed laboratory glassware with aluminum foil and heat in a muffle furnace at greater than 400^{o}C for 4 hours to reduce background interferences or rinse with the appropriate solvent before use. Volumetric pipets, disposable transfer pipets and autosampler vials are not heated or rinsed.

4.5 One sample collection bottle per lot is tested and verified to be free of contamination before sending out the bottles for sample collection. Trip Blanks are recommended for this method and they are provided as requested to the client.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of reagents and chemicals used in this SOP has not been fully established. Each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable.

5.2 Analysts who work in the lab are required to read the following MDH safety policies located in the MDH Policy and Procedure Manual:

POLICY #	TITLE
902.02	Occupational Safety and Health
420.01	Right-to-Know

In addition, the analyst should read the MDH Public Health Laboratory Division – Chemical Hygiene Plan (http://fyi.health.state.mn.us/phl/safety/index.html). Questions regarding the Chemical Hygiene Plan should be referred to the Laboratory Health and Safety Officer.

5.3 The analyst should read the Lab Building Emergency Procedures plan (http://fyi.health.state.mn.us/phl/safety/index.html) and know what to do in a variety of emergency situations.

5.4 Safety glasses should be worn by all analysts at all times while in the laboratory area. Visitors are given temporary safety glasses while in the laboratory. Lab coats and other protective clothing should be worn by analysts when appropriate.

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5.5 The analyst may contact the Employee Hazard Hotline regarding employee exposures to hazardous chemicals (1-888-673-7466 Toll Free). The system is available 24 hours per day, seven days per week.

5.6 The following chemicals have the potential to be toxic or hazardous: perfluorobutanoic acid, perfluoropentanoic acid, perfluorohexanoic acid, perfluorooctanoic acid, perfluorobutane sulfonate, perfluorohexane sulfonate, perfluorooctane sulfonate, acetonitrile, methanol, and formic acid. Consult the applicable MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Any substitutions for the equipment and supplies listed below must meet or exceed the listed product's specifications.

6.2 Balance – Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.3 Glassware - All glassware must be borosilicate. Volumetric flasks and pipettes are Class A.

6.4 Multitube Vortexer: Model DVX-2500 - VWR.

6.5 Micro Centrifuge: Model 5417R - Eppendorf.

6.6 Nitrogen Gas (desolvation) – 99.9% pure.

6.7 Argon Gas (collision) – 99.9% pure.

6.8 Pipettes: Manual adjustable volume, 1000 uL, 100 uL and 10 µL – Rainin; Repeater Plus - Eppendorf

6.9 Sample vials: Polypropylene 0.8 mL HPLC vials - National Scientific, or equivalent.

6.10 Sample bottles: Nalgene HDPE 250 ml wide mouth and 12 mL and 8 mL narrow mouth - Nalge Nunc International (Rochester, NY).

6.11 HPLC-MS/MS system: Refer to Table 8 for detailed parameters.

6.11.1 High Performance Liquid Chromatography - Agilent 1100 LC system includes a Binary Pump, Vacuum Degasser, Thermostat Autosampler, Thermostatted Column Compartment and a Control Module (Wilmington, DE) or equivalent. The guard and analytical HPLC

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columns are respectively; Thermo Betasil C8 3.0 x 30 mm, 5 um and Thermo Betasil C8, 2.1 x 50 mm, 3 um or equivalent.

6.11.2 Tandem Mass Spectrometer - Quattro Micro (QAA 048), a triple quadrupole mass spectrometer manufactured by Waters (Beverly, Massachusetts) or equivalent.

6.11.3 Data System - MassLynx Software version 4.1 or higher running with Windows XP, version 2002 platform or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 All standard solutions are prepared to volume using volumetric flasks and transferred to HDPE bottles for storage in a refrigerator (2 ^{o}C to 6^{o}C). All preparation and storage containers are Teflon®-free. Disposable containers have been shown to be PFC-free and reusable glassware is washed and heated in a muffle furnace prior to use.

7.2 HPLC grade acetonitrile, methanol, and reagent water. Reagent water is ASTM Type I or equivalent with a resistivity of > 18 megaohm (mΩ)/cm at 25^{o}C, free of the analytes of interest or any interfering compounds greater than 1/2 the report level.

7.3 Formic acid – reagent grade from Sigma or equivalent.

7.4 Mobile Phase A: 0.1% formic acid in water. Add 1 mL of formic acid to 1000 mL reagent water and mix well.

7.5 Mobile Phase B: 0.1% formic acid in acetonitrile. Add 1 mL of formic acid to 1000 mL reagent water and mix well.

7.6 Wash Solution: Injector needle is washed for 10 seconds prior to sample injection by a 75:25 methanol:water solution.

7.7 Parent Standard Solutions: High purity source material for each analyte should be purchased from Wellington Labs or equivalent. Standards that are 100% linear are preferred but not required. All structural isomers present for an analyte are integrated and treated as a sum total. For the perfluoroalkyl sulfonate analytes, a salt-correction must be applied to determine the free anion concentration. Vendor certificates of authenticity (COA) and characterization spectra for each lot of material are saved and filed.

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Certificates of Analysis are verified upon receipt to determine the presence of any structural isomers or other PFCs. See Table 4 for details.

7.8 Internal Standards (IS): Isotopically labeled source solutions are commercially available from Wellington Labs for PFBA, PFPeA, PFHxA, PFOA, PFHxS, and PFOS. Sample purity is verified upon receipt to determine the presence of any structural isomers, other PFCs, or unlabeled (native) analytes. The amount of residual native material must be less than 1.0% for each analyte. As with the calibration standards, all structural isomers present for an analyte are integrated and treated as a sum total. Vendor certificates of authenticity (COA) and characterization spectra for each lot of material are saved and filed. See Table 4 for details.

7.9 Secondary Standard Solutions: These solutions are made by diluting the Parent Standard Solutions in high grade acetonitrile and used for preparing calibration, quality control and sample standards. See Table 5 for details.

7.9.1 Mix A 250: A mixture of all analytes at 250 ppb each is prepared by combining and diluting the purchased source materials. This mixture is used in the preparation of calibration standards and sample standards.

7.9.2 Mix A 5: A 1:50 dilution of Mix-A-250. This mixture is used in the preparation of low level calibration standards.

7.9.3 Mix B 250: A mixture of all analytes at 250 ppb each is prepared by combining and diluting the purchased source materials. If available, this solution should come from different source material lots than those used in preparing Mix-A-250. This mixture is used in the preparation of quality control samples.

7.9.4 IS 250 Mix: A mixture of all labeled IS analytes at 250 ppb is prepared by combining and diluting the purchased source IS materials. This mixture is used in the preparation of calibration standards and sample standards.

7.10 Sample Standards: These solutions are made by diluting the Secondary Standard Solutions in high grade acetonitrile and are used in unknown samples. See Table 6 for details.

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7.10.1 PFC - IS: This solution is made by diluting IS 250 Mix to a nominal concentration of 1 ppb using high purity acetonitrile. It is used for adding IS to each unknown sample and method blank.

7.10.2 PFC – IS/Spk: This solution is made by diluting IS 250 Mix and Mix A 250 to nominal concentrations of 1 ppb for the internal standards and 0.5 ppb for the native analytes using high purity acetonitrile. It is used in the preparation of matrix spikes and LCSs.

7.11 Quality Control Standard: These solutions are made by diluting the Secondary Standard Solutions in high grade acetonitrile and are used in preparing quality control samples.

7.11.1 PFC – QCS: This solution is made by diluting IS 250 Mix and Mix B 250 to nominal concentrations of 1 ppb for the internal standards and 0.5 ppb for the native analytes using high purity acetonitrile. It is used in the preparation of the QCS. See Table 7 for details.

7.12 Calibration Standards: A nine-point calibration curve is prepared in acetonitrile by diluting corresponding amounts of secondary standard solutions and IS 250 Mix in 10 mL volumetric flasks. All calibration standards are made such that 50 ul of the standard diluted into 150 ul of water gives the desired concentration of analytes and IS. See Table 7 for details.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Water samples are collected using 250 mL Nalgene bottles. Bottles must contain at least 100 mL of sample for analysis.

8.2 Sample will be stored in a refrigerator (2 ^{o}C -6 ^{o}C) for a maximum of 14 days before sample analysis.

8.3 Before sample collection, Nalgene bottle lots are tested to be free of contamination of any of the analytes.

8.4 Trip blank filled with reagent water in the laboratory may be sent out for sample collection for every 20 unknown samples. This is not required, but recommended particularly if characterizing a new sampling location.

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9.0 QUALITY CONTROL

9.1 Corrective Action: When it has been determined that a corrective action should be initiated follow the procedures in the Corrective Action SOP (most recent edition qao011).

9.2 Initial Demonstration of Capability (IDC): The analyst must be able to demonstrate that they can generate acceptable accuracy and precision data with this SOP by successful completion of the following:

9.2.1 Initial Calibration: The calibration range must be determined initially and whenever a significant change in instrument response is observed. The initial demonstration of linearity uses a calibration blank and at least 8 different calibration standards. One of the standards is near, but above the MDL. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion. The standards must bracket the range of concentrations found in samples and should define the working range of the instrument.

9.2.2 External Verification of Calibration: When available a quality control sample (QCS) from an external source is analyzed. The results of the QCS must be within ± 30 % of the established QCS value, otherwise remedial action is taken and the entire Initial Demonstration of Capability is repeated.

9.2.3 Method Detection Limit (MDL) Study: A minimum of 7 replicate laboratory fortified blanks (LFB) are spiked at a value 1 to 5 times the estimated detection limit. The MDL is determined using the procedure in 40 CFR, Part 136, Appendix B. MDL's must be low enough for regulatory/client purposes, otherwise remedial action is taken and the process is repeated. The MDL must be lower than the report level to be acceptable. The validity of the MDL should be verified by the detection (a value above zero) of a QC sample at no more than 4X the MDL. An MDL study is performed annually to establish new detection limits.

9.2.4 Initial Precision and Accuracy: To establish the ability to generate results with acceptable accuracy and precision, analyze four replicates

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of a mid-range standard. Calculate the mean concentration and the standard deviation for the data set. The percent recovery of the mean must be between 80% and 120%, while the percent relative standard deviation (%RSD) must be less than 20%. Both conditions need to be satisfied before sample analysis can begin.

9.2.5 Demonstration of Low Background: Analyze at least one Laboratory Reagent Blank (LRB) to determine reagent or laboratory contamination. The LRB result must meet the criteria established for the on-going demonstration of low background in Section 9.3.1.

9.2.6 Other Requirements for an IDC: An IDC may also be required if there are significant changes to the SOP, matrix, or instrument that could affect the precision, accuracy or sensitivity of the analysis. Consult with the Quality Assurance Officer (QAO) to determine if any changes require an IDC.

9.3 Ongoing Quality Controls: With each analysis batch (20 samples) the following criteria must be met:

9.3.1 Method Blank (MB): The MB background from method analytes and contaminants that interfere with method analytes must be less than 1/2 the RL. If method analytes are detected in the MB at concentrations equal to or greater than this level, then the affected samples must be reprocessed. If the contamination cannot be eliminated, then the results must be qualified.

9.3.2 Report Level Verification (RLV): A procedure that determines whether the established report level is valid for a target analyte within an analysis and/or analytical run. This procedure is performed by the analysis of a standard at or below the report level. For further details, see the “Policy and Procedure for Report Level Verification” in the QA Manual. For an acceptable analysis, the % recovery for all analytes shall be within 70% to 130%.

9.3.3 Laboratory Control Sample/ Calibration Verification Standard (LCS/CVS): An aliquot of reagent water known to be free of interfering amounts of target analytes or other interferences, to which known quantities of the target analytes are added in the laboratory. It is

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prepared and analyzed exactly like a sample. Its purpose is to verify that the procedure is in control and that the laboratory is capable of making accurate measurements. For an acceptable analysis, the percent recovery for all analytes shall be within 80% to 120%.

9.3.4 Matrix Spike (MS): Each unknown sample shall be spiked with a known concentration of all analytes. The calculated concentration of the spiked sample shall be within ±30% of the theoretical value; failure to meet this criterion indicates significant matrix interference, then that particular sample should be diluted and reanalyzed. It should be noted that since this method is a dilution method the spiked analytes are primarily testing for suppression and enhancement of the target ions within the triple quadrupole mass spectrometer.

9.3.5 Duplicate precision: In every 20 field samples, one pair of sample duplicates or matrix spike duplicates is processed with each batch. Duplicate samples or duplicate spikes (whichever is processed) must have a relative percent deviation (RPD) within +/- 20%. If the average of the duplicates is less than five times the reporting limit, the difference between the duplicates must be less than the report limit.

9.3.6 IS Area Count: The internal standard area count is monitored for every sample. The analytes PFBA, PFPeA, PFHxA, PFOA, PFHxS, and PFOS all utilize a matching isotopically labeled internal standard to account for any variations (including matrix effects such as ion suppression or enhancement). The analyte PFBS does not have a matched internal standard. As a guidance the area counts of the IS in the samples should be within a factor of two (ie. 50% to 200%) of the average area counts of each IS from the initial calibration standards. If these criteria are not met for **matching** internal standard/native compounds the data is accepted if the matrix spike recoveries for the respective sample are acceptable. If these criteria are not met for **nonmatching** internal standard/native compounds (PFBS) the data for the offending samples are diluted and the analyses are repeated. If the dilution corrects the area count failure the data is reported. If dilution does not correct the area count failure the data is qualified and the most undiluted sample is reported. If the area count is high for the IS

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(> 200%) but field samples show no detection for that analyte, then a result of "less than" may be reported without reanalysis.

9.3.7 If any of the criteria above are not met, correct the problem before further samples are analyzed. Rerun any samples analyzed between the last LCS that met the criteria and those that have fallen out. If this is not possible, qualify that data.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare a series of nine calibration standards and a calibration blank by diluting 50 ul of each calibration standard solution into 150 ul of reagent water, as described in Section 7.10. The lowest concentration of calibration standard must be at or below the RL.

10.2 A new calibration curve must be generated every 24 hours.

10.3 Prior to acquiring a new calibration curve, the analyst must verify the HPLC-MS/MS system stability by injecting several replicates of blanks and samples. The system is deemed stable if the analyte retention times and area counts are consistent with previously established values.

10.4 Use the MS data system software (Mass Lynx 4.1) to generate a linear regression or quadratic calibration curve using the internal standard method. The analyst is free to force the curve through zero if this best fits the data.

10.5 Acceptance criteria for the calibration of method analytes is determined by calculating the concentration of analytes according to the calibration curve. Calibration curve points must calculate to be within 25% of the true value (except the lowest value can be within 30% of the true value). The correlation coefficient (r^2) for the calibration curve must be $\geq$0.990. High or low points may be deactivated to achieve these criteria, but an acceptable curve must contain at least six active curve points. If the above criteria are not met, reanalyze the calibration samples or select an alternate calibration method.

10.6 After the calibration has been established, it must be followed by an IB, a RLV, QCS, LCS and MB prior to the analysis of samples. Subsequent batches within a 24 hour period must include an IB, a LCS and a MB. Every batch (20 samples) must include a matrix spike for each sample and a duplicate sample and/or duplicate matrix spike.

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10.6.1 For an acceptable analysis, the verification standards must meet the requirements described in Section 9.3.

10.6.2 If analytical results do not meet the above criteria, the analysis is terminated, the instrument is checked, and then re-calibrated. All samples following the last passing quality control are reanalyzed. The following exception is allowed. If a LCS/CVS failed high (recoveries were >120%) but field samples show no detection for that analyte(s), then a result of "less than" may be reported without reanalysis. The data would not be qualified unless requested by the client.

11.0 PROCEDURE

11.1 Remove samples from the cooler, shake, and place in warm water bath to come to room temperature.

11.2 Label all necessary 800 uL polypropylene HPLC vials – one set of calibration standards, an IB, a MB, a RLV, a QCS, a LCS/LCSD pair, and two vials for every field sample. For the field sample vials, designate one of the two vials as the "spike" and the other as the "sample".

11.3 Add 50 ul of each calibration standard to 150 ul of reagent water for each calibration standard.

11.4 Add 50 uL of PFC - IS to 150 uL of reagent water for the MB.

11.5 Add 50 uL of report level calibration standard to 150 uL of reagent water for RLV.

11.6 Add 50 uL of PFC - IS/Spk to 150 uL of reagent water for the LCS.

11.7 Add 50 uL of acetonitrile to 150 uL of reagent water for the IB.

11.8 Add 50 uL of PFC – QCS to 150 uL of reagent water for the QCS.

11.9 Add 50 uL of PFC - IS into each "sample" vial for the field samples. Add 50 uL of PFC - IS/Spk into each "spike" vial for the field samples. Aliquot 150 uL of field sample into each of the "sample" and "spike" vials.

11.10 For all standards and samples, the final solution shall be in 3:1 water:acetonitrile. Cap the vials and vortex at 4000 rpm for 4 min using a multi-vortexer.

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12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative analysis: All blanks, standards, and samples must be analyzed on an HPLC-MS/MS system in an identical manner using the same instrument settings and injection parameters (as listed in Table 8). The qualitative identification of compounds determined by this method is based on retention time, molecular ion, primary fragment ion transition, secondary fragment ion transition, and the ion ratio of the primary fragment ion and the secondary fragment ion.

12.1.1 Retention time: After separation on a liquid chromatography system, each analyte has a specific retention time. In field samples, the retention time of each analyte should match the retention time of the respective analyte in calibration standards (within ± 0.2 min of deviation).

12.1.2 Molecular ion: In the electrospray ionization source of the mass spectrometer, the analytes are ionized in negative mode, generating molecular ions which are ions with mass to charge ratios (*m/z*) of $[M-1]^{-1}$. The mass to charge ratio of the molecular ion is specific to the molecular mass of the analyte and the charge on the ion; under the electrospray negative conditions employed with this method, the charge is almost always 1. During analysis, the molecular ion is selected in the first quadrupole section of the HPLC-MS/MS instrument.

12.1.3 Primary fragment ion transition: After the molecular ion of the analyte is selected in the first quadrupole, it passes into a collision cell, where argon gas is used to fragment the ion. The fragmentation pattern of each analyte is unique, and usually the most abundant fragment ion (primary fragment ion) is chosen to be monitored in the second quadrupole. This molecular ion to primary fragment ion transition is used to differentiate the analyte from other compounds with identical retention time and molecular ion (or molecular weight). In field samples, each analyte must have the same primary fragmentation ion transition as in the calibration standards in order to be qualitatively identified. The primary fragment ion is also used for quantitation.

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12.1.4 Secondary fragment ion transition: In addition to the primary fragment ion, another fragment ion (the secondary fragment ion) may be formed in the collision cell and monitored with the second quadrupole of the HPLC-MS/MS. This secondary fragment ion is also unique to the compound, so it differentiates the analyte from other compounds and also serves as an additional confirmation of its identity. In field samples, each analyte must have the same secondary fragment ion transition as in the calibration standards.

12.1.5 Primary/secondary fragment ion ratio: The primary/secondary fragment ion ratio is characteristic to each analyte so it may be used to support confirmation. However, because several of the PFC analytes have multiple structural isomers (where ratios may differ between isomers) and isomers are integrated as a sum total, the primary/secondary fragment ion ratio is only utilized as a qualitative guidance tool.

Secondary fragment detection limits: PFBA and PFPeA do not have suitable secondary fragment ions, therefore, the retention time and the primary fragment ion and not the secondary fragment ion are used in the identification of PFBA and PFPeA. All other analytes must demonstrate the presence of a secondary fragment ion to be reportable without qualification when reported at or above the report limit. If the secondary fragment ion is not present the result must be qualified on the report. Below the report limit the secondary fragment ion may not always be observed due to weak signal and compound identification is then based on retention time and the primary fragment ion. Typical detection limits and expected ion ratio are as follows:

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Analyte	Detection Limit (ug/L)	Ratio
PFBA	**-NA-**	**-NA-**
PFPeA	**-NA-**	**-NA-**
PFHxA	**> 0.1**	18 ± 4
PFOA	**> 0.025**	3.0 ± 1
PFBS	**> 0.01**	3.4 ± 1
PFHxS	**> 0.01**	3.0 ± 1
PFOS	**> 0.01**	2.3 ± 1

12.2 Quantitative Analysis: To quantify the amount of identified analyte, the MS software uses the internal standard calibration method to compare the response of the primary fragment ion in the sample to the same ion used during calibration.

12.2.1 Abundance is measured by the area of the peak.

12.2.2 Integration should be performed automatically by the software integrator following the same parameters used for calibration.

12.2.3 Manual integration is performed if necessary in cases where the software incorrectly integrates the baseline or incorrectly identifies the correct peak. Manual integrations follow the most recent revision of the Manual Integration of Chromatographic Data SOP ops018.

12.2.4 Both the automatic and manual integrations are saved for review.

12.2.5 Dilutions are performed to produce results that are within the quantification limits or to remove matrix interference.

12.2.5.1 Dilutions are performed serially, using one dilution level as the source for the next until the correct dilution factor is reached. A measured amount of sample (or diluted sample from the previous level) is added to Nanopure water in a 2mL vial. Use the table below as a dilution guide.

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Dilution factor	Amount of sample (mL)	Final volume (mL)
5x	0.3	1.5
20x	0.4	1.6
100x	0.3	1.5

12.3 Reporting Rules: These rules apply for all routine data that is reported. Clients may request data to be reported in a manner that suits their needs for a specific project.

12.3.1 Results are in ug/L.

12.3.2 The number of significant figures is two.

12.3.3 Preparation and analysis date are required.

12.3.4 Values below the report level are qualified with a "J" flag.

12.3.5 Qualifiers must mark analytes that have associated QC which is unacceptable.

12.3.6 Diluted results require a report level increase that reflects the dilution factor.

12.3.7 Data transfer, QC calculations, qualifiers and reporting functions are handled by the LIMS system; Promium – Element.

12.3.8 Results reports are reviewed by Unit Supervisor or designee according to established procedure prior to transmittal to client.

12.4 Initial Demonstration of Capability (IDC):

12.4.1 Mean Accuracy: Percent Recovery

%Recovery = (Mean Sample Conc./ Theoretical Sample Conc.) * 100

Mean: $X_m = (\sum_{i=1}^{n} (Xi)) / n$

Where: X_m = Mean of X concentrations

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Xi = individual observed or calculated concentrations
n = number of observation

12.4.2 Sample Conc. = IS Conc. * [(Sample Area /IS Area) - Intercept] /Slope

12.4.3 Precision: Relative Standard Deviation (RSD)

$$\%RSD = (SD/ X_m)*100$$

Where: SD = Standard Deviation
X_m = Mean of X concentrations

12.4.4 Method Detection Limit (MDL):

$$MDL = S(t_{(n-1)})$$

Where: S = Standard deviation of the seven replicates
t = Students t value at the 99% confidence level
n = Number of replicates

12.4.5 Standard Deviation (SD):

$$SD = \sqrt{\left(\sum_{i=1}^{n} (Xi - X_m)^2\right) / (n-1)}$$

Where: X_m = Mean of X concentrations
Xi = individual observed or calculated concentrations
n = number of observation

12.5 Ongoing Demonstration of Quality Control:

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12.5.1 Accuracy: Percent Recovery (%R) – Percent recovery is used as an accuracy check for the demonstration of ongoing quality control for LCS and RLV:

%R = (Observed Sample Conc./ Theoretical Sample Conc.) * 100

12.5.2 Accuracy: Sample Spike Recovery (%SR)

%SR = ((Spike Conc. – Sample Conc.)/Theoretical Spike Conc.)*100

12.5.3 Precision: Relative Percent Difference (RPD) for duplicates.

% RPD = |((A – C)/((A + C)/2))|*100

Where: A = measured concentration for the initial sample or spike.
B = measured concentration for the sample or spike duplicate.

13.0 PERFORMANCE

13.1 Summary data from the method validation (precision, accuracy, method detection limits) are listed in section 17.0.

14.0 POLLUTION PREVENTION

14.1 For information regarding the laboratory's pollution prevention policy and procedures, see the current version of the Public Health Laboratory Division Hazardous Waste Manual. http://fyi.health.state.mn.us/phl/safety/index.html

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life, space available for storage, and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratory operations, consult, "Less is Better: Laboratory Chemical

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Management to Waste Reduction" available from the American Chemical Society, Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C., 20036.

15.0 WASTE MANAGEMENT

15.1 The Public Health Laboratory, in carrying out its mission, will do so in such a manner as to minimize pollution of the environment and manage its hazardous wastes in a safe and environmentally sound manner.

The Public Health Laboratory Division shall:

- Conserve natural resources through reduction, reclamation, recycling.
- Ensure that the Division meets all Federal, State, and Local regulations pertaining to hazardous waste disposal.
- Prevent pollution at the source whenever possible.
- Consider environmental impact when purchasing materials, handling chemicals and disposing of waste.
- Promote awareness and provide training opportunities for pollution prevention and hazardous waste management within the Division.
- Define the responsibilities of managers, supervisors and staff so that Division activities will be conducted appropriately and effectively with regard to waste management.
- Develop policies and procedures as needed to further these objectives.

15.2 Acetonitrile and formic acid waste is disposed of in accordance with our laboratory's chemical waste disposal guidelines.

15.3 PFC containing waste is disposed of by a certified hazardous waste contractor.

15.4 For additional information regarding the laboratory's waste management policy, see the current version of the Public Health Laboratory Division Hazardous Waste Manual. http://fyi.health.state.mn.us/phl/safety/index.html

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16.0 BIBLIOGRAPHY

16.1 "Quantification of Perfluorooctane Sulfonate and Perfluorooctanoic Acid in Water Samples by HPLC-MS/MS"; Yongyi Julia Jiang; Minnesota Department of Health, Method 553.

16.2 "Perfluorooctanoate and Perfluorooctane Sulfonate Concentrations in Surface Water in Japan"; Norimitsu Saito, Kouji Harada, Kayoko Inoue, Kazuaki Sasaki, Takeo Yoshinaga and Akio Kouzumi; *Journal of Occupational Health 2004;46:49-59.*

16.3 "A Pilot Study on the Determination of Perfluorooctanesulfonate and other Perfluorinated Compounds in Blood of Canadians"; Cariton Kubwabo, Natalia Vais and Frank M. Benoit; *J. Environ. Monit., 2004; 6:540-545.*

16.4 "Perfluorooactanesulfonate and Other Fluorochemicals in the Serum of American Red Cross Adult Blood Donors"; Geary W. Olsen, Timothy R.Churth, John P. Miller, Jean M. Burris, Kristen J. Hansen, James K. Lundberg, John B. Armitage, Ross M. Herron, Zahra Medhdizadehkashi, John B. Nobiletti, E. Mary O'Neill, Jeffray H. Mandel and Larry R. Zobel; *Environmental Health Perspectives 2003; 111:1892-1901.*

16.5 "A Survey of Perfluorooctane Sulfonate and Related Perfluorinated Organic Compounds in Water, Fish, Birds, and Humans from Japan"; Sachi Taniyasu, Kurunthachalam Kannan, Yuichi Horii, Nobuyasu Hanari and Nobuyoshi Yamashita; *Environmental Science and Technology 2003; 37: 2634-2639.*

16.6 "Quantitative Characterization of Trace Levels of PFOS and PFOA in the Tennessee River"; K. J. Hanson, H. O. Johnson, J. S. Eldridge, J. L. Butenhoff and L. A. Dick; *Environmental Science and Technology 2002; 36:1681-1685.*

16.7 "Appendix B to Part 136 – Definition and Procedure for the Determination of Method Detection Limit – Revision 1.11", *Federal Register 10/26/1984, Vol. 49, No. 209, 198-204.*

16.8 "Guidance for Perfluorochemicals Analysis", (Minnesota Pollution Control Agency), May 2010.

16.9 "Perfluorinated Chemicals in Drinking Water and Nonpotable Water Samples by HPLC/MS-MS, MDH Assessment Checklist", 6/25/2010.

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17.0 TABLES, FIGURES, VALIDATION DATA

TABLE 1 – STRUCTURE OF ANALYTES

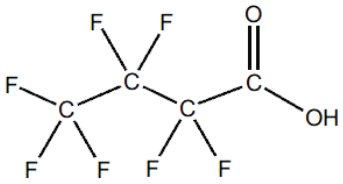

Figure 1. Perfluorobutanoic acid (PFBA)
Formula: $C_4HF_7O_2$
Formula Weight: 214.04

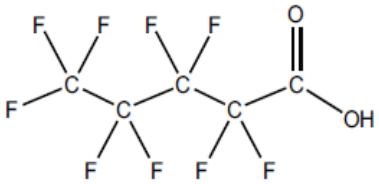

Figure 2. Perfluoropentanoic acid (PFPeA)
Formula: $C_5HF_9O_2$
Formula Weight: 264.04

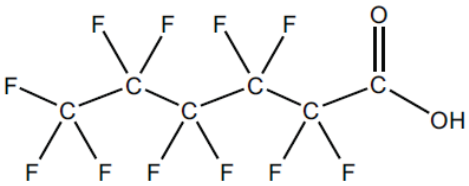

Figure 3. Perfluorohexanoic acid (PFHxA)
Formula: $C_6HF_{11}O_2$
Formula Weight: 314.05

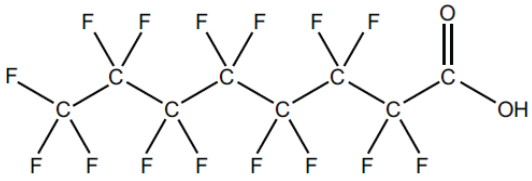

Figure 4. Perfluorooctanoic acid (PFOA)
Formula: $C_8HF_{15}O_2$
Formula Weight: 414.06

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Figure 5. Perfluorobutane sulfonate, potassium salt (PFBS)
Formula: $C_4F_9O_3S^-K^+$
Formula Weight: 338.19

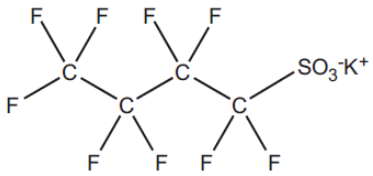

Figure 6. Perfluorohexane sulfonate, sodium salt (PFHxS)
Formula: $C_6F_{13}O_3S^-Na^+$
Formula Weight: 422.10

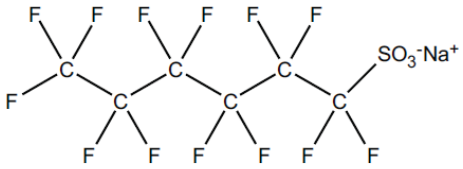

Figure 7. Perfluorooctane sulfonate, sodium salt (PFOS)
Formula: $C_8F_{17}O_3S^-Na^+$
Formula Weight: 522.11

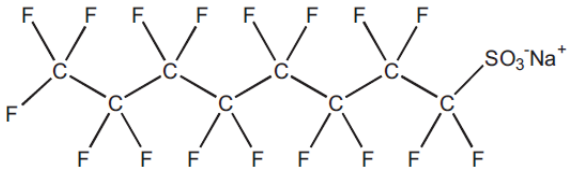

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TABLE 2 – METHOD DETECTION LIMITS

Analysis Date: 8/20/2012, 8/31/2012										Reported Units: ug/L		
Analyte	Rep. #1 8/20/12	Rep. #2 8/20/12	Rep. #3 8/20/12	Rep. #4 8/20/12	Rep. #5 8/31/12	Rep. #6 8/31/12	Rep. #7 8/31/12	Mean	Std. Dev.	% Rec.	True Value	MDL
PFBA	0.0239	0.0238	0.0250	0.0214	0.0231	0.0234	0.0241	0.0235	0.001	94%	0.025	0.004
PFPeA	0.0253	0.0238	0.0253	0.0240	0.0243	0.0240	0.0268	0.0248	0.001	99%	0.025	0.003
PFHxA	0.0252	0.0238	0.0246	0.0241	0.0228	0.0241	0.0218	0.0238	0.001	95%	0.025	0.004
PFOA	0.0227	0.0242	0.0238	0.0227	0.0206	0.0234	0.0233	0.0230	0.001	92%	0.025	0.004
PFBS	0.0201	0.0217	0.0212	0.0208	0.0251	0.0224	0.0244	0.0222	0.002	89%	0.025	0.006
PFHxS	0.0261	0.0256	0.0259	0.0264	0.0240	0.0245	0.0264	0.0256	0.001	102%	0.025	0.003
PFOS	0.0239	0.0255	0.0265	0.0271	0.0264	0.0239	0.0256	0.0256	0.001	102%	0.025	0.004

TABLE 3 – INITIAL DEMONSTRATION OF CAPABILITY: ACCURACY AND PRECISON

	Analysis Date: 3/3/2009						Reported Units: ug/L					
Analyte	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Mean	Std. Dev.	True Value	% Recovery	% RSD
PFBA	2.7021	2.7913	2.7369	2.8172	2.7538	2.7740	2.8334	2.7727	0.046	2.5	111%	1.7%
PFPeA	2.2737	2.3607	2.3684	2.3114	2.2749	2.2689	2.3374	2.3137	0.043	2.5	93%	1.8%
PFHxA	2.4602	2.5691	2.6110	2.6086	2.5750	2.5296	2.6435	2.5710	0.061	2.5	103%	2.4%
PFOA	2.7359	2.6663	2.5788	2.7846	2.7747	2.6513	2.6301	2.6888	0.078	2.5	108%	2.9%
PFBS	2.3957	2.4368	2.4516	2.3800	2.3359	2.3629	2.4914	2.4078	0.055	2.5	96%	2.3%
PFHxS	2.3809	2.3466	2.4059	2.3273	2.4088	2.4048	2.3786	2.3790	0.032	2.5	95%	1.3%
PFOS	2.6063	2.5413	2.5451	2.4084	2.4776	2.5367	2.5234	2.5198	0.062	2.5	101%	2.5%

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TABLE 4 – PARENT STANDARDS (WELLINGTON) current as of 6/17/13

A side - Calibration Standards and Matrix Spikes

Element #	Compound	CAS #	Date Received	Lot #	MW	Salt MW	Salt Ratio	Purity	Linear Isomer	Conc ppm (ug/mL)
2C01002	Perfluorobutyric acid	375-22-4	2/1/2012	PFBA0111	214.04	na	1.000	>98	100.0%	50.0 +/- 2.5
2C01011	Perfluoropentanoic acid	2706-90-3	2/1/2012	PFPeA1111	264.05	na	1.000	>98	100.0%	50.0 +/- 2.5
2C01012	Perfluorohexanoic acid	307-24-4	2/1/2012	PFHxA1011	314.05	na	1.000	>98	100.0%	50.0 +/- 2.5
2C01013	Perfluorooctanoic acid	335-67-1	2/1/2012	PFOA0711	414.07	na	1.000	>98	~98.5%	50.0 +/- 2.5
2C01014	K^+ Perfluorobutane sulfonate	29420-49-3	2/1/2012	LPFBS1111	299.09	338.19	1.131	>98	100.0%	44.2 +/- 2.2
2C01015	Na^+ Perfluorohexane sulfonate	82382-12-5	2/1/2012	LPFHxS1011	399.11	422.10	1.058	>98	100.0%	47.3 +/- 2.4
2C01016	Na^+ Perfluorooctane sulfonate	4021-47-0	2/1/2012	LPFOS0511	499.12	522.11	1.046	>98	100.0%	47.8 +/- 2.4

B side - Quality Control

1B07017	Perfluorobutyric acid	375-22-4	5/1/2010	PFBA1209	214.04	na	1.000	>98	100.0%	50.0 +/- 2.5
1B07018	Perfluoropentanoic acid	2706-90-3	5/1/2010	PFPeA1209	264.05	na	1.000	>98	100.0%	50.0 +/- 2.5
1B07019	Perfluorohexanoic acid	307-24-4	5/1/2010	PFHxA1209	314.05	na	1.000	>98	100.0%	50.0 +/- 2.5
1B07020	Perfluorooctanoic acid	335-67-1	5/1/2010	PFOA0410	414.07	na	1.000	>98	~98.5%	50.0 +/- 2.5
1B07021	K^+ Perfluorobutane sulfonate	29420-49-3	5/1/2010	LPFBS1209	299.09	338.19	1.131	>98	100.0%	44.2 +/- 2.2
1B07022	Na^+ Perfluorohexane sulfonate	na	5/1/2010	LPFHxS0210	399.11	422.10	1.058	>98	100.0%	47.3 +/- 2.4
1B07023	Na^+ Perfluorooctane sulfonate	2795-39-3	5/1/2010	LPFOS0310	499.12	522.11	1.046	>98	100.0%	47.8 +/- 2.4

ISTD Solutions

Element #	Compound	Lot	Conc.(ppm)
1B07024	13C4-PFBA	MPFBA1209	50
1B07026	13C5-PFPeA	M5PFPeA0810	50
1B07025	13-C4-PFHxA	MPFHxA0210	50
1B07027	13C4-PFOA	MPFOA0110	50
1B07028	16O2-PFHxS	MPFHxS0210	50
1B07029	13C4-PFOS	MPFOS1209	50

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TABLE 5 – SECONDARY STANDARD SOLUTIONS current as of 6/17/13

Primary Dilution Standard	Parent Element #	Parent Lot #	Parent Conc ppm (mg/L)	Final Conc ppb (µg/L)	Final Vol (mL)	Aliquot Vol (ul)
2C14008 Mix A 250	2C01002	PFBA0111	50.0	250	25	125.0
	2C01011	PFPeA1111	50.0	250	25	125.0
	2C01012	PFHxA1011	50.0	250	25	125.0
	2C01013	PFOA0711	50.0	250	25	125.0
	2C01014	LPFBS1111	44.2	250	25	141.4
	2C01015	LPFHxS1011	47.3	250	25	132.1
	2C01016	LPFOS0511	47.8	250	25	130.8
2C14009 Mix A 5	2C14008	Mix A 250	250	5	10	200.0
2C14010 Mix B 250	1B07017	PFBA1209	50.0	250	10	50.0
	1B07018	PFPeA1209	50.0	250	10	50.0
	1B07019	PFHxA1209	50.0	250	10	50.0
	1B07020	PFOA0410	50.0	250	10	50.0
	1B07021	LPFBS1209	44.2	250	10	56.6
	1B07022	LPFHxS0210	47.3	250	10	52.9
	1B07023	LPFOS0310	47.8	250	10	52.3
1L05013 IS 250 Mix	1B07024	MPFBA1209	50.0	250	10	50.0
	1B07026	M5PFPeA0810	50.0	250	10	50.0
	1B07025	MPFHxA0210	50.0	250	10	50.0
	1B07027	MPFOA0110	50.0	250	10	50.0
	1B07028	MPFHxS0210	50.0	250	10	50.0
	1B07029	MPFOS1209	50.0	250	10	50.0

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http://fyi.health.state.mn.us/phl/environmental/organics/index.html

TABLE 6 – SAMPLE STANDARDS current as of 6/17/13

	Date Prepared:	06/05/13						
Element #	Sample Standard	Parent Solution	Parent Conc. (ppb)	Aliquot (mL)	Final Vol. (mL)	Final Conc. (ppb)	Inst Conc. (ppb)	Nominal conc. (ppb)
3F05045	PFC - IS	1L05013 IS 250 Mix	250	0.300	25	3	**0.75**	**1**
		ACN		Dil to Vol				
3F05044	PFC - IS/Spk	1L05013 IS 250 Mix	250	0.300	25	3	**0.75**	**1**
		2C14008 Mix A 250	250	0.150	25	1.5	**0.375**	**0.5**
		ACN		Dil to Vol				

TABLE 7 – CALIBRATION AND QUALITY CONTROL STANDARDS current as of 6/17/13

Date Prepared:	01/11/13								IS Mix used:	1L05013 IS 250 Mix		
Element #	Calibration Standard	Final Vol. (mL)	Source Solution	Source Conc. (ppb)	Aliquot Vol. (mL)	Final Conc. (ppb)	Inst Conc. (ppb)	Nominal conc. (ppb)	IS Source Conc. (ppb)	IS Aliquot Vol. (ul)	Final IS Conc. (ppb)	Nominal IS conc. (ppb)
3A15016	Std B - 10	10	2C14008 Mix A 250	250	1.200	30.0	**7.5**	**10**	250	120	3.00	**1**
3A15017	Std C - 5	10		250	0.600	15.0	**3.75**	**5**	250	120	3.00	**1**
3A15018	Std D - 1.0	10		250	0.120	3.00	**0.75**	**1**	250	120	3.00	**1**
3A15019	Std E - 0.5	10		250	0.060	1.500	**0.375**	**0.5**	250	120	3.00	**1**
3A15020	Std F - 0.25	10		250	0.030	0.750	**0.1875**	**0.25**	250	120	3.00	**1**
3A15021	Std G - 0.10	10	2C14009 Mix A 5	5	0.600	0.300	**0.075**	**0.1**	250	120	3.00	**1**
3A15022	Std H - 0.05	10		5	0.300	0.150	**0.0375**	**0.05**	250	120	3.00	**1**
3A15023	Std I - 0.025	10		5	0.150	0.075	**0.01875**	**0.025**	250	120	3.00	**1**
3A15024	Std J - 0.010	10		5	0.060	0.030	**0.0075**	**0.01**	250	120	3.00	**1**

Element #	QC Standard	Final Vol. (mL)	Source Solution	Source Conc. (ppb)	Aliquot Vol. (mL)	Final Conc. (ppb)	Inst Conc. (ppb)	Nominal conc. (ppb)	IS Source Conc. (ppb)	IS Aliquot Vol. (ul)	IS Mix Conc. (ppb)	Nominal conc. (ppb)
3A15025	PFC - QCS	10	2C14010 Mix B 250	250	0.060	1.50	**0.375**	**0.5**	250	120	3.00	**1**

This procedure has been prepared for the sole use of the Minnesota Department of Health (MDH) Environmental Laboratory and may not be specifically applicable to the activities of other organizations.

Note: Unless viewed online via the designated PHL Environmental Laboratory area of the MDH intranet, the user must verify the accuracy of any printed or electronic convenience copies by comparing the revision number and content with the controlled document.

TABLE 8 - INSTRUMENT ANALYSIS USING HPLC-MS/MS current as of 6/17/13

MS/MS Source Parameters:

Source	Set
Polarity	ES-
Capillary (kv)	0.40
Cone (v)	19
Extractor (v)	1
RF Lens (v)	0.2
Source Temperature (^{o}C)	120
Desolvation Temperature (^{o}C)	350
Desolvation Gas Flow (L/hr)	700
Cone Gas Flow (L/hr)	0

MS/MS Analyzer Parmeters:

Analyzer	Set
LM1 Resolution	10.0
HM1 Resolution	10.0
Ion Energy 1	1.0
Entrance	-5
Collision	15
Exit	1
LM2 Resolution	13.0
HM2 Resolution	13.0
Ion Energy 2	1.5
Multiplier (v)	750
Gas Cell Pirami Pressure (mbar)	About 3.0 e-3

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MS/MS Mass Collection Parameters:

MS Function	Time (min)	Analyte	Ion Transition	Type	Dwell (Sec)	Cone (V)	Collision Energy (eV)
Func 1	0.6 - 1.3	PFBA	212.9 > 168.9	Primary	0.200	18	9
		MPFBA	216.9 > 172.1	Primary	0.100	15	10
Func 2	1.3 - 2.3	PFPeA	262.8 > 219.0	Primary	0.200	16	9
		MPFPeA	267.8 > 223.1	Primary	0.200	15	9
Func 3	2.3 - 3.2	PFBS	298.7 > 79.8	Primary	0.100	45	29
			298.7 > 98.8	Secondary	0.050	45	29
		PFHxA	312.8 > 269.0	Primary	0.100	15	9
			312.8 > 118.9	Secondary	0.050	15	21
		MPFHxA	314.6 > 270.0	Primary	0.100	15	10
Func 4	3.1 - 3.9	PFHxS	398.6 > 79.8	Primary	0.100	50	35
			398.6 > 98.8	Secondary	0.050	50	30
		MPFHxS	402.6 > 83.8	Primary	0.100	55	35
		PFOA	412.6 > 369.0	Primary	0.100	18	10
			412.6 > 169.0	Secondary	0.050	18	18
		MPFOA	416.7 > 371.9	Primary	0.100	15	11
Func 5	3.8 - 4.5	PFOS	498.5 > 79.8	Primary	0.100	60	45
			498.5 > 98.9	Secondary	0.100	60	40
		MPFOS	502.5 > 79.9	Primary	0.100	60	40

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HPLC General Conditions:

Autosampler Temperature	28 ^{o}C
Analytical Column	Thermo Betasil C8, 50 x 2.1 mm, 3 µm with Upchurch PEEK 0.5 µm prefilter
Guard Column	Thermo Betasil C8 3.0 x 30 mm, 5 um
Column Temperature	30 ^{o}C
Sample Temperature	5 ^{o}C
Injection Volume	10 µL
Mobile Phase A	0.1% formic acid in water
Mobile Phase B	0.1% formic acid in acetonitrile
Run Time	6.75 mins

HPLC Gradient Elution Parameters:

Time (min)	**% A**	**% B**	**Flow Rate (mL/min)**
0.0	70	30	0.4
0.25	55	45	0.4
3.75	10	90	0.4
4.5	10	90	0.6
4.75	10	90	0.6
4.76	70	30	0.6
5.75	70	30	0.6
6.25	70	30	0.4

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http://fyi.health.state.mn.us/phl/environmental/organics/index.html

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Written By:___/s/Martin Bevan/Andrew Mittendorff__ Date: 6/17/2013

Approved By:__/S/Paul Swedenborg_____ Date: 9/16/13

Paul Swedenborg, Organic Chemistry Unit Leader

Approved By:___/S/Paul Moyer_____ Date: 9/16/13

Paul Moyer, Environmental Laboratory Section Manager

This procedure has been prepared for the sole use of the Minnesota Department of Health (MDH) Environmental Laboratory and may not be specifically applicable to the activities of other organizations.

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Appendix C

Laboratories Certifications
(Pace – Minneapolis, MN,
and Minnesota Department of Health – Saint Paul, MN)
MDH Accreditations)

Minnesota Department of Health
Environmental Laboratory Accreditation Program

Issues accreditation to

State Laboratory ID: 027-053-137

EPA Lab Code: MN00064

Pace Analytical Services, LLC - Minneapolis MN
1700 Elm Street SE
Minneapolis, MN 55414-2485

for fields of accreditation listed on the laboratory's accompanying Scope of Certification in accordance with the provisions in Minnesota Laws and Rules.

Continued accreditation is contingent upon successful on-going compliance with Minnesota Statutes 144.97 to 144.98, 2009 TNI Standard and applicable Minnesota Rules 4740.2010 to 4740.2120. The laboratory's Scope of Certification cites the specific programs, methods, analytes and matrices for which MDH issues this accreditation.

This certificate is valid proof of accreditation only when associated with its accompanying Scope of Certification.

The Scope of Certification and reports of on-site assessments are on file at the Minnesota Department of Health, 601 Robert Street North, Saint Paul, Minnesota. Customers may verify the laboratory's accreditation status in Minnesota by contacting MNELAP at (651) 201-5324.

Effective Date: 09/27/2018
Expires: 12/31/2018
Certificate Number: 1468244

Issued under the authority delegated by the Commissioner of Health, State of Minnesota

Environmental Laboratory Accreditation Program

Scope of Certification

THIS LISTING OF FIELDS OF ACCREDITATION MUST BE ACCOMPANIED BY CERTIFICATE NUMBER: 1468244

State Laboratory ID: 027-053-137 **EPA Lab Code: MN00064** **Issue Date: 9/27/2018**
Expiration Date: 12/31/2018

Pace Analytical Services, LLC - Minneapolis MN
1700 Elm Street SE
Minneapolis, MN 55414-2485

Clean Air Act

EPA TO-10A (GC/ECD)

Preparation Techniques: Extraction, soxhlet;

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-10A (GC/ECD)	Aroclor-1016 (PCB-1016)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1221 (PCB-1221)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1232 (PCB-1232)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1242 (PCB-1242)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1248 (PCB-1248)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1254 (PCB-1254)	AIR	MN	
CAA	EPA TO-10A (GC/ECD)	Aroclor-1260 (PCB-1260)	AIR	MN	

EPA TO-4A

Preparation Techniques: Extraction, soxhlet;

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-4A	Aroclor-1016 (PCB-1016)	AIR	MN	
CAA	EPA TO-4A	Aroclor-1221 (PCB-1221)	AIR	MN	
CAA	EPA TO-4A	Aroclor-1232 (PCB-1232)	AIR	MN	
CAA	EPA TO-4A	Aroclor-1242 (PCB-1242)	AIR	MN	
CAA	EPA TO-4A	Aroclor-1248 (PCB-1248)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-4A	Aroclor-1254 (PCB-1254)	AIR	MN	
CAA	EPA TO-4A	Aroclor-1260 (PCB-1260)	AIR	MN	

EPA 3C

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA 3C	Carbon dioxide	AIR	MN	
CAA	EPA 3C	Carbon monoxide	AIR	MN	
CAA	EPA 3C	Methane	AIR	MN	
CAA	EPA 3C	Nitrogen	AIR	MN	
CAA	EPA 3C	Oxygen	AIR	MN	

EPA Method 23

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA Method 23	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	AIR	MN	
CAA	EPA Method 23	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	AIR	MN	
CAA	EPA Method 23	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	AIR	MN	
CAA	EPA Method 23	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	AIR	MN	
CAA	EPA Method 23	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	AIR	MN	
CAA	EPA Method 23	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	AIR	MN	
CAA	EPA Method 23	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA Method 23	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	AIR	MN	
CAA	EPA Method 23	2,3,4,6,7,8-Hexachlorodibenzofuran	AIR	MN	
CAA	EPA Method 23	2,3,4,7,8-Pentachlorodibenzofuran	AIR	MN	
CAA	EPA Method 23	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	AIR	MN	
CAA	EPA Method 23	2,3,7,8-Tetrachlorodibenzofuran	AIR	MN	

EPA RSK-175 (GC/FID)

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA RSK-175 (GC/FID)	Ethane	AIR	MN	
CAA	EPA RSK-175 (GC/FID)	Ethene	AIR	MN	
CAA	EPA RSK-175 (GC/FID)	Methane	AIR	MN	
CAA	EPA RSK-175 (GC/FID)	n-Propane	AIR	MN	

EPA TO-14A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-14A	1,1,1-Trichloroethane	AIR	MN	
CAA	EPA TO-14A	1,1,2,2-Tetrachloroethane	AIR	MN	
CAA	EPA TO-14A	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	AIR	MN	
CAA	EPA TO-14A	1,1,2-Trichloroethane	AIR	MN	
CAA	EPA TO-14A	1,1-Dichloroethane	AIR	MN	
CAA	EPA TO-14A	1,1-Dichloroethylene	AIR	MN	
CAA	EPA TO-14A	1,2,4-Trichlorobenzene	AIR	MN	
CAA	EPA TO-14A	1,2,4-Trimethylbenzene	AIR	MN	
CAA	EPA TO-14A	1,2-Dibromoethane (EDB, Ethylene dibromide)	AIR	MN	
CAA	EPA TO-14A	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)	AIR	MN	
CAA	EPA TO-14A	1,2-Dichlorobenzene	AIR	MN	
CAA	EPA TO-14A	1,2-Dichloroethane (Ethylene dichloride)	AIR	MN	
CAA	EPA TO-14A	1,2-Dichloroethene (total)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-14A	1,2-Dichloropropane	AIR	MN	
CAA	EPA TO-14A	1,3-Dichlorobenzene	AIR	MN	
CAA	EPA TO-14A	1,4-Dichlorobenzene	AIR	MN	
CAA	EPA TO-14A	Benzene	AIR	MN	
CAA	EPA TO-14A	Bromomethane	AIR	MN	
CAA	EPA TO-14A	Carbon tetrachloride	AIR	MN	
CAA	EPA TO-14A	Chlorobenzene	AIR	MN	
CAA	EPA TO-14A	Chloroethane (Ethyl chloride)	AIR	MN	
CAA	EPA TO-14A	Chloroform	AIR	MN	
CAA	EPA TO-14A	cis-1,2-Dichloroethylene	AIR	MN	
CAA	EPA TO-14A	cis-1,3-Dichloropropene	AIR	MN	
CAA	EPA TO-14A	Dichlorodifluoromethane (Freon-12)	AIR	MN	
CAA	EPA TO-14A	Ethylbenzene	AIR	MN	
CAA	EPA TO-14A	Hexachloro-1,3-butadiene	AIR	MN	
CAA	EPA TO-14A	Hexachlorobutadiene	AIR	MN	
CAA	EPA TO-14A	m+p-xylene	AIR	MN	
CAA	EPA TO-14A	Methyl chloride (Chloromethane)	AIR	MN	
CAA	EPA TO-14A	Methyl tert-butyl ether (MTBE)	AIR	MN	
CAA	EPA TO-14A	Methylene chloride (Dichloromethane)	AIR	MN	
CAA	EPA TO-14A	n-Hexane	AIR	MN	
CAA	EPA TO-14A	o-Xylene	AIR	MN	
CAA	EPA TO-14A	Styrene	AIR	MN	
CAA	EPA TO-14A	Tetrachloroethene	AIR	MN	
CAA	EPA TO-14A	THC as Gas	AIR	MN	
CAA	EPA TO-14A	Toluene	AIR	MN	
CAA	EPA TO-14A	trans-1,2-Dichloroethylene	AIR	MN	
CAA	EPA TO-14A	trans-1,3-Dichloropropylene	AIR	MN	
CAA	EPA TO-14A	Trichloroethene (Trichloroethylene)	AIR	MN	
CAA	EPA TO-14A	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	AIR	MN	
CAA	EPA TO-14A	Vinyl chloride	AIR	MN	
CAA	EPA TO-14A	Xylene (total)	AIR	MN	

EPA TO-15

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-15	Isopropylbenzene	AIR	MN	
CAA	EPA TO-15	Methyl methacrylate	AIR	MN	
CAA	EPA TO-15	Vinyl bromide (Bromoethane)	AIR	MN	

EPA TO-17

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-17	1,1,2,2-Tetrachloroethane	AIR	MN	
CAA	EPA TO-17	1,1,2-Trichloroethane	AIR	MN	
CAA	EPA TO-17	1,1-Dichloroethylene	AIR	MN	
CAA	EPA TO-17	1,2,4-Trimethylbenzene	AIR	MN	
CAA	EPA TO-17	1,2-Dichloroethane (Ethylene dichloride)	AIR	MN	
CAA	EPA TO-17	1,3,5-Trimethylbenzene	AIR	MN	
CAA	EPA TO-17	Benzene	AIR	MN	
CAA	EPA TO-17	cis-1,2-Dichloroethylene	AIR	MN	
CAA	EPA TO-17	Ethylbenzene	AIR	MN	
CAA	EPA TO-17	Isopropylbenzene	AIR	MN	
CAA	EPA TO-17	m+p-xylene	AIR	MN	
CAA	EPA TO-17	Naphthalene	AIR	MN	
CAA	EPA TO-17	o-Xylene	AIR	MN	
CAA	EPA TO-17	Styrene	AIR	MN	
CAA	EPA TO-17	Tetrachloroethene	AIR	MN	
CAA	EPA TO-17	Toluene	AIR	MN	
CAA	EPA TO-17	trans-1,2-Dichloroethylene	AIR	MN	
CAA	EPA TO-17	Trichloroethene (Trichloroethylene)	AIR	MN	
CAA	EPA TO-17	Vinyl chloride	AIR	MN	

EPA TO-3

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-3	1,2,4-Trimethylbenzene	AIR	MN	
CAA	EPA TO-3	Benzene	AIR	MN	
CAA	EPA TO-3	Ethane	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-3	Ethene	AIR	MN	
CAA	EPA TO-3	Ethylbenzene	AIR	MN	
CAA	EPA TO-3	m+p-xylene	AIR	MN	
CAA	EPA TO-3	Methane	AIR	MN	
CAA	EPA TO-3	Methyl tert-butyl ether (MTBE)	AIR	MN	
CAA	EPA TO-3	n-Hexane	AIR	MN	
CAA	EPA TO-3	o-Xylene	AIR	MN	
CAA	EPA TO-3	THC as C1-C4	AIR	MN	
CAA	EPA TO-3	THC as Gas	AIR	MN	
CAA	EPA TO-3	Toluene	AIR	MN	
CAA	EPA TO-3	Total BTEX	AIR	MN	
CAA	EPA TO-3	Xylene (total)	AIR	MN	

EPA TO-9A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-9A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	AIR	MN	
CAA	EPA TO-9A	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	AIR	MN	
CAA	EPA TO-9A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	AIR	MN	
CAA	EPA TO-9A	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	AIR	MN	
CAA	EPA TO-9A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	AIR	MN	
CAA	EPA TO-9A	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	AIR	MN	
CAA	EPA TO-9A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	AIR	MN	
CAA	EPA TO-9A	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CAA	EPA TO-9A	2,3,4,6,7,8-Hexachlorodibenzofuran	AIR	MN	
CAA	EPA TO-9A	2,3,4,7,8-Pentachlorodibenzofuran	AIR	MN	
CAA	EPA TO-9A	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	AIR	MN	
CAA	EPA TO-9A	2,3,7,8-Tetrachlorodibenzofuran	AIR	MN	

Clean Water Program

ASTM D516-07

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	ASTM D516-07	Sulfate	NPW	MN	

ASTM D516-11

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	ASTM D516-11	Sulfate	NPW	MN	

ASTM D516-90

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	ASTM D516-90	Sulfate	NPW	MN	

EPA 120.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 120.1	Conductivity	NPW	MN	

EPA 160.4

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 160.4	Residue-volatile	NPW	MN	

EPA 1664A (HEM)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664A (HEM)	Oil & Grease	NPW	MN	

EPA 1664A (SGT-HEM)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664A (SGT-HEM)	Oil & Grease	NPW	MN	

EPA 1664B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664B	Oil & Grease	NPW	MN	

EPA 1664B (SGT-HEM)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1664B (SGT-HEM)	Oil & Grease	NPW	MN	

EPA 180.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 180.1	Turbidity	NPW	MN	

EPA 300.0

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 300.0	Bromide	NPW	MN	
CWP	EPA 300.0	Chloride	NPW	MN	
CWP	EPA 300.0	Fluoride	NPW	MN	
CWP	EPA 300.0	Nitrate as N	NPW	MN	
CWP	EPA 300.0	Nitrite as N	NPW	MN	
CWP	EPA 300.0	Sulfate	NPW	MN	

EPA 350.1

Preparation Techniques: Gas Diffusion;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 350.1	Ammonia as N	NPW	MN	

EPA 353.2

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 353.2	Nitrate-nitrite	NPW	MN	
CWP	EPA 353.2	Nitrite as N	NPW	MN	

EPA 353.2 (calc.)

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 353.2 (calc.)	Nitrate as N	NPW	MN	

EPA 410.4

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 410.4	Chemical oxygen demand	NPW	MN	

EPA 420.4

Preparation Techniques: Distillation, MIDI;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 420.4	Total Phenolics	NPW	MN	

Hach 10360

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	Hach 10360	Biochemical oxygen demand	NPW	MN	
CWP	Hach 10360	Carbonaceous BOD, CBOD	NPW	MN	
CWP	Hach 10360	Oxygen, dissolved	NPW	MN	

HACH 10360 Rev 1.2 (2011)

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	HACH 10360 Rev 1.2 (2011)	Biochemical oxygen demand	NPW	MN	
CWP	HACH 10360 Rev 1.2 (2011)	Carbonaceous BOD, CBOD	NPW	MN	
CWP	HACH 10360 Rev 1.2 (2011)	Oxygen, dissolved	NPW	MN	

SM 2320 B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2320 B-2011	Alkalinity as CaCO3	NPW	MN	

SM 2320 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2320 B-97	Alkalinity as CaCO3	NPW	MN	

SM 2340 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2340 B-97	Total hardness as CaCO3	NPW	MN	

SM 2510 B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2510 B-2011	Conductivity	NPW	MN	

SM 2510 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2510 B-97	Conductivity	NPW	MN	

SM 2540 B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 B-2011	Residue-total	NPW	MN	

SM 2540 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 B-97	Residue-total	NPW	MN	

SM 2540 C-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 C-2011	Residue-filterable (TDS)	NPW	MN	

SM 2540 C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 C-97	Residue-filterable (TDS)	NPW	MN	

SM 2540 D-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 D-2011	Residue-nonfilterable (TSS)	NPW	MN	

SM 2540 D-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 D-97	Residue-nonfilterable (TSS)	NPW	MN	

SM 2540 F-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 F-2011	Residue-settleable	NPW	MN	

SM 2540 F-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 2540 F-97	Residue-settleable	NPW	MN	

SM 4500-Cl G-2000

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl G-2000	Total residual chlorine	NPW	MN	

SM 4500-Cl G-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl G-2011	Total residual chlorine	NPW	MN	

SM 4500-Cl G-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl G-93	Total residual chlorine	NPW	MN	

SM 4500-Cl^- E-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl^-E-2011	Chloride	NPW	MN	

SM 4500-Cl^- E-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-Cl^-E-97	Chloride	NPW	MN	

SM 4500-CN^- E-2011

Preparation Techniques: Distillation, micro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-CN⁻E-2011	Total Cyanide	NPW	MN	

SM 4500-CN⁻E-97

Preparation Techniques: Distillation, micro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-CN⁻E-97	Total Cyanide	NPW	MN	

SM 4500-CN⁻G-1999

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-CN⁻G-1999	Amenable cyanide	NPW	MN	

SM 4500-CN⁻G-2011

Preparation Techniques: Distillation, micro;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-CN⁻G-2011	Amenable cyanide	NPW	MN	

SM 4500-F⁻C-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-F⁻C-2011	Fluoride	NPW	MN	

SM 4500-F⁻C-97

Preparation Techniques: N/A;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-F⁻C-97	Fluoride	NPW	MN	

SM 4500-H+ B-2000

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-H+ B-2000	pH	NPW	MN	

SM 4500-H+ B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-H+ B-2011	pH	NPW	MN	

SM 4500-H+ B-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-H+ B-96	pH	NPW	MN	

SM 4500-NO_2^- B-2000

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO_2^- B-2000	Nitrite as N	NPW	MN	

SM 4500-NO_2^- B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO_2^- B-2011	Nitrite as N	NPW	MN	

SM 4500-NO_2^- B-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO2⁻B-93	Nitrite as N	NPW	MN	

SM 4500-NO3⁻H-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-NO3⁻H-97	Nitrate-nitrite	NPW	MN	

SM 4500-P E-1999

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P E-1999	Total Phosphorus	NPW	MN	

SM 4500-P E-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P E-2011	Total Phosphorus	NPW	MN	

SM 4500-P E-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P E-97	Total Phosphorus	NPW	MN	

SM 4500-P G-1999

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P G-1999	Orthophosphate as P	NPW	MN	

SM 4500-P G-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 4500-P G-2011	Orthophosphate as P	NPW	MN	

SM 5220 D-2011

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5220 D-2011	Chemical oxygen demand	NPW	MN	

SM 5220 D-97

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 5220 D-97	Chemical oxygen demand	NPW	MN	

EPA 200.7

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Aluminum	NPW	MN	
CWP	EPA 200.7	Antimony	NPW	MN	
CWP	EPA 200.7	Arsenic	NPW	MN	
CWP	EPA 200.7	Barium	NPW	MN	
CWP	EPA 200.7	Beryllium	NPW	MN	
CWP	EPA 200.7	Boron	NPW	MN	
CWP	EPA 200.7	Cadmium	NPW	MN	
CWP	EPA 200.7	Calcium	NPW	MN	
CWP	EPA 200.7	Chromium	NPW	MN	
CWP	EPA 200.7	Cobalt	NPW	MN	
CWP	EPA 200.7	Copper	NPW	MN	
CWP	EPA 200.7	Iron	NPW	MN	
CWP	EPA 200.7	Lead	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.7	Magnesium	NPW	MN	
CWP	EPA 200.7	Manganese	NPW	MN	
CWP	EPA 200.7	Molybdenum	NPW	MN	
CWP	EPA 200.7	Nickel	NPW	MN	
CWP	EPA 200.7	Potassium	NPW	MN	
CWP	EPA 200.7	Selenium	NPW	MN	
CWP	EPA 200.7	Silver	NPW	MN	
CWP	EPA 200.7	Sodium	NPW	MN	
CWP	EPA 200.7	Thallium	NPW	MN	
CWP	EPA 200.7	Tin	NPW	MN	
CWP	EPA 200.7	Titanium	NPW	MN	
CWP	EPA 200.7	Total chromium	NPW	MN	
CWP	EPA 200.7	Total hardness as CaCO3	NPW	MN	
CWP	EPA 200.7	Vanadium	NPW	MN	
CWP	EPA 200.7	Zinc	NPW	MN	

EPA 200.8

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.8	Aluminum	NPW	MN	
CWP	EPA 200.8	Antimony	NPW	MN	
CWP	EPA 200.8	Arsenic	NPW	MN	
CWP	EPA 200.8	Barium	NPW	MN	
CWP	EPA 200.8	Beryllium	NPW	MN	
CWP	EPA 200.8	Bismuth	NPW	MN	
CWP	EPA 200.8	Boron	NPW	MN	
CWP	EPA 200.8	Cadmium	NPW	MN	
CWP	EPA 200.8	Calcium	NPW	MN	
CWP	EPA 200.8	Chromium	NPW	MN	
CWP	EPA 200.8	Cobalt	NPW	MN	
CWP	EPA 200.8	Copper	NPW	MN	
CWP	EPA 200.8	Iron	NPW	MN	
CWP	EPA 200.8	Lead	NPW	MN	
CWP	EPA 200.8	Lithium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 200.8	Magnesium	NPW	MN	
CWP	EPA 200.8	Manganese	NPW	MN	
CWP	EPA 200.8	Molybdenum	NPW	MN	
CWP	EPA 200.8	Nickel	NPW	MN	
CWP	EPA 200.8	Palladium	NPW	MN	
CWP	EPA 200.8	Platinum	NPW	MN	
CWP	EPA 200.8	Potassium	NPW	MN	
CWP	EPA 200.8	Selenium	NPW	MN	
CWP	EPA 200.8	Silicon	NPW	MN	
CWP	EPA 200.8	Silver	NPW	MN	
CWP	EPA 200.8	Sodium	NPW	MN	
CWP	EPA 200.8	Strontium	NPW	MN	
CWP	EPA 200.8	Thallium	NPW	MN	
CWP	EPA 200.8	Tin	NPW	MN	
CWP	EPA 200.8	Titanium	NPW	MN	
CWP	EPA 200.8	Total chromium	NPW	MN	
CWP	EPA 200.8	Uranium	NPW	MN	
CWP	EPA 200.8	Vanadium	NPW	MN	
CWP	EPA 200.8	Zinc	NPW	MN	

EPA 245.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 245.1	Mercury	NPW	MN	

SM 3500-Cr B-2009

Preparation Techniques: N/A;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Cr B-2009	Chromium VI	NPW	MN	

SM 3500-Cr B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Cr B-2011	Chromium VI	NPW	MN	

SM 3500-Cr B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Cr B-97	Chromium VI	NPW	MN	

SM 3500-Fe B-2011

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Fe B-2011	Iron	NPW	MN	

SM 3500-Fe B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 3500-Fe B-97	Iron	NPW	MN	

SM 9222 B (M-Endo)-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 9222 B (M-Endo)-97	Total coliforms	NPW	MN	

SM 9222 D (m-FC)-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 9222 D (m-FC)-97	Fecal coliforms	NPW	MN	

SM 9223 B (Colilert® Quanti-Tray®)-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	SM 9223 B (Colilert® Quanti-Tray®)-97	Escherichia coli	NPW	MN	

EPA 1613B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, automated soxhlet; Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	NPW	MN	
CWP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	NPW	MN	
CWP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	NPW	MN	
CWP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	NPW	MN	
CWP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	NPW	MN	
CWP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	NPW	MN	
CWP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	NPW	MN	
CWP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	NPW	MN	
CWP	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	NPW	MN	
CWP	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran	NPW	MN	
CWP	EPA 1613B	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	NPW	MN	
CWP	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran	NPW	MN	
CWP	EPA 1613B	Total HpCDD	NPW	MN	
CWP	EPA 1613B	Total HpCDF	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1613B	Total HxCDD	NPW	MN	
CWP	EPA 1613B	Total HxCDF	NPW	MN	
CWP	EPA 1613B	Total PeCDD	NPW	MN	
CWP	EPA 1613B	Total PeCDF	NPW	MN	
CWP	EPA 1613B	Total TCDD	NPW	MN	
CWP	EPA 1613B	Total TCDF	NPW	MN	

EPA 1668A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	NPW	MN	
CWP	EPA 1668A	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	NPW	MN	
CWP	EPA 1668A	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	NPW	MN	
CWP	EPA 1668A	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	NPW	MN	
CWP	EPA 1668A	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	NPW	MN	
CWP	EPA 1668A	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	NPW	MN	
CWP	EPA 1668A	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	NPW	MN	
CWP	EPA 1668A	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	NPW	MN	
CWP	EPA 1668A	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	NPW	MN	
CWP	EPA 1668A	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	NPW	MN	
CWP	EPA 1668A	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	NPW	MN	
CWP	EPA 1668A	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	NPW	MN	
CWP	EPA 1668A	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	NPW	MN	
CWP	EPA 1668A	2,2',3-Trichlorobiphenyl (BZ-16)	NPW	MN	
CWP	EPA 1668A	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	NPW	MN	
CWP	EPA 1668A	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	NPW	MN	
CWP	EPA 1668A	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	NPW	MN	
CWP	EPA 1668A	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	NPW	MN	
CWP	EPA 1668A	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	NPW	MN	
CWP	EPA 1668A	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	NPW	MN	
CWP	EPA 1668A	2,2',4-Trichlorobiphenyl (BZ-17)	NPW	MN	
CWP	EPA 1668A	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	NPW	MN	
CWP	EPA 1668A	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	NPW	MN	
CWP	EPA 1668A	2,2',6-Trichlorobiphenyl (BZ-19)	NPW	MN	
CWP	EPA 1668A	2,2'-Dichlorobiphenyl (BZ-4)	NPW	MN	
CWP	EPA 1668A	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	NPW	MN	
CWP	EPA 1668A	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	NPW	MN	
CWP	EPA 1668A	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	NPW	MN	
CWP	EPA 1668A	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	NPW	MN	
CWP	EPA 1668A	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	NPW	MN	
CWP	EPA 1668A	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	NPW	MN	
CWP	EPA 1668A	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	NPW	MN	
CWP	EPA 1668A	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	NPW	MN	
CWP	EPA 1668A	2,3',4-Trichlorobiphenyl (BZ-25)	NPW	MN	
CWP	EPA 1668A	2,3',5'-Trichlorobiphenyl (BZ-34)	NPW	MN	
CWP	EPA 1668A	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	NPW	MN	
CWP	EPA 1668A	2,3',6-Trichlorobiphenyl (BZ-27)	NPW	MN	
CWP	EPA 1668A	2,3'-Dichlorobiphenyl (BZ-6)	NPW	MN	
CWP	EPA 1668A	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	NPW	MN	
CWP	EPA 1668A	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	NPW	MN	
CWP	EPA 1668A	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	NPW	MN	
CWP	EPA 1668A	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	NPW	MN	
CWP	EPA 1668A	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	NPW	MN	
CWP	EPA 1668A	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	NPW	MN	
CWP	EPA 1668A	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	NPW	MN	
CWP	EPA 1668A	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	NPW	MN	
CWP	EPA 1668A	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	NPW	MN	
CWP	EPA 1668A	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	NPW	MN	
CWP	EPA 1668A	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	NPW	MN	
CWP	EPA 1668A	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	NPW	MN	
CWP	EPA 1668A	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	NPW	MN	
CWP	EPA 1668A	2,3,4'-Trichlorobiphenyl (BZ-22)	NPW	MN	
CWP	EPA 1668A	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	NPW	MN	
CWP	EPA 1668A	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	NPW	MN	
CWP	EPA 1668A	2,3,5-Trichlorobiphenyl (BZ-23)	NPW	MN	
CWP	EPA 1668A	2,3,6-Trichlorobiphenyl (BZ-24)	NPW	MN	
CWP	EPA 1668A	2,3-Dichlorobiphenyl (BZ-5)	NPW	MN	
CWP	EPA 1668A	2,4',5-Trichlorobiphenyl (BZ-31)	NPW	MN	
CWP	EPA 1668A	2,4',6-Trichlorobiphenyl (BZ-32)	NPW	MN	
CWP	EPA 1668A	2,4'-Dichlorobiphenyl (BZ-8)	NPW	MN	
CWP	EPA 1668A	2,4-Dichlorobiphenyl (BZ-7)	NPW	MN	
CWP	EPA 1668A	2,5-Dichlorobiphenyl (BZ-9)	NPW	MN	
CWP	EPA 1668A	2,6-Dichlorobiphenyl (BZ-10)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	2-Chlorobiphenyl (BZ-1)	NPW	MN	
CWP	EPA 1668A	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	NPW	MN	
CWP	EPA 1668A	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	NPW	MN	
CWP	EPA 1668A	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	NPW	MN	
CWP	EPA 1668A	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	NPW	MN	
CWP	EPA 1668A	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	NPW	MN	
CWP	EPA 1668A	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	NPW	MN	
CWP	EPA 1668A	3,3',4-Trichlorobiphenyl (BZ-35)	NPW	MN	
CWP	EPA 1668A	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	NPW	MN	
CWP	EPA 1668A	3,3',5-Trichlorobiphenyl (BZ-36)	NPW	MN	
CWP	EPA 1668A	3,3'-Dichlorobiphenyl (BZ-11)	NPW	MN	
CWP	EPA 1668A	3,4',5-Trichlorobiphenyl (BZ-39)	NPW	MN	
CWP	EPA 1668A	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	NPW	MN	
CWP	EPA 1668A	3,4,4'-Trichlorobiphenyl (BZ-37)	NPW	MN	
CWP	EPA 1668A	3,4,5-Trichlorobiphenyl (BZ-38)	NPW	MN	
CWP	EPA 1668A	3,5-Dichlorobiphenyl (BZ-14)	NPW	MN	
CWP	EPA 1668A	3-Chlorobiphenyl (BZ-2)	NPW	MN	
CWP	EPA 1668A	4,4'-Dichlorobiphenyl (BZ-15)	NPW	MN	
CWP	EPA 1668A	4-Chlorobiphenyl (BZ-3)	NPW	MN	
CWP	EPA 1668A	Decachlorobiphenyl (BZ-209)	NPW	MN	
CWP	EPA 1668A	PCB-(100/93/102/98)	NPW	MN	
CWP	EPA 1668A	PCB-(107/124)	NPW	MN	
CWP	EPA 1668A	PCB-(108/119/86/97/125/87)	NPW	MN	
CWP	EPA 1668A	PCB-(110/115)	NPW	MN	
CWP	EPA 1668A	PCB-(113/90/101)	NPW	MN	
CWP	EPA 1668A	PCB-(117/116/85)	NPW	MN	
CWP	EPA 1668A	PCB-(128/166)	NPW	MN	
CWP	EPA 1668A	PCB-(13/12)	NPW	MN	
CWP	EPA 1668A	PCB-(134/143)	NPW	MN	
CWP	EPA 1668A	PCB-(138/163/129)	NPW	MN	
CWP	EPA 1668A	PCB-(139/140)	NPW	MN	
CWP	EPA 1668A	PCB-(147/149)	NPW	MN	
CWP	EPA 1668A	PCB-(151/135)	NPW	MN	
CWP	EPA 1668A	PCB-(153/168)	NPW	MN	
CWP	EPA 1668A	PCB-(156/157)	NPW	MN	
CWP	EPA 1668A	PCB-(171/173)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 1668A	PCB-(180/193)	NPW	MN	
CWP	EPA 1668A	PCB-(183/185)	NPW	MN	
CWP	EPA 1668A	PCB-(197/200)	NPW	MN	
CWP	EPA 1668A	PCB-(198/199)	NPW	MN	
CWP	EPA 1668A	PCB-(21/33)	NPW	MN	
CWP	EPA 1668A	PCB-(26/29)	NPW	MN	
CWP	EPA 1668A	PCB-(28/20)	NPW	MN	
CWP	EPA 1668A	PCB-(30/18)	NPW	MN	
CWP	EPA 1668A	PCB-(41/40/71)	NPW	MN	
CWP	EPA 1668A	PCB-(44/47/65)	NPW	MN	
CWP	EPA 1668A	PCB-(45/51)	NPW	MN	
CWP	EPA 1668A	PCB-(50/53)	NPW	MN	
CWP	EPA 1668A	PCB-(59/62/75)	NPW	MN	
CWP	EPA 1668A	PCB-(61/70/74/76)	NPW	MN	
CWP	EPA 1668A	PCB-(69/49)	NPW	MN	
CWP	EPA 1668A	PCB-(73/43)	NPW	MN	
CWP	EPA 1668A	PCB-(88/91)	NPW	MN	

EPA 625

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, continuous liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	1,2,4-Trichlorobenzene	NPW	MN	
CWP	EPA 625	2,4,5-Trichlorophenol	NPW	MN	
CWP	EPA 625	2,4,6-Trichlorophenol	NPW	MN	
CWP	EPA 625	2,4-Dichlorophenol	NPW	MN	
CWP	EPA 625	2,4-Dimethylphenol	NPW	MN	
CWP	EPA 625	2,4-Dinitrophenol	NPW	MN	
CWP	EPA 625	2,4-Dinitrotoluene (2,4-DNT)	NPW	MN	
CWP	EPA 625	2,6-Dinitrotoluene (2,6-DNT)	NPW	MN	
CWP	EPA 625	2-Chloronaphthalene	NPW	MN	
CWP	EPA 625	2-Chlorophenol	NPW	MN	
CWP	EPA 625	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	MN	
CWP	EPA 625	2-Nitrophenol	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	3,3'-Dichlorobenzidine	NPW	MN	
CWP	EPA 625	4-Bromophenyl phenyl ether	NPW	MN	
CWP	EPA 625	4-Chloro-3-methylphenol	NPW	MN	
CWP	EPA 625	4-Chlorophenyl phenylether	NPW	MN	
CWP	EPA 625	4-Nitrophenol	NPW	MN	
CWP	EPA 625	Acenaphthene	NPW	MN	
CWP	EPA 625	Acenaphthylene	NPW	MN	
CWP	EPA 625	Anthracene	NPW	MN	
CWP	EPA 625	Benzidine	NPW	MN	
CWP	EPA 625	Benzo(a)anthracene	NPW	MN	
CWP	EPA 625	Benzo(a)pyrene	NPW	MN	
CWP	EPA 625	Benzo(g,h,i)perylene	NPW	MN	
CWP	EPA 625	Benzo(k)fluoranthene	NPW	MN	
CWP	EPA 625	Benzo[b]fluoranthene	NPW	MN	
CWP	EPA 625	bis(2-Chloroethoxy)methane	NPW	MN	
CWP	EPA 625	bis(2-Chloroethyl) ether	NPW	MN	
CWP	EPA 625	bis(2-Chloroisopropyl) ether	NPW	MN	
CWP	EPA 625	Butyl benzyl phthalate	NPW	MN	
CWP	EPA 625	Chrysene	NPW	MN	
CWP	EPA 625	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	MN	
CWP	EPA 625	Di-n-butyl phthalate	NPW	MN	
CWP	EPA 625	Di-n-octyl phthalate	NPW	MN	
CWP	EPA 625	Dibenz(a,h) anthracene	NPW	MN	
CWP	EPA 625	Diethyl phthalate	NPW	MN	
CWP	EPA 625	Dimethyl phthalate	NPW	MN	
CWP	EPA 625	Fluoranthene	NPW	MN	
CWP	EPA 625	Fluorene	NPW	MN	
CWP	EPA 625	Hexachlorobenzene	NPW	MN	
CWP	EPA 625	Hexachlorobutadiene	NPW	MN	
CWP	EPA 625	Hexachlorocyclopentadiene	NPW	MN	
CWP	EPA 625	Hexachloroethane	NPW	MN	
CWP	EPA 625	Indeno(1,2,3-cd) pyrene	NPW	MN	
CWP	EPA 625	Isophorone	NPW	MN	
CWP	EPA 625	n-Nitrosodi-n-propylamine	NPW	MN	
CWP	EPA 625	n-Nitrosodimethylamine	NPW	MN	
CWP	EPA 625	n-Nitrosodiphenylamine	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 625	Naphthalene	NPW	MN	
CWP	EPA 625	Nitrobenzene	NPW	MN	
CWP	EPA 625	Pentachlorophenol	NPW	MN	
CWP	EPA 625	Phenanthrene	NPW	MN	
CWP	EPA 625	Phenol	NPW	MN	
CWP	EPA 625	Pyrene	NPW	MN	

EPA 624

Preparation Techniques: Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	1,1,1-Trichloroethane	NPW	MN	
CWP	EPA 624	1,1,2,2-Tetrachloroethane	NPW	MN	
CWP	EPA 624	1,1,2-Trichloroethane	NPW	MN	
CWP	EPA 624	1,1-Dichloroethane	NPW	MN	
CWP	EPA 624	1,1-Dichloroethylene	NPW	MN	
CWP	EPA 624	1,2,4-Trichlorobenzene	NPW	MN	
CWP	EPA 624	1,2-Dichlorobenzene	NPW	MN	
CWP	EPA 624	1,2-Dichloroethane (Ethylene dichloride)	NPW	MN	
CWP	EPA 624	1,2-Dichloropropane	NPW	MN	
CWP	EPA 624	1,3-Dichlorobenzene	NPW	MN	
CWP	EPA 624	1,4-Dichlorobenzene	NPW	MN	
CWP	EPA 624	2-Butanone (Methyl ethyl ketone, MEK)	NPW	MN	
CWP	EPA 624	2-Chloroethyl vinyl ether	NPW	MN	
CWP	EPA 624	Acetone	NPW	MN	
CWP	EPA 624	Acrolein (Propenal)	NPW	MN	
CWP	EPA 624	Acrylonitrile	NPW	MN	
CWP	EPA 624	Benzene	NPW	MN	
CWP	EPA 624	Bromodichloromethane	NPW	MN	
CWP	EPA 624	Bromoform	NPW	MN	
CWP	EPA 624	Carbon tetrachloride	NPW	MN	
CWP	EPA 624	Chlorobenzene	NPW	MN	
CWP	EPA 624	Chlorodibromomethane	NPW	MN	
CWP	EPA 624	Chloroethane (Ethyl chloride)	NPW	MN	
CWP	EPA 624	Chloroform	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
CWP	EPA 624	cis-1,3-Dichloropropene	NPW	MN	
CWP	EPA 624	Ethylbenzene	NPW	MN	
CWP	EPA 624	Isopropyl acetate	NPW	MN	
CWP	EPA 624	Isopropyl alcohol (2-Propanol, Isopropanol)	NPW	MN	
CWP	EPA 624	Isopropylbenzene	NPW	MN	
CWP	EPA 624	Methyl bromide (Bromomethane)	NPW	MN	
CWP	EPA 624	Methyl chloride (Chloromethane)	NPW	MN	
CWP	EPA 624	Methylene chloride (Dichloromethane)	NPW	MN	
CWP	EPA 624	Tetrachloroethylene (Perchloroethylene)	NPW	MN	
CWP	EPA 624	Toluene	NPW	MN	
CWP	EPA 624	trans-1,2-Dichloroethylene	NPW	MN	
CWP	EPA 624	trans-1,3-Dichloropropylene	NPW	MN	
CWP	EPA 624	Trichloroethene (Trichloroethylene)	NPW	MN	
CWP	EPA 624	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	MN	
CWP	EPA 624	Vinyl chloride	NPW	MN	

Resource Conservation Recovery Program

MDA GD24 (Ag List 2)

Preparation Techniques: Extraction, microwave; Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	MDA GD24 (Ag List 2)	2,4,5-T	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	2,4,5-T	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	2,4-D	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	2,4-D	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	2,4-DB	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	2,4-DB	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	Bentazon	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	Bentazon	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	Dicamba	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	Dicamba	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	Garlon (Triclopyr)	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	MCPA	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	MCPA	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	MDA GD24 (Ag List 2)	Picloram	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	Picloram	SCM	MN	
RCRP	MDA GD24 (Ag List 2)	Silvex (2,4,5-TP)	NPW	MN	
RCRP	MDA GD24 (Ag List 2)	Silvex (2,4,5-TP)	SCM	MN	

MPCA Guidance PFCs

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	MPCA Guidance PFCs	Dodecafluoro-3H-4,8-dioxanonoate (NaDONA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Dodecafluoro-3H-4,8-dioxanonoate (NaDONA)	SCM	MN	
RCRP	MPCA Guidance PFCs	N-Ethylperfluorooctanesulfonamidoacetic acid	SCM	MN	
RCRP	MPCA Guidance PFCs	N-Ethylperfluorooctanesulfonamidoacetic acid	NPW	MN	
RCRP	MPCA Guidance PFCs	N-Methylperfluorooctanesulfonamidoacetic acid	SCM	MN	
RCRP	MPCA Guidance PFCs	N-Methylperfluorooctanesulfonamidoacetic acid	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorobutane sulfonate (PFBS)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorobutane sulfonate (PFBS)	DW	MN	
RCRP	MPCA Guidance PFCs	Perfluorobutanoic acid (pfba)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorodecane sulfonic acid	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorodecane sulfonic acid	SCM	MN	
RCRP	MPCA Guidance PFCs	Perfluorodecanoic acid (PFDA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorododecanoic acid (PFDOA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluoroheptanoic acid (PFHpA)	DW	MN	
RCRP	MPCA Guidance PFCs	Perfluoroheptanoic acid (PFHpA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorohexadecanoic acid (pfhxda)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorohexane sulfonate (PFHxS)	DW	MN	
RCRP	MPCA Guidance PFCs	Perfluorohexane sulfonate (PFHxS)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorohexanoic acid (pfhxa)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorononanoic acid (pfna)	DW	MN	
RCRP	MPCA Guidance PFCs	Perfluorononanoic acid (pfna)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorooctadecanoic acid (pfoda)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorooctane sulfonate (PFOS)	DW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	MPCA Guidance PFCs	Perfluorooctane sulfonate (PFOS)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorooctanoic acid (PFOA)	DW	MN	
RCRP	MPCA Guidance PFCs	Perfluorooctanoic acid (PFOA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluoropentanoic acid (PFPeA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorotetradecanoic acid (PFTDA)	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorotridecanoic acid	NPW	MN	
RCRP	MPCA Guidance PFCs	Perfluorotridecanoic acid	SCM	MN	
RCRP	MPCA Guidance PFCs	Perfluoroundecanoic acid (PFUDA)	NPW	MN	

EPA 9045D

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9045D	pH	SCM	MN	

EPA 9056A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9056A	Bromide	NPW	MN	
RCRP	EPA 9056A	Chloride	NPW	MN	
RCRP	EPA 9056A	Fluoride	NPW	MN	
RCRP	EPA 9056A	Nitrate	NPW	MN	
RCRP	EPA 9056A	Nitrite	NPW	MN	
RCRP	EPA 9056A	Sulfate	NPW	MN	

EPA 9071B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9071B	n-Hexane Extractable Material (O&G)	SCM	MN	
RCRP	EPA 9071B	Oil & Grease	SCM	MN	

EPA 6010B

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Aluminum	SCM	MN	
RCRP	EPA 6010B	Aluminum	NPW	MN	
RCRP	EPA 6010B	Antimony	SCM	MN	
RCRP	EPA 6010B	Antimony	NPW	MN	
RCRP	EPA 6010B	Arsenic	NPW	MN	
RCRP	EPA 6010B	Arsenic	SCM	MN	
RCRP	EPA 6010B	Barium	SCM	MN	
RCRP	EPA 6010B	Barium	NPW	MN	
RCRP	EPA 6010B	Beryllium	NPW	MN	
RCRP	EPA 6010B	Beryllium	SCM	MN	
RCRP	EPA 6010B	Boron	SCM	MN	
RCRP	EPA 6010B	Boron	NPW	MN	
RCRP	EPA 6010B	Cadmium	NPW	MN	
RCRP	EPA 6010B	Cadmium	SCM	MN	
RCRP	EPA 6010B	Calcium	SCM	MN	
RCRP	EPA 6010B	Calcium	NPW	MN	
RCRP	EPA 6010B	Chromium	SCM	MN	
RCRP	EPA 6010B	Cobalt	SCM	MN	
RCRP	EPA 6010B	Cobalt	NPW	MN	
RCRP	EPA 6010B	Copper	NPW	MN	
RCRP	EPA 6010B	Copper	SCM	MN	
RCRP	EPA 6010B	Iron	NPW	MN	
RCRP	EPA 6010B	Iron	SCM	MN	
RCRP	EPA 6010B	Lead	SCM	MN	
RCRP	EPA 6010B	Lead	NPW	MN	
RCRP	EPA 6010B	Magnesium	SCM	MN	
RCRP	EPA 6010B	Magnesium	NPW	MN	
RCRP	EPA 6010B	Manganese	NPW	MN	
RCRP	EPA 6010B	Manganese	SCM	MN	
RCRP	EPA 6010B	Molybdenum	NPW	MN	
RCRP	EPA 6010B	Molybdenum	SCM	MN	
RCRP	EPA 6010B	Nickel	NPW	MN	
RCRP	EPA 6010B	Nickel	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010B	Potassium	NPW	MN	
RCRP	EPA 6010B	Potassium	SCM	MN	
RCRP	EPA 6010B	Selenium	NPW	MN	
RCRP	EPA 6010B	Selenium	SCM	MN	
RCRP	EPA 6010B	Silver	SCM	MN	
RCRP	EPA 6010B	Silver	NPW	MN	
RCRP	EPA 6010B	Sodium	SCM	MN	
RCRP	EPA 6010B	Sodium	NPW	MN	
RCRP	EPA 6010B	Thallium	NPW	MN	
RCRP	EPA 6010B	Thallium	SCM	MN	
RCRP	EPA 6010B	Tin	NPW	MN	
RCRP	EPA 6010B	Tin	SCM	MN	
RCRP	EPA 6010B	Titanium	SCM	MN	
RCRP	EPA 6010B	Titanium	NPW	MN	
RCRP	EPA 6010B	Total chromium	NPW	MN	
RCRP	EPA 6010B	Vanadium	SCM	MN	
RCRP	EPA 6010B	Vanadium	NPW	MN	
RCRP	EPA 6010B	Zinc	SCM	MN	
RCRP	EPA 6010B	Zinc	NPW	MN	

EPA 6010C

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010C	Aluminum	NPW	MN	
RCRP	EPA 6010C	Aluminum	SCM	MN	
RCRP	EPA 6010C	Antimony	SCM	MN	
RCRP	EPA 6010C	Antimony	NPW	MN	
RCRP	EPA 6010C	Arsenic	NPW	MN	
RCRP	EPA 6010C	Arsenic	SCM	MN	
RCRP	EPA 6010C	Barium	SCM	MN	
RCRP	EPA 6010C	Barium	NPW	MN	
RCRP	EPA 6010C	Beryllium	NPW	MN	
RCRP	EPA 6010C	Beryllium	SCM	MN	
RCRP	EPA 6010C	Boron	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010C	Boron	NPW	MN	
RCRP	EPA 6010C	Cadmium	SCM	MN	
RCRP	EPA 6010C	Cadmium	NPW	MN	
RCRP	EPA 6010C	Calcium	NPW	MN	
RCRP	EPA 6010C	Calcium	SCM	MN	
RCRP	EPA 6010C	Chromium	NPW	MN	
RCRP	EPA 6010C	Chromium	SCM	MN	
RCRP	EPA 6010C	Cobalt	NPW	MN	
RCRP	EPA 6010C	Cobalt	SCM	MN	
RCRP	EPA 6010C	Copper	NPW	MN	
RCRP	EPA 6010C	Copper	SCM	MN	
RCRP	EPA 6010C	Iron	NPW	MN	
RCRP	EPA 6010C	Iron	SCM	MN	
RCRP	EPA 6010C	Lead	NPW	MN	
RCRP	EPA 6010C	Lead	SCM	MN	
RCRP	EPA 6010C	Magnesium	SCM	MN	
RCRP	EPA 6010C	Magnesium	NPW	MN	
RCRP	EPA 6010C	Manganese	SCM	MN	
RCRP	EPA 6010C	Manganese	NPW	MN	
RCRP	EPA 6010C	Molybdenum	SCM	MN	
RCRP	EPA 6010C	Molybdenum	NPW	MN	
RCRP	EPA 6010C	Nickel	SCM	MN	
RCRP	EPA 6010C	Nickel	NPW	MN	
RCRP	EPA 6010C	Potassium	SCM	MN	
RCRP	EPA 6010C	Potassium	NPW	MN	
RCRP	EPA 6010C	Selenium	SCM	MN	
RCRP	EPA 6010C	Selenium	NPW	MN	
RCRP	EPA 6010C	Silver	NPW	MN	
RCRP	EPA 6010C	Silver	SCM	MN	
RCRP	EPA 6010C	Sodium	NPW	MN	
RCRP	EPA 6010C	Sodium	SCM	MN	
RCRP	EPA 6010C	Thallium	NPW	MN	
RCRP	EPA 6010C	Thallium	SCM	MN	
RCRP	EPA 6010C	Tin	NPW	MN	
RCRP	EPA 6010C	Tin	SCM	MN	
RCRP	EPA 6010C	Titanium	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010C	Titanium	NPW	MN	
RCRP	EPA 6010C	Vanadium	NPW	MN	
RCRP	EPA 6010C	Vanadium	SCM	MN	
RCRP	EPA 6010C	Zinc	SCM	MN	
RCRP	EPA 6010C	Zinc	NPW	MN	

EPA 6010D (Rev 2014)

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010D (Rev 2014)	Aluminum	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Aluminum	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Antimony	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Antimony	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Arsenic	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Arsenic	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Barium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Barium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Beryllium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Beryllium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Boron	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Boron	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Cadmium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Cadmium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Calcium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Calcium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Chromium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Chromium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Cobalt	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Cobalt	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Copper	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Copper	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Iron	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Iron	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Lead	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6010D (Rev 2014)	Lead	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Magnesium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Magnesium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Manganese	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Manganese	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Molybdenum	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Molybdenum	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Nickel	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Nickel	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Potassium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Potassium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Selenium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Selenium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Silver	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Silver	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Sodium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Sodium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Thallium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Thallium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Tin	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Tin	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Titanium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Titanium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Vanadium	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Vanadium	NPW	MN	
RCRP	EPA 6010D (Rev 2014)	Zinc	SCM	MN	
RCRP	EPA 6010D (Rev 2014)	Zinc	NPW	MN	

EPA 6020

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Aluminum	SCM	MN	
RCRP	EPA 6020	Aluminum	NPW	MN	
RCRP	EPA 6020	Antimony	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Antimony	SCM	MN	
RCRP	EPA 6020	Arsenic	SCM	MN	
RCRP	EPA 6020	Arsenic	NPW	MN	
RCRP	EPA 6020	Barium	SCM	MN	
RCRP	EPA 6020	Barium	NPW	MN	
RCRP	EPA 6020	Beryllium	SCM	MN	
RCRP	EPA 6020	Beryllium	NPW	MN	
RCRP	EPA 6020	Bismuth	SCM	MN	
RCRP	EPA 6020	Bismuth	NPW	MN	
RCRP	EPA 6020	Boron	NPW	MN	
RCRP	EPA 6020	Boron	SCM	MN	
RCRP	EPA 6020	Cadmium	SCM	MN	
RCRP	EPA 6020	Cadmium	NPW	MN	
RCRP	EPA 6020	Calcium	NPW	MN	
RCRP	EPA 6020	Calcium	SCM	MN	
RCRP	EPA 6020	Chromium	NPW	MN	
RCRP	EPA 6020	Chromium	SCM	MN	
RCRP	EPA 6020	Cobalt	SCM	MN	
RCRP	EPA 6020	Cobalt	NPW	MN	
RCRP	EPA 6020	Copper	SCM	MN	
RCRP	EPA 6020	Copper	NPW	MN	
RCRP	EPA 6020	Iron	SCM	MN	
RCRP	EPA 6020	Iron	NPW	MN	
RCRP	EPA 6020	Lead	NPW	MN	
RCRP	EPA 6020	Lead	SCM	MN	
RCRP	EPA 6020	Lithium	SCM	MN	
RCRP	EPA 6020	Lithium	NPW	MN	
RCRP	EPA 6020	Magnesium	NPW	MN	
RCRP	EPA 6020	Magnesium	SCM	MN	
RCRP	EPA 6020	Manganese	NPW	MN	
RCRP	EPA 6020	Manganese	SCM	MN	
RCRP	EPA 6020	Molybdenum	SCM	MN	
RCRP	EPA 6020	Molybdenum	NPW	MN	
RCRP	EPA 6020	Nickel	SCM	MN	
RCRP	EPA 6020	Nickel	NPW	MN	
RCRP	EPA 6020	Palladium	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020	Platinum	NPW	MN	
RCRP	EPA 6020	Potassium	NPW	MN	
RCRP	EPA 6020	Potassium	SCM	MN	
RCRP	EPA 6020	Selenium	SCM	MN	
RCRP	EPA 6020	Selenium	NPW	MN	
RCRP	EPA 6020	Silicon	SCM	MN	
RCRP	EPA 6020	Silicon	NPW	MN	
RCRP	EPA 6020	Silver	NPW	MN	
RCRP	EPA 6020	Silver	SCM	MN	
RCRP	EPA 6020	Sodium	NPW	MN	
RCRP	EPA 6020	Sodium	SCM	MN	
RCRP	EPA 6020	Strontium	SCM	MN	
RCRP	EPA 6020	Strontium	NPW	MN	
RCRP	EPA 6020	Thallium	NPW	MN	
RCRP	EPA 6020	Thallium	SCM	MN	
RCRP	EPA 6020	Tin	NPW	MN	
RCRP	EPA 6020	Tin	SCM	MN	
RCRP	EPA 6020	Titanium	NPW	MN	
RCRP	EPA 6020	Titanium	SCM	MN	
RCRP	EPA 6020	Total chromium	NPW	MN	
RCRP	EPA 6020	Uranium	SCM	MN	
RCRP	EPA 6020	Uranium	NPW	MN	
RCRP	EPA 6020	Vanadium	NPW	MN	
RCRP	EPA 6020	Vanadium	SCM	MN	
RCRP	EPA 6020	Zinc	NPW	MN	
RCRP	EPA 6020	Zinc	SCM	MN	

EPA 6020A

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020A	Aluminum	SCM	MN	
RCRP	EPA 6020A	Aluminum	NPW	MN	
RCRP	EPA 6020A	Antimony	SCM	MN	
RCRP	EPA 6020A	Antimony	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020A	Arsenic	SCM	MN	
RCRP	EPA 6020A	Arsenic	NPW	MN	
RCRP	EPA 6020A	Barium	NPW	MN	
RCRP	EPA 6020A	Barium	SCM	MN	
RCRP	EPA 6020A	Beryllium	SCM	MN	
RCRP	EPA 6020A	Beryllium	NPW	MN	
RCRP	EPA 6020A	Boron	SCM	MN	
RCRP	EPA 6020A	Boron	NPW	MN	
RCRP	EPA 6020A	Cadmium	NPW	MN	
RCRP	EPA 6020A	Cadmium	SCM	MN	
RCRP	EPA 6020A	Chromium	NPW	MN	
RCRP	EPA 6020A	Chromium	SCM	MN	
RCRP	EPA 6020A	Cobalt	NPW	MN	
RCRP	EPA 6020A	Cobalt	SCM	MN	
RCRP	EPA 6020A	Copper	NPW	MN	
RCRP	EPA 6020A	Copper	SCM	MN	
RCRP	EPA 6020A	Lead	SCM	MN	
RCRP	EPA 6020A	Lead	NPW	MN	
RCRP	EPA 6020A	Manganese	SCM	MN	
RCRP	EPA 6020A	Manganese	NPW	MN	
RCRP	EPA 6020A	Molybdenum	NPW	MN	
RCRP	EPA 6020A	Molybdenum	SCM	MN	
RCRP	EPA 6020A	Nickel	SCM	MN	
RCRP	EPA 6020A	Nickel	NPW	MN	
RCRP	EPA 6020A	Selenium	NPW	MN	
RCRP	EPA 6020A	Selenium	SCM	MN	
RCRP	EPA 6020A	Silver	NPW	MN	
RCRP	EPA 6020A	Silver	SCM	MN	
RCRP	EPA 6020A	Strontium	NPW	MN	
RCRP	EPA 6020A	Strontium	SCM	MN	
RCRP	EPA 6020A	Thallium	NPW	MN	
RCRP	EPA 6020A	Thallium	SCM	MN	
RCRP	EPA 6020A	Tin	NPW	MN	
RCRP	EPA 6020A	Tin	SCM	MN	
RCRP	EPA 6020A	Vanadium	NPW	MN	
RCRP	EPA 6020A	Vanadium	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020A	Zinc	SCM	MN	
RCRP	EPA 6020A	Zinc	NPW	MN	

EPA 6020A

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020A	Bismuth	SCM	MN	
RCRP	EPA 6020A	Bismuth	NPW	MN	
RCRP	EPA 6020A	Calcium	SCM	MN	
RCRP	EPA 6020A	Calcium	NPW	MN	
RCRP	EPA 6020A	Iron	NPW	MN	
RCRP	EPA 6020A	Iron	SCM	MN	
RCRP	EPA 6020A	Lithium	SCM	MN	
RCRP	EPA 6020A	Lithium	NPW	MN	
RCRP	EPA 6020A	Magnesium	SCM	MN	
RCRP	EPA 6020A	Magnesium	NPW	MN	
RCRP	EPA 6020A	Palladium	NPW	MN	
RCRP	EPA 6020A	Platinum	NPW	MN	
RCRP	EPA 6020A	Potassium	SCM	MN	
RCRP	EPA 6020A	Potassium	NPW	MN	
RCRP	EPA 6020A	Silicon	SCM	MN	
RCRP	EPA 6020A	Silicon	NPW	MN	
RCRP	EPA 6020A	Sodium	NPW	MN	
RCRP	EPA 6020A	Sodium	SCM	MN	
RCRP	EPA 6020A	Titanium	SCM	MN	
RCRP	EPA 6020A	Titanium	NPW	MN	
RCRP	EPA 6020A	Uranium	SCM	MN	
RCRP	EPA 6020A	Uranium	NPW	MN	

EPA 6020B (Rev 2014)

Preparation Techniques: Extraction, EPA 1311 TCLP, non-volatiles; Digestion, hotplate or HotBlock; Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020B (Rev 2014)	Aluminum	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Aluminum	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Antimony	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Antimony	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Arsenic	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Arsenic	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Barium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Barium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Beryllium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Beryllium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Bismuth	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Bismuth	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Boron	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Boron	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Cadmium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Cadmium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Calcium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Calcium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Chromium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Chromium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Cobalt	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Cobalt	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Copper	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Copper	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Iron	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Iron	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Lead	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Lead	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Lithium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Lithium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Magnesium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Magnesium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Manganese	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Manganese	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Molybdenum	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Molybdenum	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 6020B (Rev 2014)	Nickel	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Nickel	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Palladium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Platinum	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Potassium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Potassium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Selenium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Selenium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Silicon	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Silicon	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Silver	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Silver	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Sodium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Sodium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Strontium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Strontium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Thallium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Thallium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Tin	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Tin	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Titanium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Titanium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Uranium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Uranium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Vanadium	SCM	MN	
RCRP	EPA 6020B (Rev 2014)	Vanadium	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Zinc	NPW	MN	
RCRP	EPA 6020B (Rev 2014)	Zinc	SCM	MN	

EPA 7470A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7470A	Mercury	NPW	MN	
RCRP	EPA 7470A	Mercury	SCM	MN	User Defined S-MN-I-490 Rev. 02

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7470A	Mercury	SCM	MN	User Defined S-MN-I-306 Rev. 02

EPA 7471A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7471A	Mercury	SCM	MN	

EPA 7471B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7471B	Mercury	SCM	MN	

EPA 7471B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 7471B	Mercury	SCM	MN	User Defined S-MN-I-490 Rev. 02
RCRP	EPA 7471B	Mercury	SCM	MN	User Defined S-MN-I-306 Rev. 02

EPA 1613B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, automated soxhlet; Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	SCM	MN	
RCRP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	SCM	MN	
RCRP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	SCM	MN	
RCRP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	TISSUE	MN	
RCRP	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	SCM	MN	
RCRP	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	SCM	MN	
RCRP	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran	SCM	MN	
RCRP	EPA 1613B	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1613B	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	TISSUE	MN	
RCRP	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran	SCM	MN	
RCRP	EPA 1613B	Total HpCDD	SCM	MN	
RCRP	EPA 1613B	Total HpCDD	TISSUE	MN	
RCRP	EPA 1613B	Total HpCDF	TISSUE	MN	
RCRP	EPA 1613B	Total HpCDF	SCM	MN	
RCRP	EPA 1613B	Total HxCDD	TISSUE	MN	
RCRP	EPA 1613B	Total HxCDD	SCM	MN	
RCRP	EPA 1613B	Total HxCDF	SCM	MN	
RCRP	EPA 1613B	Total HxCDF	TISSUE	MN	
RCRP	EPA 1613B	Total PeCDD	SCM	MN	
RCRP	EPA 1613B	Total PeCDD	TISSUE	MN	
RCRP	EPA 1613B	Total PeCDF	SCM	MN	
RCRP	EPA 1613B	Total PeCDF	TISSUE	MN	
RCRP	EPA 1613B	Total TCDD	SCM	MN	
RCRP	EPA 1613B	Total TCDD	TISSUE	MN	
RCRP	EPA 1613B	Total TCDF	SCM	MN	
RCRP	EPA 1613B	Total TCDF	TISSUE	MN	

EPA 1668A

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	SCM	MN	
RCRP	EPA 1668A	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	SCM	MN	
RCRP	EPA 1668A	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	SCM	MN	
RCRP	EPA 1668A	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	SCM	MN	
RCRP	EPA 1668A	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	SCM	MN	
RCRP	EPA 1668A	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	SCM	MN	
RCRP	EPA 1668A	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	SCM	MN	
RCRP	EPA 1668A	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	TISSUE	MN	
RCRP	EPA 1668A	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	SCM	MN	
RCRP	EPA 1668A	2,2',3-Trichlorobiphenyl (BZ-16)	SCM	MN	
RCRP	EPA 1668A	2,2',3-Trichlorobiphenyl (BZ-16)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	SCM	MN	
RCRP	EPA 1668A	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	SCM	MN	
RCRP	EPA 1668A	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	SCM	MN	
RCRP	EPA 1668A	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	SCM	MN	
RCRP	EPA 1668A	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	SCM	MN	
RCRP	EPA 1668A	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	SCM	MN	
RCRP	EPA 1668A	2,2',4-Trichlorobiphenyl (BZ-17)	TISSUE	MN	
RCRP	EPA 1668A	2,2',4-Trichlorobiphenyl (BZ-17)	SCM	MN	
RCRP	EPA 1668A	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	SCM	MN	
RCRP	EPA 1668A	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	TISSUE	MN	
RCRP	EPA 1668A	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	TISSUE	MN	
RCRP	EPA 1668A	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	SCM	MN	
RCRP	EPA 1668A	2,2',6-Trichlorobiphenyl (BZ-19)	SCM	MN	
RCRP	EPA 1668A	2,2',6-Trichlorobiphenyl (BZ-19)	TISSUE	MN	
RCRP	EPA 1668A	2,2'-Dichlorobiphenyl (BZ-4)	TISSUE	MN	
RCRP	EPA 1668A	2,2'-Dichlorobiphenyl (BZ-4)	SCM	MN	
RCRP	EPA 1668A	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	SCM	MN	
RCRP	EPA 1668A	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	SCM	MN	
RCRP	EPA 1668A	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	SCM	MN	
RCRP	EPA 1668A	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	SCM	MN	
RCRP	EPA 1668A	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	SCM	MN	
RCRP	EPA 1668A	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	SCM	MN	
RCRP	EPA 1668A	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	SCM	MN	
RCRP	EPA 1668A	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	TISSUE	MN	
RCRP	EPA 1668A	2,3',4-Trichlorobiphenyl (BZ-25)	SCM	MN	
RCRP	EPA 1668A	2,3',4-Trichlorobiphenyl (BZ-25)	TISSUE	MN	
RCRP	EPA 1668A	2,3',5'-Trichlorobiphenyl (BZ-34)	SCM	MN	
RCRP	EPA 1668A	2,3',5'-Trichlorobiphenyl (BZ-34)	TISSUE	MN	
RCRP	EPA 1668A	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	TISSUE	MN	
RCRP	EPA 1668A	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	SCM	MN	
RCRP	EPA 1668A	2,3',6-Trichlorobiphenyl (BZ-27)	TISSUE	MN	
RCRP	EPA 1668A	2,3',6-Trichlorobiphenyl (BZ-27)	SCM	MN	
RCRP	EPA 1668A	2,3'-Dichlorobiphenyl (BZ-6)	SCM	MN	
RCRP	EPA 1668A	2,3'-Dichlorobiphenyl (BZ-6)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	SCM	MN	
RCRP	EPA 1668A	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	SCM	MN	
RCRP	EPA 1668A	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	SCM	MN	
RCRP	EPA 1668A	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	SCM	MN	
RCRP	EPA 1668A	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	TISSUE	MN	
RCRP	EPA 1668A	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	SCM	MN	
RCRP	EPA 1668A	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	SCM	MN	
RCRP	EPA 1668A	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	TISSUE	MN	
RCRP	EPA 1668A	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	TISSUE	MN	
RCRP	EPA 1668A	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	SCM	MN	
RCRP	EPA 1668A	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	TISSUE	MN	
RCRP	EPA 1668A	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	SCM	MN	
RCRP	EPA 1668A	2,3,4'-Trichlorobiphenyl (BZ-22)	TISSUE	MN	
RCRP	EPA 1668A	2,3,4'-Trichlorobiphenyl (BZ-22)	SCM	MN	
RCRP	EPA 1668A	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	TISSUE	MN	
RCRP	EPA 1668A	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	SCM	MN	
RCRP	EPA 1668A	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	SCM	MN	
RCRP	EPA 1668A	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	TISSUE	MN	
RCRP	EPA 1668A	2,3,5-Trichlorobiphenyl (BZ-23)	SCM	MN	
RCRP	EPA 1668A	2,3,5-Trichlorobiphenyl (BZ-23)	TISSUE	MN	
RCRP	EPA 1668A	2,3,6-Trichlorobiphenyl (BZ-24)	TISSUE	MN	
RCRP	EPA 1668A	2,3,6-Trichlorobiphenyl (BZ-24)	SCM	MN	
RCRP	EPA 1668A	2,3-Dichlorobiphenyl (BZ-5)	TISSUE	MN	
RCRP	EPA 1668A	2,3-Dichlorobiphenyl (BZ-5)	SCM	MN	
RCRP	EPA 1668A	2,4',5-Trichlorobiphenyl (BZ-31)	TISSUE	MN	
RCRP	EPA 1668A	2,4',5-Trichlorobiphenyl (BZ-31)	SCM	MN	
RCRP	EPA 1668A	2,4',6-Trichlorobiphenyl (BZ-32)	SCM	MN	
RCRP	EPA 1668A	2,4',6-Trichlorobiphenyl (BZ-32)	TISSUE	MN	
RCRP	EPA 1668A	2,4'-Dichlorobiphenyl (BZ-8)	TISSUE	MN	
RCRP	EPA 1668A	2,4'-Dichlorobiphenyl (BZ-8)	SCM	MN	
RCRP	EPA 1668A	2,4-Dichlorobiphenyl (BZ-7)	TISSUE	MN	
RCRP	EPA 1668A	2,4-Dichlorobiphenyl (BZ-7)	SCM	MN	
RCRP	EPA 1668A	2,5-Dichlorobiphenyl (BZ-9)	TISSUE	MN	
RCRP	EPA 1668A	2,5-Dichlorobiphenyl (BZ-9)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	2,6-Dichlorobiphenyl (BZ-10)	SCM	MN	
RCRP	EPA 1668A	2,6-Dichlorobiphenyl (BZ-10)	TISSUE	MN	
RCRP	EPA 1668A	2-Chlorobiphenyl (BZ-1)	SCM	MN	
RCRP	EPA 1668A	2-Chlorobiphenyl (BZ-1)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	SCM	MN	
RCRP	EPA 1668A	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	SCM	MN	
RCRP	EPA 1668A	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	SCM	MN	
RCRP	EPA 1668A	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	SCM	MN	
RCRP	EPA 1668A	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	SCM	MN	
RCRP	EPA 1668A	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	SCM	MN	
RCRP	EPA 1668A	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	TISSUE	MN	
RCRP	EPA 1668A	3,3',4-Trichlorobiphenyl (BZ-35)	SCM	MN	
RCRP	EPA 1668A	3,3',4-Trichlorobiphenyl (BZ-35)	TISSUE	MN	
RCRP	EPA 1668A	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	TISSUE	MN	
RCRP	EPA 1668A	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	SCM	MN	
RCRP	EPA 1668A	3,3',5-Trichlorobiphenyl (BZ-36)	TISSUE	MN	
RCRP	EPA 1668A	3,3',5-Trichlorobiphenyl (BZ-36)	SCM	MN	
RCRP	EPA 1668A	3,3'-Dichlorobiphenyl (BZ-11)	SCM	MN	
RCRP	EPA 1668A	3,3'-Dichlorobiphenyl (BZ-11)	TISSUE	MN	
RCRP	EPA 1668A	3,4',5-Trichlorobiphenyl (BZ-39)	SCM	MN	
RCRP	EPA 1668A	3,4',5-Trichlorobiphenyl (BZ-39)	TISSUE	MN	
RCRP	EPA 1668A	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	SCM	MN	
RCRP	EPA 1668A	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	TISSUE	MN	
RCRP	EPA 1668A	3,4,4'-Trichlorobiphenyl (BZ-37)	TISSUE	MN	
RCRP	EPA 1668A	3,4,4'-Trichlorobiphenyl (BZ-37)	SCM	MN	
RCRP	EPA 1668A	3,4,5-Trichlorobiphenyl (BZ-38)	TISSUE	MN	
RCRP	EPA 1668A	3,4,5-Trichlorobiphenyl (BZ-38)	SCM	MN	
RCRP	EPA 1668A	3,5-Dichlorobiphenyl (BZ-14)	SCM	MN	
RCRP	EPA 1668A	3,5-Dichlorobiphenyl (BZ-14)	TISSUE	MN	
RCRP	EPA 1668A	3-Chlorobiphenyl (BZ-2)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	3-Chlorobiphenyl (BZ-2)	SCM	MN	
RCRP	EPA 1668A	4,4'-Dichlorobiphenyl (BZ-15)	SCM	MN	
RCRP	EPA 1668A	4,4'-Dichlorobiphenyl (BZ-15)	TISSUE	MN	
RCRP	EPA 1668A	4-Chlorobiphenyl (BZ-3)	TISSUE	MN	
RCRP	EPA 1668A	4-Chlorobiphenyl (BZ-3)	SCM	MN	
RCRP	EPA 1668A	Decachlorobiphenyl (BZ-209)	TISSUE	MN	
RCRP	EPA 1668A	Decachlorobiphenyl (BZ-209)	SCM	MN	
RCRP	EPA 1668A	PCB-(100/93/102/98)	SCM	MN	
RCRP	EPA 1668A	PCB-(100/93/102/98)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(107/124)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(107/124)	SCM	MN	
RCRP	EPA 1668A	PCB-(108/119/86/97/125/87)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(108/119/86/97/125/87)	SCM	MN	
RCRP	EPA 1668A	PCB-(110/115)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(110/115)	SCM	MN	
RCRP	EPA 1668A	PCB-(113/90/101)	SCM	MN	
RCRP	EPA 1668A	PCB-(113/90/101)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(117/116/85)	SCM	MN	
RCRP	EPA 1668A	PCB-(117/116/85)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(128/166)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(128/166)	SCM	MN	
RCRP	EPA 1668A	PCB-(13/12)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(13/12)	SCM	MN	
RCRP	EPA 1668A	PCB-(134/143)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(134/143)	SCM	MN	
RCRP	EPA 1668A	PCB-(138/163/129)	SCM	MN	
RCRP	EPA 1668A	PCB-(138/163/129)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(139/140)	SCM	MN	
RCRP	EPA 1668A	PCB-(139/140)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(147/149)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(147/149)	SCM	MN	
RCRP	EPA 1668A	PCB-(151/135)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(151/135)	SCM	MN	
RCRP	EPA 1668A	PCB-(153/168)	SCM	MN	
RCRP	EPA 1668A	PCB-(153/168)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(156/157)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	PCB-(156/157)	SCM	MN	
RCRP	EPA 1668A	PCB-(171/173)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(171/173)	SCM	MN	
RCRP	EPA 1668A	PCB-(180/193)	SCM	MN	
RCRP	EPA 1668A	PCB-(180/193)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(183/185)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(183/185)	SCM	MN	
RCRP	EPA 1668A	PCB-(197/200)	SCM	MN	
RCRP	EPA 1668A	PCB-(197/200)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(198/199)	SCM	MN	
RCRP	EPA 1668A	PCB-(198/199)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(21/33)	SCM	MN	
RCRP	EPA 1668A	PCB-(21/33)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(26/29)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(26/29)	SCM	MN	
RCRP	EPA 1668A	PCB-(28/20)	SCM	MN	
RCRP	EPA 1668A	PCB-(28/20)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(30/18)	SCM	MN	
RCRP	EPA 1668A	PCB-(30/18)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(41/40/71)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(41/40/71)	SCM	MN	
RCRP	EPA 1668A	PCB-(44/47/65)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(44/47/65)	SCM	MN	
RCRP	EPA 1668A	PCB-(45/51)	SCM	MN	
RCRP	EPA 1668A	PCB-(45/51)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(50/53)	SCM	MN	
RCRP	EPA 1668A	PCB-(50/53)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(59/62/75)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(59/62/75)	SCM	MN	
RCRP	EPA 1668A	PCB-(61/70/74/76)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(61/70/74/76)	SCM	MN	
RCRP	EPA 1668A	PCB-(69/49)	SCM	MN	
RCRP	EPA 1668A	PCB-(69/49)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(73/43)	TISSUE	MN	
RCRP	EPA 1668A	PCB-(73/43)	SCM	MN	
RCRP	EPA 1668A	PCB-(88/91)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668A	PCB-(88/91)	TISSUE	MN	

EPA 1668C

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ-196)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ-207)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ-175)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5'-Hexachlorobiphenyl (BZ-130)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ-208)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ-172)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ-176)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4,6-Hexachlorobiphenyl (BZ-131)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',4-Pentachlorobiphenyl (BZ-82)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ-202)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ-178)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',5,5'-Hexachlorobiphenyl (BZ-133)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ-179)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',5-Pentachlorobiphenyl (BZ-83)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,3',6,6'-Hexachlorobiphenyl (BZ-136)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	SCM	MN	
RCRP	EPA 1668C	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	NPW	MN	
RCRP	EPA 1668C	2,2',3,3',6-Pentachlorobiphenyl (BZ-84)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ-146)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4',5,6'-Hexachlorobiphenyl (BZ-148)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ-188)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4',6,6'-Hexachlorobiphenyl (BZ-150)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4'-Tetrachlorobiphenyl (BZ-42)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ-182)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ-204)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ-181)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',5-Hexachlorobiphenyl (BZ-137)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,5',6-Hexachlorobiphenyl (BZ-144)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ-186)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,5,6-Hexachlorobiphenyl (BZ-142)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	NPW	MN	
RCRP	EPA 1668C	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,6'-Pentachlorobiphenyl (BZ-89)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	SCM	MN	
RCRP	EPA 1668C	2,2',3,4,6,6'-Hexachlorobiphenyl (BZ-145)	NPW	MN	
RCRP	EPA 1668C	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	SCM	MN	
RCRP	EPA 1668C	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	SCM	MN	
RCRP	EPA 1668C	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	NPW	MN	
RCRP	EPA 1668C	2,2',3,5,5'-Pentachlorobiphenyl (BZ-92)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	NPW	MN	
RCRP	EPA 1668C	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,5,6'-Pentachlorobiphenyl (BZ-94)	SCM	MN	
RCRP	EPA 1668C	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	NPW	MN	
RCRP	EPA 1668C	2,2',3,5,6,6'-Hexachlorobiphenyl (BZ-152)	SCM	MN	
RCRP	EPA 1668C	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	NPW	MN	
RCRP	EPA 1668C	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,6'-Tetrachlorobiphenyl (BZ-46)	SCM	MN	
RCRP	EPA 1668C	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	SCM	MN	
RCRP	EPA 1668C	2,2',3,6,6'-Pentachlorobiphenyl (BZ-96)	NPW	MN	
RCRP	EPA 1668C	2,2',3-Trichlorobiphenyl (BZ-16)	TISSUE	MN	
RCRP	EPA 1668C	2,2',3-Trichlorobiphenyl (BZ-16)	SCM	MN	
RCRP	EPA 1668C	2,2',3-Trichlorobiphenyl (BZ-16)	NPW	MN	
RCRP	EPA 1668C	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	NPW	MN	
RCRP	EPA 1668C	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)	SCM	MN	
RCRP	EPA 1668C	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	NPW	MN	
RCRP	EPA 1668C	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)	SCM	MN	
RCRP	EPA 1668C	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	NPW	MN	
RCRP	EPA 1668C	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,4',6,6'-Hexachlorobiphenyl (BZ-155)	SCM	MN	
RCRP	EPA 1668C	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	NPW	MN	
RCRP	EPA 1668C	2,2',4,5',6-Pentachlorobiphenyl (BZ-103)	SCM	MN	
RCRP	EPA 1668C	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	NPW	MN	
RCRP	EPA 1668C	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,2',4,5-Tetrachlorobiphenyl (BZ-48)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	SCM	MN	
RCRP	EPA 1668C	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	TISSUE	MN	
RCRP	EPA 1668C	2,2',4,6,6'-Pentachlorobiphenyl (BZ-104)	NPW	MN	
RCRP	EPA 1668C	2,2',4-Trichlorobiphenyl (BZ-17)	SCM	MN	
RCRP	EPA 1668C	2,2',4-Trichlorobiphenyl (BZ-17)	NPW	MN	
RCRP	EPA 1668C	2,2',4-Trichlorobiphenyl (BZ-17)	TISSUE	MN	
RCRP	EPA 1668C	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	TISSUE	MN	
RCRP	EPA 1668C	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	NPW	MN	
RCRP	EPA 1668C	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)	SCM	MN	
RCRP	EPA 1668C	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	NPW	MN	
RCRP	EPA 1668C	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	SCM	MN	
RCRP	EPA 1668C	2,2',6,6'-Tetrachlorobiphenyl (BZ-54)	TISSUE	MN	
RCRP	EPA 1668C	2,2',6-Trichlorobiphenyl (BZ-19)	NPW	MN	
RCRP	EPA 1668C	2,2',6-Trichlorobiphenyl (BZ-19)	TISSUE	MN	
RCRP	EPA 1668C	2,2',6-Trichlorobiphenyl (BZ-19)	SCM	MN	
RCRP	EPA 1668C	2,2'-Dichlorobiphenyl (BZ-4)	SCM	MN	
RCRP	EPA 1668C	2,2'-Dichlorobiphenyl (BZ-4)	TISSUE	MN	
RCRP	EPA 1668C	2,2'-Dichlorobiphenyl (BZ-4)	NPW	MN	
RCRP	EPA 1668C	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	SCM	MN	
RCRP	EPA 1668C	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)	NPW	MN	
RCRP	EPA 1668C	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	SCM	MN	
RCRP	EPA 1668C	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)	NPW	MN	
RCRP	EPA 1668C	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	SCM	MN	
RCRP	EPA 1668C	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)	NPW	MN	
RCRP	EPA 1668C	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	SCM	MN	
RCRP	EPA 1668C	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)	NPW	MN	
RCRP	EPA 1668C	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	NPW	MN	
RCRP	EPA 1668C	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,3',4,5',6-Pentachlorobiphenyl (BZ-121)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	NPW	MN	
RCRP	EPA 1668C	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	SCM	MN	
RCRP	EPA 1668C	2,3',4,5'-Tetrachlorobiphenyl (BZ-68)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	NPW	MN	
RCRP	EPA 1668C	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,5,5'-Pentachlorobiphenyl (BZ-120)	SCM	MN	
RCRP	EPA 1668C	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	TISSUE	MN	
RCRP	EPA 1668C	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	NPW	MN	
RCRP	EPA 1668C	2,3',4,5-Tetrachlorobiphenyl (BZ-67)	SCM	MN	
RCRP	EPA 1668C	2,3',4-Trichlorobiphenyl (BZ-25)	NPW	MN	
RCRP	EPA 1668C	2,3',4-Trichlorobiphenyl (BZ-25)	SCM	MN	
RCRP	EPA 1668C	2,3',4-Trichlorobiphenyl (BZ-25)	TISSUE	MN	
RCRP	EPA 1668C	2,3',5',6-Tetrachlorobiphenyl (BZ-73)	NPW	MN	
RCRP	EPA 1668C	2,3',5',6-Tetrachlorobiphenyl (BZ-73)	SCM	MN	
RCRP	EPA 1668C	2,3',5',6-Tetrachlorobiphenyl (BZ-73)	TISSUE	MN	
RCRP	EPA 1668C	2,3',5'-Trichlorobiphenyl (BZ-34)	NPW	MN	
RCRP	EPA 1668C	2,3',5'-Trichlorobiphenyl (BZ-34)	SCM	MN	
RCRP	EPA 1668C	2,3',5'-Trichlorobiphenyl (BZ-34)	TISSUE	MN	
RCRP	EPA 1668C	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	TISSUE	MN	
RCRP	EPA 1668C	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	NPW	MN	
RCRP	EPA 1668C	2,3',5,5'-Tetrachlorobiphenyl (BZ-72)	SCM	MN	
RCRP	EPA 1668C	2,3',6-Trichlorobiphenyl (BZ-27)	NPW	MN	
RCRP	EPA 1668C	2,3',6-Trichlorobiphenyl (BZ-27)	SCM	MN	
RCRP	EPA 1668C	2,3',6-Trichlorobiphenyl (BZ-27)	TISSUE	MN	
RCRP	EPA 1668C	2,3'-Dichlorobiphenyl (BZ-6)	NPW	MN	
RCRP	EPA 1668C	2,3'-Dichlorobiphenyl (BZ-6)	TISSUE	MN	
RCRP	EPA 1668C	2,3'-Dichlorobiphenyl (BZ-6)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4',5',6-Hexachlorobiphenyl (BZ-164)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,3,3',4',5'-Pentachlorobiphenyl (BZ-122)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4',5,5'-Hexachlorobiphenyl (BZ-162)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ-191)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ-205)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ-190)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,3,3',4,5',6-Hexachlorobiphenyl (BZ-161)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ-192)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,5,5'-Hexachlorobiphenyl (BZ-159)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,5,6-Hexachlorobiphenyl (BZ-160)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4,5-Pentachlorobiphenyl (BZ-106)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	NPW	MN	
RCRP	EPA 1668C	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4,6-Pentachlorobiphenyl (BZ-109)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	SCM	MN	
RCRP	EPA 1668C	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',4-Tetrachlorobiphenyl (BZ-55)	NPW	MN	
RCRP	EPA 1668C	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	SCM	MN	
RCRP	EPA 1668C	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',5'-Tetrachlorobiphenyl (BZ-58)	NPW	MN	
RCRP	EPA 1668C	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	NPW	MN	
RCRP	EPA 1668C	2,3,3',5,5',6-Hexachlorobiphenyl (BZ-165)	SCM	MN	
RCRP	EPA 1668C	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	NPW	MN	
RCRP	EPA 1668C	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',5,5'-Pentachlorobiphenyl (BZ-111)	SCM	MN	
RCRP	EPA 1668C	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,3,3',5,6-Pentachlorobiphenyl (BZ-112)	NPW	MN	
RCRP	EPA 1668C	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	TISSUE	MN	
RCRP	EPA 1668C	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	SCM	MN	
RCRP	EPA 1668C	2,3,3',5-Tetrachlorobiphenyl (BZ-57)	NPW	MN	
RCRP	EPA 1668C	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	SCM	MN	
RCRP	EPA 1668C	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	NPW	MN	
RCRP	EPA 1668C	2,3,4',5-Tetrachlorobiphenyl (BZ-63)	TISSUE	MN	
RCRP	EPA 1668C	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	TISSUE	MN	
RCRP	EPA 1668C	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	NPW	MN	
RCRP	EPA 1668C	2,3,4',6-Tetrachlorobiphenyl (BZ-64)	SCM	MN	
RCRP	EPA 1668C	2,3,4'-Trichlorobiphenyl (BZ-22)	SCM	MN	
RCRP	EPA 1668C	2,3,4'-Trichlorobiphenyl (BZ-22)	TISSUE	MN	
RCRP	EPA 1668C	2,3,4'-Trichlorobiphenyl (BZ-22)	NPW	MN	
RCRP	EPA 1668C	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	TISSUE	MN	
RCRP	EPA 1668C	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	NPW	MN	
RCRP	EPA 1668C	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)	SCM	MN	
RCRP	EPA 1668C	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	NPW	MN	
RCRP	EPA 1668C	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	TISSUE	MN	
RCRP	EPA 1668C	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)	SCM	MN	
RCRP	EPA 1668C	2,3,5-Trichlorobiphenyl (BZ-23)	TISSUE	MN	
RCRP	EPA 1668C	2,3,5-Trichlorobiphenyl (BZ-23)	NPW	MN	
RCRP	EPA 1668C	2,3,5-Trichlorobiphenyl (BZ-23)	SCM	MN	
RCRP	EPA 1668C	2,3,6-Trichlorobiphenyl (BZ-24)	TISSUE	MN	
RCRP	EPA 1668C	2,3,6-Trichlorobiphenyl (BZ-24)	NPW	MN	
RCRP	EPA 1668C	2,3,6-Trichlorobiphenyl (BZ-24)	SCM	MN	
RCRP	EPA 1668C	2,3-Dichlorobiphenyl (BZ-5)	TISSUE	MN	
RCRP	EPA 1668C	2,3-Dichlorobiphenyl (BZ-5)	NPW	MN	
RCRP	EPA 1668C	2,3-Dichlorobiphenyl (BZ-5)	SCM	MN	
RCRP	EPA 1668C	2,4',5-Trichlorobiphenyl (BZ-31)	SCM	MN	
RCRP	EPA 1668C	2,4',5-Trichlorobiphenyl (BZ-31)	NPW	MN	
RCRP	EPA 1668C	2,4',5-Trichlorobiphenyl (BZ-31)	TISSUE	MN	
RCRP	EPA 1668C	2,4',6-Trichlorobiphenyl (BZ-32)	TISSUE	MN	
RCRP	EPA 1668C	2,4',6-Trichlorobiphenyl (BZ-32)	SCM	MN	
RCRP	EPA 1668C	2,4',6-Trichlorobiphenyl (BZ-32)	NPW	MN	
RCRP	EPA 1668C	2,4'-Dichlorobiphenyl (BZ-8)	SCM	MN	
RCRP	EPA 1668C	2,4'-Dichlorobiphenyl (BZ-8)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	2,4'-Dichlorobiphenyl (BZ-8)	TISSUE	MN	
RCRP	EPA 1668C	2,4-Dichlorobiphenyl (BZ-7)	TISSUE	MN	
RCRP	EPA 1668C	2,4-Dichlorobiphenyl (BZ-7)	SCM	MN	
RCRP	EPA 1668C	2,4-Dichlorobiphenyl (BZ-7)	NPW	MN	
RCRP	EPA 1668C	2,5-Dichlorobiphenyl (BZ-9)	TISSUE	MN	
RCRP	EPA 1668C	2,5-Dichlorobiphenyl (BZ-9)	NPW	MN	
RCRP	EPA 1668C	2,5-Dichlorobiphenyl (BZ-9)	SCM	MN	
RCRP	EPA 1668C	2,6-Dichlorobiphenyl (BZ-10)	TISSUE	MN	
RCRP	EPA 1668C	2,6-Dichlorobiphenyl (BZ-10)	NPW	MN	
RCRP	EPA 1668C	2,6-Dichlorobiphenyl (BZ-10)	SCM	MN	
RCRP	EPA 1668C	2-Chlorobiphenyl (BZ-1)	NPW	MN	
RCRP	EPA 1668C	2-Chlorobiphenyl (BZ-1)	SCM	MN	
RCRP	EPA 1668C	2-Chlorobiphenyl (BZ-1)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	SCM	MN	
RCRP	EPA 1668C	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)	NPW	MN	
RCRP	EPA 1668C	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	NPW	MN	
RCRP	EPA 1668C	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)	SCM	MN	
RCRP	EPA 1668C	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	SCM	MN	
RCRP	EPA 1668C	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	NPW	MN	
RCRP	EPA 1668C	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	SCM	MN	
RCRP	EPA 1668C	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	NPW	MN	
RCRP	EPA 1668C	3,3',4,5'-Tetrachlorobiphenyl (BZ-79)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	NPW	MN	
RCRP	EPA 1668C	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,5,5'-Pentachlorobiphenyl (BZ-127)	SCM	MN	
RCRP	EPA 1668C	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	SCM	MN	
RCRP	EPA 1668C	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	TISSUE	MN	
RCRP	EPA 1668C	3,3',4,5-Tetrachlorobiphenyl (BZ-78)	NPW	MN	
RCRP	EPA 1668C	3,3',4-Trichlorobiphenyl (BZ-35)	NPW	MN	
RCRP	EPA 1668C	3,3',4-Trichlorobiphenyl (BZ-35)	SCM	MN	
RCRP	EPA 1668C	3,3',4-Trichlorobiphenyl (BZ-35)	TISSUE	MN	
RCRP	EPA 1668C	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	NPW	MN	
RCRP	EPA 1668C	3,3',5,5'-Tetrachlorobiphenyl (BZ-80)	SCM	MN	
RCRP	EPA 1668C	3,3',5-Trichlorobiphenyl (BZ-36)	TISSUE	MN	
RCRP	EPA 1668C	3,3',5-Trichlorobiphenyl (BZ-36)	SCM	MN	
RCRP	EPA 1668C	3,3',5-Trichlorobiphenyl (BZ-36)	NPW	MN	
RCRP	EPA 1668C	3,3'-Dichlorobiphenyl (BZ-11)	TISSUE	MN	
RCRP	EPA 1668C	3,3'-Dichlorobiphenyl (BZ-11)	SCM	MN	
RCRP	EPA 1668C	3,3'-Dichlorobiphenyl (BZ-11)	NPW	MN	
RCRP	EPA 1668C	3,4',5-Trichlorobiphenyl (BZ-39)	TISSUE	MN	
RCRP	EPA 1668C	3,4',5-Trichlorobiphenyl (BZ-39)	SCM	MN	
RCRP	EPA 1668C	3,4',5-Trichlorobiphenyl (BZ-39)	NPW	MN	
RCRP	EPA 1668C	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	NPW	MN	
RCRP	EPA 1668C	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	TISSUE	MN	
RCRP	EPA 1668C	3,4,4',5-Tetrachlorobiphenyl (BZ-81)	SCM	MN	
RCRP	EPA 1668C	3,4,4'-Trichlorobiphenyl (BZ-37)	TISSUE	MN	
RCRP	EPA 1668C	3,4,4'-Trichlorobiphenyl (BZ-37)	NPW	MN	
RCRP	EPA 1668C	3,4,4'-Trichlorobiphenyl (BZ-37)	SCM	MN	
RCRP	EPA 1668C	3,4,5-Trichlorobiphenyl (BZ-38)	SCM	MN	
RCRP	EPA 1668C	3,4,5-Trichlorobiphenyl (BZ-38)	TISSUE	MN	
RCRP	EPA 1668C	3,4,5-Trichlorobiphenyl (BZ-38)	NPW	MN	
RCRP	EPA 1668C	3,5-Dichlorobiphenyl (BZ-14)	NPW	MN	
RCRP	EPA 1668C	3,5-Dichlorobiphenyl (BZ-14)	TISSUE	MN	
RCRP	EPA 1668C	3,5-Dichlorobiphenyl (BZ-14)	SCM	MN	
RCRP	EPA 1668C	3-Chlorobiphenyl (BZ-2)	NPW	MN	
RCRP	EPA 1668C	3-Chlorobiphenyl (BZ-2)	TISSUE	MN	
RCRP	EPA 1668C	3-Chlorobiphenyl (BZ-2)	SCM	MN	
RCRP	EPA 1668C	4,4'-Dichlorobiphenyl (BZ-15)	NPW	MN	
RCRP	EPA 1668C	4,4'-Dichlorobiphenyl (BZ-15)	SCM	MN	
RCRP	EPA 1668C	4,4'-Dichlorobiphenyl (BZ-15)	TISSUE	MN	
RCRP	EPA 1668C	4-Chlorobiphenyl (BZ-3)	SCM	MN	
RCRP	EPA 1668C	4-Chlorobiphenyl (BZ-3)	TISSUE	MN	
RCRP	EPA 1668C	4-Chlorobiphenyl (BZ-3)	NPW	MN	
RCRP	EPA 1668C	Decachlorobiphenyl (BZ-209)	SCM	MN	
RCRP	EPA 1668C	Decachlorobiphenyl (BZ-209)	TISSUE	MN	
RCRP	EPA 1668C	Decachlorobiphenyl (BZ-209)	NPW	MN	
RCRP	EPA 1668C	PCB-(100/93/102/98)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	PCB-(100/93/102/98)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(100/93/102/98)	NPW	MN	
RCRP	EPA 1668C	PCB-(107/124)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(107/124)	NPW	MN	
RCRP	EPA 1668C	PCB-(107/124)	SCM	MN	
RCRP	EPA 1668C	PCB-(108/119/86/97/125/87)	NPW	MN	
RCRP	EPA 1668C	PCB-(108/119/86/97/125/87)	SCM	MN	
RCRP	EPA 1668C	PCB-(108/119/86/97/125/87)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(110/115)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(110/115)	SCM	MN	
RCRP	EPA 1668C	PCB-(110/115)	NPW	MN	
RCRP	EPA 1668C	PCB-(113/90/101)	NPW	MN	
RCRP	EPA 1668C	PCB-(113/90/101)	SCM	MN	
RCRP	EPA 1668C	PCB-(113/90/101)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(117/116/85)	NPW	MN	
RCRP	EPA 1668C	PCB-(117/116/85)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(117/116/85)	SCM	MN	
RCRP	EPA 1668C	PCB-(128/166)	SCM	MN	
RCRP	EPA 1668C	PCB-(128/166)	NPW	MN	
RCRP	EPA 1668C	PCB-(128/166)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(13/12)	NPW	MN	
RCRP	EPA 1668C	PCB-(13/12)	SCM	MN	
RCRP	EPA 1668C	PCB-(13/12)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(134/143)	SCM	MN	
RCRP	EPA 1668C	PCB-(134/143)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(134/143)	NPW	MN	
RCRP	EPA 1668C	PCB-(138/163/129)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(138/163/129)	NPW	MN	
RCRP	EPA 1668C	PCB-(138/163/129)	SCM	MN	
RCRP	EPA 1668C	PCB-(139/140)	SCM	MN	
RCRP	EPA 1668C	PCB-(139/140)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(139/140)	NPW	MN	
RCRP	EPA 1668C	PCB-(147/149)	SCM	MN	
RCRP	EPA 1668C	PCB-(147/149)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(147/149)	NPW	MN	
RCRP	EPA 1668C	PCB-(151/135)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	PCB-(151/135)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(151/135)	SCM	MN	
RCRP	EPA 1668C	PCB-(153/168)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(153/168)	NPW	MN	
RCRP	EPA 1668C	PCB-(153/168)	SCM	MN	
RCRP	EPA 1668C	PCB-(156/157)	SCM	MN	
RCRP	EPA 1668C	PCB-(156/157)	NPW	MN	
RCRP	EPA 1668C	PCB-(156/157)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(171/173)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(171/173)	NPW	MN	
RCRP	EPA 1668C	PCB-(171/173)	SCM	MN	
RCRP	EPA 1668C	PCB-(180/193)	SCM	MN	
RCRP	EPA 1668C	PCB-(180/193)	NPW	MN	
RCRP	EPA 1668C	PCB-(180/193)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(183/185)	SCM	MN	
RCRP	EPA 1668C	PCB-(183/185)	NPW	MN	
RCRP	EPA 1668C	PCB-(183/185)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(197/200)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(197/200)	NPW	MN	
RCRP	EPA 1668C	PCB-(197/200)	SCM	MN	
RCRP	EPA 1668C	PCB-(198/199)	SCM	MN	
RCRP	EPA 1668C	PCB-(198/199)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(198/199)	NPW	MN	
RCRP	EPA 1668C	PCB-(21/33)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(21/33)	SCM	MN	
RCRP	EPA 1668C	PCB-(21/33)	NPW	MN	
RCRP	EPA 1668C	PCB-(26/29)	NPW	MN	
RCRP	EPA 1668C	PCB-(26/29)	SCM	MN	
RCRP	EPA 1668C	PCB-(26/29)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(28/20)	NPW	MN	
RCRP	EPA 1668C	PCB-(28/20)	SCM	MN	
RCRP	EPA 1668C	PCB-(28/20)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(30/18)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(30/18)	SCM	MN	
RCRP	EPA 1668C	PCB-(30/18)	NPW	MN	
RCRP	EPA 1668C	PCB-(41/40/71)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 1668C	PCB-(41/40/71)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(41/40/71)	NPW	MN	
RCRP	EPA 1668C	PCB-(44/47/65)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(44/47/65)	SCM	MN	
RCRP	EPA 1668C	PCB-(44/47/65)	NPW	MN	
RCRP	EPA 1668C	PCB-(45/51)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(45/51)	SCM	MN	
RCRP	EPA 1668C	PCB-(45/51)	NPW	MN	
RCRP	EPA 1668C	PCB-(50/53)	SCM	MN	
RCRP	EPA 1668C	PCB-(50/53)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(50/53)	NPW	MN	
RCRP	EPA 1668C	PCB-(59/62/75)	NPW	MN	
RCRP	EPA 1668C	PCB-(59/62/75)	SCM	MN	
RCRP	EPA 1668C	PCB-(59/62/75)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(61/70/74/76)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(61/70/74/76)	SCM	MN	
RCRP	EPA 1668C	PCB-(61/70/74/76)	NPW	MN	
RCRP	EPA 1668C	PCB-(69/49)	NPW	MN	
RCRP	EPA 1668C	PCB-(69/49)	SCM	MN	
RCRP	EPA 1668C	PCB-(69/49)	TISSUE	MN	
RCRP	EPA 1668C	PCB-(88/91)	SCM	MN	
RCRP	EPA 1668C	PCB-(88/91)	NPW	MN	
RCRP	EPA 1668C	PCB-(88/91)	TISSUE	MN	

EPA 8011

Preparation Techniques: Extraction, continuous liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)	NPW	MN	
RCRP	EPA 8011	1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	MN	

EPA 8081A

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, EPA 1311 TCLP, non-volatiles; Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	4,4'-DDD	SCM	MN	
RCRP	EPA 8081A	4,4'-DDD	NPW	MN	
RCRP	EPA 8081A	4,4'-DDE	NPW	MN	
RCRP	EPA 8081A	4,4'-DDE	SCM	MN	
RCRP	EPA 8081A	4,4'-DDT	NPW	MN	
RCRP	EPA 8081A	4,4'-DDT	SCM	MN	
RCRP	EPA 8081A	Aldrin	NPW	MN	
RCRP	EPA 8081A	Aldrin	SCM	MN	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081A	alpha-BHC (alpha-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081A	alpha-Chlordane	NPW	MN	
RCRP	EPA 8081A	alpha-Chlordane	SCM	MN	
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081A	beta-BHC (beta-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081A	Chlordane (tech.)	NPW	MN	
RCRP	EPA 8081A	Chlordane (tech.)	SCM	MN	
RCRP	EPA 8081A	delta-BHC	NPW	MN	
RCRP	EPA 8081A	delta-BHC	SCM	MN	
RCRP	EPA 8081A	Dieldrin	SCM	MN	
RCRP	EPA 8081A	Dieldrin	NPW	MN	
RCRP	EPA 8081A	Endosulfan I	SCM	MN	
RCRP	EPA 8081A	Endosulfan I	NPW	MN	
RCRP	EPA 8081A	Endosulfan II	NPW	MN	
RCRP	EPA 8081A	Endosulfan II	SCM	MN	
RCRP	EPA 8081A	Endosulfan sulfate	SCM	MN	
RCRP	EPA 8081A	Endosulfan sulfate	NPW	MN	
RCRP	EPA 8081A	Endrin	NPW	MN	
RCRP	EPA 8081A	Endrin	SCM	MN	
RCRP	EPA 8081A	Endrin aldehyde	SCM	MN	
RCRP	EPA 8081A	Endrin aldehyde	NPW	MN	
RCRP	EPA 8081A	Endrin ketone	SCM	MN	
RCRP	EPA 8081A	Endrin ketone	NPW	MN	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	SCM	MN	
RCRP	EPA 8081A	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081A	gamma-Chlordane	SCM	MN	
RCRP	EPA 8081A	gamma-Chlordane	NPW	MN	
RCRP	EPA 8081A	Heptachlor	NPW	MN	
RCRP	EPA 8081A	Heptachlor	SCM	MN	
RCRP	EPA 8081A	Heptachlor epoxide	SCM	MN	
RCRP	EPA 8081A	Heptachlor epoxide	NPW	MN	
RCRP	EPA 8081A	Methoxychlor	SCM	MN	
RCRP	EPA 8081A	Methoxychlor	NPW	MN	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	SCM	MN	
RCRP	EPA 8081A	Toxaphene (Chlorinated camphene)	NPW	MN	

EPA 8081B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, EPA 1311 TCLP, non-volatiles; Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081B	4,4'-DDD	NPW	MN	
RCRP	EPA 8081B	4,4'-DDD	SCM	MN	
RCRP	EPA 8081B	4,4'-DDE	SCM	MN	
RCRP	EPA 8081B	4,4'-DDE	NPW	MN	
RCRP	EPA 8081B	4,4'-DDT	SCM	MN	
RCRP	EPA 8081B	4,4'-DDT	NPW	MN	
RCRP	EPA 8081B	Aldrin	SCM	MN	
RCRP	EPA 8081B	Aldrin	NPW	MN	
RCRP	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081B	alpha-Chlordane	NPW	MN	
RCRP	EPA 8081B	alpha-Chlordane	SCM	MN	
RCRP	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)	SCM	MN	
RCRP	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)	NPW	MN	
RCRP	EPA 8081B	Chlordane (tech.)	NPW	MN	
RCRP	EPA 8081B	Chlordane (tech.)	SCM	MN	
RCRP	EPA 8081B	delta-BHC	SCM	MN	
RCRP	EPA 8081B	delta-BHC	NPW	MN	
RCRP	EPA 8081B	Dieldrin	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8081B	Dieldrin	NPW	MN	
RCRP	EPA 8081B	Endosulfan I	SCM	MN	
RCRP	EPA 8081B	Endosulfan I	NPW	MN	
RCRP	EPA 8081B	Endosulfan II	NPW	MN	
RCRP	EPA 8081B	Endosulfan II	SCM	MN	
RCRP	EPA 8081B	Endosulfan sulfate	NPW	MN	
RCRP	EPA 8081B	Endosulfan sulfate	SCM	MN	
RCRP	EPA 8081B	Endrin	SCM	MN	
RCRP	EPA 8081B	Endrin	NPW	MN	
RCRP	EPA 8081B	Endrin aldehyde	NPW	MN	
RCRP	EPA 8081B	Endrin aldehyde	SCM	MN	
RCRP	EPA 8081B	Endrin ketone	NPW	MN	
RCRP	EPA 8081B	Endrin ketone	SCM	MN	
RCRP	EPA 8081B	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	SCM	MN	
RCRP	EPA 8081B	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	NPW	MN	
RCRP	EPA 8081B	gamma-Chlordane	SCM	MN	
RCRP	EPA 8081B	gamma-Chlordane	NPW	MN	
RCRP	EPA 8081B	Heptachlor	NPW	MN	
RCRP	EPA 8081B	Heptachlor	SCM	MN	
RCRP	EPA 8081B	Heptachlor epoxide	SCM	MN	
RCRP	EPA 8081B	Heptachlor epoxide	NPW	MN	
RCRP	EPA 8081B	Methoxychlor	NPW	MN	
RCRP	EPA 8081B	Methoxychlor	SCM	MN	
RCRP	EPA 8081B	Toxaphene (Chlorinated camphene)	SCM	MN	
RCRP	EPA 8081B	Toxaphene (Chlorinated camphene)	NPW	MN	

EPA 8082

Preparation Techniques: Extraction, microwave; Extraction, separatory funnel liquid-liquid (LLE); Extraction, soxhlet; Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	NPW	MN	
RCRP	EPA 8082	Aroclor-1016 (PCB-1016)	SCM	MN	
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	SCM	MN	
RCRP	EPA 8082	Aroclor-1221 (PCB-1221)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	NPW	MN	
RCRP	EPA 8082	Aroclor-1232 (PCB-1232)	SCM	MN	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	SCM	MN	
RCRP	EPA 8082	Aroclor-1242 (PCB-1242)	NPW	MN	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	SCM	MN	
RCRP	EPA 8082	Aroclor-1248 (PCB-1248)	NPW	MN	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	NPW	MN	
RCRP	EPA 8082	Aroclor-1254 (PCB-1254)	SCM	MN	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	SCM	MN	
RCRP	EPA 8082	Aroclor-1260 (PCB-1260)	NPW	MN	
RCRP	EPA 8082	Aroclor-1262 (PCB-1262)	SCM	MN	
RCRP	EPA 8082	Aroclor-1262 (PCB-1262)	NPW	MN	
RCRP	EPA 8082	Aroclor-1268 (PCB-1268)	NPW	MN	
RCRP	EPA 8082	Aroclor-1268 (PCB-1268)	SCM	MN	
RCRP	EPA 8082	PCBs	SCM	MN	
RCRP	EPA 8082	PCBs	NPW	MN	

EPA 8082A (Rev 2007)

Preparation Techniques: Extraction, microwave; Extraction, separatory funnel liquid-liquid (LLE); Extraction, soxhlet; Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082A (Rev 2007)	Aroclor-1016 (PCB-1016)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1016 (PCB-1016)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1221 (PCB-1221)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1221 (PCB-1221)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1232 (PCB-1232)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1232 (PCB-1232)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1242 (PCB-1242)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1242 (PCB-1242)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1248 (PCB-1248)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1248 (PCB-1248)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1254 (PCB-1254)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1254 (PCB-1254)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1260 (PCB-1260)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1260 (PCB-1260)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8082A (Rev 2007)	Aroclor-1262 (PCB-1262)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1262 (PCB-1262)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1268 (PCB-1268)	SCM	MN	
RCRP	EPA 8082A (Rev 2007)	Aroclor-1268 (PCB-1268)	NPW	MN	
RCRP	EPA 8082A (Rev 2007)	PCBs	SCM	MN	

EPA 8270C

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, EPA 1311 TCLP, non-volatiles; Extraction, soxhlet; Extraction, ultrasonic; Extraction, continuous liquid-liquid (LLE); Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,2,4-Trichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,2-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,2-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	NPW	MN	
RCRP	EPA 8270C	1,2-Diphenylhydrazine	SCM	MN	
RCRP	EPA 8270C	1,3-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1,3-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,4-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270C	1,4-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270C	1-Methylnaphthalene	NPW	MN	
RCRP	EPA 8270C	1-Methylnaphthalene	SCM	MN	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4,5-Trichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4,6-Trichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dichlorophenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dichlorophenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dimethylphenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dimethylphenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dinitrophenol	NPW	MN	
RCRP	EPA 8270C	2,4-Dinitrophenol	SCM	MN	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	NPW	MN	
RCRP	EPA 8270C	2,4-Dinitrotoluene (2,4-DNT)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	NPW	MN	
RCRP	EPA 8270C	2,6-Dinitrotoluene (2,6-DNT)	SCM	MN	
RCRP	EPA 8270C	2-Chloronaphthalene	SCM	MN	
RCRP	EPA 8270C	2-Chloronaphthalene	NPW	MN	
RCRP	EPA 8270C	2-Chlorophenol	SCM	MN	
RCRP	EPA 8270C	2-Chlorophenol	NPW	MN	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	MN	
RCRP	EPA 8270C	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	SCM	MN	
RCRP	EPA 8270C	2-Methylnaphthalene	SCM	MN	
RCRP	EPA 8270C	2-Methylnaphthalene	NPW	MN	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	SCM	MN	
RCRP	EPA 8270C	2-Methylphenol (o-Cresol)	NPW	MN	
RCRP	EPA 8270C	2-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	2-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	2-Nitrophenol	SCM	MN	
RCRP	EPA 8270C	2-Nitrophenol	NPW	MN	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	SCM	MN	
RCRP	EPA 8270C	3,3'-Dichlorobenzidine	NPW	MN	
RCRP	EPA 8270C	3-Methylphenol (m-Cresol)	NPW	MN	
RCRP	EPA 8270C	3-Methylphenol (m-Cresol)	SCM	MN	
RCRP	EPA 8270C	3-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	3-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	NPW	MN	
RCRP	EPA 8270C	4-Bromophenyl phenyl ether	SCM	MN	
RCRP	EPA 8270C	4-Chloro-3-methylphenol	NPW	MN	
RCRP	EPA 8270C	4-Chloro-3-methylphenol	SCM	MN	
RCRP	EPA 8270C	4-Chloroaniline	SCM	MN	
RCRP	EPA 8270C	4-Chloroaniline	NPW	MN	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	NPW	MN	
RCRP	EPA 8270C	4-Chlorophenyl phenylether	SCM	MN	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	NPW	MN	
RCRP	EPA 8270C	4-Methylphenol (p-Cresol)	SCM	MN	
RCRP	EPA 8270C	4-Nitroaniline	SCM	MN	
RCRP	EPA 8270C	4-Nitroaniline	NPW	MN	
RCRP	EPA 8270C	4-Nitrophenol	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	4-Nitrophenol	SCM	MN	
RCRP	EPA 8270C	Acenaphthene	NPW	MN	
RCRP	EPA 8270C	Acenaphthene	SCM	MN	
RCRP	EPA 8270C	Acenaphthylene	SCM	MN	
RCRP	EPA 8270C	Acenaphthylene	NPW	MN	
RCRP	EPA 8270C	Anthracene	NPW	MN	
RCRP	EPA 8270C	Anthracene	SCM	MN	
RCRP	EPA 8270C	Benzidine	SCM	MN	
RCRP	EPA 8270C	Benzidine	NPW	MN	
RCRP	EPA 8270C	Benzo(a)anthracene	NPW	MN	
RCRP	EPA 8270C	Benzo(a)anthracene	SCM	MN	
RCRP	EPA 8270C	Benzo(a)pyrene	NPW	MN	
RCRP	EPA 8270C	Benzo(a)pyrene	SCM	MN	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	SCM	MN	
RCRP	EPA 8270C	Benzo(g,h,i)perylene	NPW	MN	
RCRP	EPA 8270C	Benzo(k)fluoranthene	SCM	MN	
RCRP	EPA 8270C	Benzo(k)fluoranthene	NPW	MN	
RCRP	EPA 8270C	Benzo[b]fluoranthene	SCM	MN	
RCRP	EPA 8270C	Benzo[b]fluoranthene	NPW	MN	
RCRP	EPA 8270C	Benzoic acid	SCM	MN	
RCRP	EPA 8270C	Benzoic acid	NPW	MN	
RCRP	EPA 8270C	Benzyl alcohol	SCM	MN	
RCRP	EPA 8270C	Benzyl alcohol	NPW	MN	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroethoxy)methane	NPW	MN	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroethyl) ether	NPW	MN	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	SCM	MN	
RCRP	EPA 8270C	bis(2-Chloroisopropyl) ether	NPW	MN	
RCRP	EPA 8270C	Butyl benzyl phthalate	SCM	MN	
RCRP	EPA 8270C	Butyl benzyl phthalate	NPW	MN	
RCRP	EPA 8270C	Carbazole	SCM	MN	
RCRP	EPA 8270C	Carbazole	NPW	MN	
RCRP	EPA 8270C	Chrysene	SCM	MN	
RCRP	EPA 8270C	Chrysene	NPW	MN	
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	MN	
RCRP	EPA 8270C	Di-n-butyl phthalate	NPW	MN	
RCRP	EPA 8270C	Di-n-butyl phthalate	SCM	MN	
RCRP	EPA 8270C	Di-n-octyl phthalate	NPW	MN	
RCRP	EPA 8270C	Di-n-octyl phthalate	SCM	MN	
RCRP	EPA 8270C	Dibenz(a,h) anthracene	SCM	MN	
RCRP	EPA 8270C	Dibenz(a,h) anthracene	NPW	MN	
RCRP	EPA 8270C	Dibenzofuran	NPW	MN	
RCRP	EPA 8270C	Dibenzofuran	SCM	MN	
RCRP	EPA 8270C	Diethyl phthalate	NPW	MN	
RCRP	EPA 8270C	Diethyl phthalate	SCM	MN	
RCRP	EPA 8270C	Dimethyl phthalate	SCM	MN	
RCRP	EPA 8270C	Dimethyl phthalate	NPW	MN	
RCRP	EPA 8270C	Fluoranthene	NPW	MN	
RCRP	EPA 8270C	Fluoranthene	SCM	MN	
RCRP	EPA 8270C	Fluorene	NPW	MN	
RCRP	EPA 8270C	Fluorene	SCM	MN	
RCRP	EPA 8270C	Hexachlorobenzene	SCM	MN	
RCRP	EPA 8270C	Hexachlorobenzene	NPW	MN	
RCRP	EPA 8270C	Hexachlorobutadiene	NPW	MN	
RCRP	EPA 8270C	Hexachlorobutadiene	SCM	MN	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	NPW	MN	
RCRP	EPA 8270C	Hexachlorocyclopentadiene	SCM	MN	
RCRP	EPA 8270C	Hexachloroethane	NPW	MN	
RCRP	EPA 8270C	Hexachloroethane	SCM	MN	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	NPW	MN	
RCRP	EPA 8270C	Indeno(1,2,3-cd) pyrene	SCM	MN	
RCRP	EPA 8270C	Isophorone	SCM	MN	
RCRP	EPA 8270C	Isophorone	NPW	MN	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	NPW	MN	
RCRP	EPA 8270C	n-Nitrosodi-n-propylamine	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodimethylamine	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodimethylamine	NPW	MN	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	SCM	MN	
RCRP	EPA 8270C	n-Nitrosodiphenylamine	NPW	MN	
RCRP	EPA 8270C	Naphthalene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270C	Naphthalene	SCM	MN	
RCRP	EPA 8270C	Nitrobenzene	SCM	MN	
RCRP	EPA 8270C	Nitrobenzene	NPW	MN	
RCRP	EPA 8270C	Pentachlorophenol	NPW	MN	
RCRP	EPA 8270C	Pentachlorophenol	SCM	MN	
RCRP	EPA 8270C	Phenanthrene	NPW	MN	
RCRP	EPA 8270C	Phenanthrene	SCM	MN	
RCRP	EPA 8270C	Phenol	SCM	MN	
RCRP	EPA 8270C	Phenol	NPW	MN	
RCRP	EPA 8270C	Pyrene	NPW	MN	
RCRP	EPA 8270C	Pyrene	SCM	MN	
RCRP	EPA 8270C	Pyridine	NPW	MN	
RCRP	EPA 8270C	Pyridine	SCM	MN	

EPA 8270D (Rev 2014)

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, EPA 1311 TCLP, non-volatiles; Extraction, soxhlet; Extraction, ultrasonic; Extraction, continuous liquid-liquid (LLE); Extraction, EPA 1312 SPLP, non-volatiles;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270D (Rev 2014)	1,2,4-Trichlorobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1,2,4-Trichlorobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,2-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1,2-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,2-Diphenylhydrazine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,2-Diphenylhydrazine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1,3-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,3-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1,4-Dichlorobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1,4-Dichlorobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,4-Dioxane (1,4- Diethyleneoxide)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1,4-Dioxane (1,4- Diethyleneoxide)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	1-Methylnaphthalene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	1-Methylnaphthalene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,2'-Oxybis(1-chloropropane),bis(2-Chloro-1-methylethyl)ether	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270D (Rev 2014)	2,4,5-Trichlorophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,4,5-Trichlorophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4,6-Trichlorophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,4,6-Trichlorophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dichlorophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dichlorophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dimethylphenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dimethylphenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dinitrophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dinitrophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dinitrotoluene (2,4-DNT)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,4-Dinitrotoluene (2,4-DNT)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2,6-Dinitrotoluene (2,6-DNT)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2,6-Dinitrotoluene (2,6-DNT)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Chloronaphthalene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Chloronaphthalene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Chlorophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Chlorophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methylnaphthalene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methylnaphthalene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methylphenol (o-Cresol)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Methylphenol (o-Cresol)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Nitroaniline	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Nitroaniline	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	2-Nitrophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	2-Nitrophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	3,3'-Dichlorobenzidine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	3,3'-Dichlorobenzidine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	3-Methylphenol (m-Cresol)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	3-Methylphenol (m-Cresol)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	3-Nitroaniline	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	3-Nitroaniline	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Bromophenyl phenyl ether	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270D (Rev 2014)	4-Bromophenyl phenyl ether	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chloro-3-methylphenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chloro-3-methylphenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chloroaniline	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chloroaniline	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chlorophenyl phenylether	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	4-Chlorophenyl phenylether	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Methylphenol (p-Cresol)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	4-Methylphenol (p-Cresol)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Nitroaniline	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Nitroaniline	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	4-Nitrophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	4-Nitrophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Acenaphthene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Acenaphthene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Acenaphthylene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Acenaphthylene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Anthracene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Anthracene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzidine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzidine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(a)anthracene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(a)anthracene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(a)pyrene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(a)pyrene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(g,h,i)perylene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(g,h,i)perylene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(k)fluoranthene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo(k)fluoranthene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo[b]fluoranthene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzo[b]fluoranthene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzoic acid	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzoic acid	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Benzyl alcohol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Benzyl alcohol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroethoxy)methane	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroethoxy)methane	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroethyl) ether	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroethyl) ether	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroisopropyl) ether	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	bis(2-Chloroisopropyl) ether	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Butyl benzyl phthalate	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Butyl benzyl phthalate	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Carbazole	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Carbazole	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Chrysene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Chrysene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Di-n-butyl phthalate	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Di-n-butyl phthalate	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Di-n-octyl phthalate	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Di-n-octyl phthalate	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Dibenz(a,h) anthracene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Dibenz(a,h) anthracene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Dibenzofuran	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Dibenzofuran	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Diethyl phthalate	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Diethyl phthalate	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Dimethyl phthalate	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Dimethyl phthalate	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Fluoranthene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Fluoranthene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Fluorene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Fluorene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorobutadiene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorobutadiene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorocyclopentadiene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachlorocyclopentadiene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8270D (Rev 2014)	Hexachloroethane	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Hexachloroethane	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Indeno(1,2,3-cd) pyrene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Indeno(1,2,3-cd) pyrene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Isophorone	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Isophorone	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodi-n-propylamine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodi-n-propylamine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodimethylamine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodimethylamine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodiphenylamine	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	n-Nitrosodiphenylamine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Naphthalene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Naphthalene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Nitrobenzene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Nitrobenzene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Pentachlorophenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Pentachlorophenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Phenanthrene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Phenanthrene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Phenol	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Phenol	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Pyrene	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Pyrene	SCM	MN	
RCRP	EPA 8270D (Rev 2014)	Pyridine	NPW	MN	
RCRP	EPA 8270D (Rev 2014)	Pyridine	SCM	MN	

EPA 8280B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8280B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8280B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8280B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8280B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8280B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8280B	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8280B	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	SCM	MN	
RCRP	EPA 8280B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	NPW	MN	
RCRP	EPA 8280B	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	SCM	MN	
RCRP	EPA 8280B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	SCM	MN	
RCRP	EPA 8280B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	NPW	MN	
RCRP	EPA 8280B	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	NPW	MN	
RCRP	EPA 8280B	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	SCM	MN	
RCRP	EPA 8280B	2,3,4,6,7,8-Hexachlorodibenzofuran	NPW	MN	
RCRP	EPA 8280B	2,3,4,6,7,8-Hexachlorodibenzofuran	SCM	MN	
RCRP	EPA 8280B	2,3,4,7,8-Pentachlorodibenzofuran	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8280B	2,3,4,7,8-Pentachlorodibenzofuran	NPW	MN	
RCRP	EPA 8280B	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	SCM	MN	
RCRP	EPA 8280B	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	NPW	MN	
RCRP	EPA 8280B	2,3,7,8-Tetrachlorodibenzofuran	NPW	MN	
RCRP	EPA 8280B	2,3,7,8-Tetrachlorodibenzofuran	SCM	MN	
RCRP	EPA 8280B	Total HpCDD	NPW	MN	
RCRP	EPA 8280B	Total HpCDD	SCM	MN	
RCRP	EPA 8280B	Total HpCDF	SCM	MN	
RCRP	EPA 8280B	Total HpCDF	NPW	MN	
RCRP	EPA 8280B	Total HxCDD	SCM	MN	
RCRP	EPA 8280B	Total HxCDD	NPW	MN	
RCRP	EPA 8280B	Total HxCDF	SCM	MN	
RCRP	EPA 8280B	Total HxCDF	NPW	MN	
RCRP	EPA 8280B	Total PeCDD	NPW	MN	
RCRP	EPA 8280B	Total PeCDD	SCM	MN	
RCRP	EPA 8280B	Total PeCDF	NPW	MN	
RCRP	EPA 8280B	Total PeCDF	SCM	MN	
RCRP	EPA 8280B	Total TCDD	SCM	MN	
RCRP	EPA 8280B	Total TCDD	NPW	MN	
RCRP	EPA 8280B	Total TCDF	SCM	MN	
RCRP	EPA 8280B	Total TCDF	NPW	MN	

EPA 8290

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, soxhlet;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	NPW	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	NPW	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	NPW	MN	
RCRP	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	NPW	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	SCM	MN	
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	NPW	MN	
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	NPW	MN	
RCRP	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	SCM	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	NPW	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	SCM	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	TISSUE	MN	
RCRP	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	NPW	MN	
RCRP	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	NPW	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	SCM	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	TISSUE	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290	Total Heptachlorodibenzo-p-dioxin (HpCDD, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Heptachlorodibenzofuran (HpCDF, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Hexachlorodibenzo-p-dioxin (HxCDD, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Hexachlorodibenzofuran (HxCDF, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Hpcdd	NPW	MN	
RCRP	EPA 8290	Total Hpcdd	SCM	MN	
RCRP	EPA 8290	Total Hpcdf	SCM	MN	
RCRP	EPA 8290	Total Hpcdf	NPW	MN	
RCRP	EPA 8290	Total Hxcdd	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290	Total Hxcdd	NPW	MN	
RCRP	EPA 8290	Total Hxcdf	SCM	MN	
RCRP	EPA 8290	Total Hxcdf	NPW	MN	
RCRP	EPA 8290	Total Pecdd	NPW	MN	
RCRP	EPA 8290	Total Pecdd	SCM	MN	
RCRP	EPA 8290	Total Pecdf	SCM	MN	
RCRP	EPA 8290	Total Pecdf	NPW	MN	
RCRP	EPA 8290	Total Pentachlorodibenzo-p-dioxin (PeCDD, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Pentachlorodibenzofuran (PeCDF, Total)	TISSUE	MN	
RCRP	EPA 8290	Total TCDD	SCM	MN	
RCRP	EPA 8290	Total TCDD	NPW	MN	
RCRP	EPA 8290	Total TCDF	NPW	MN	
RCRP	EPA 8290	Total TCDF	SCM	MN	
RCRP	EPA 8290	Total Tetrachlorodibenzo-p-dioxin (TCDD, Total)	TISSUE	MN	
RCRP	EPA 8290	Total Tetrachlorodibenzofuran (TCDF, Total)	TISSUE	MN	

EPA 8290A

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, soxhlet;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (1,2,3,4,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,4,7,8-Hexachlorodibenzofuran (1,2,3,4,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	NPW	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	SCM	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin(1,2,3,6,7,8-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	NPW	MN	
RCRP	EPA 8290A	1,2,3,6,7,8-Hexachlorodibenzofuran (1,2,3,6,7,8-Hxcdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	SCM	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	NPW	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (1,2,3,7,8,9-Hxcdd)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	NPW	MN	
RCRP	EPA 8290A	1,2,3,7,8,9-Hexachlorodibenzofuran (1,2,3,7,8,9-Hxcdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	SCM	MN	
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (1,2,3,7,8-Pecdd)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	TISSUE	MN	
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	SCM	MN	
RCRP	EPA 8290A	1,2,3,7,8-Pentachlorodibenzofuran (1,2,3,7,8-Pecdf)	NPW	MN	
RCRP	EPA 8290A	2,3,4,6,7,8-Hexachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290A	2,3,4,6,7,8-Hexachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290A	2,3,4,6,7,8-Hexachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290A	2,3,4,7,8-Pentachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290A	2,3,4,7,8-Pentachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290A	2,3,4,7,8-Pentachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	NPW	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	SCM	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	TISSUE	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzofuran	TISSUE	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzofuran	SCM	MN	
RCRP	EPA 8290A	2,3,7,8-Tetrachlorodibenzofuran	NPW	MN	
RCRP	EPA 8290A	Total Heptachlorodibenzo-p-dioxin (HpCDD, Total)	SCM	MN	
RCRP	EPA 8290A	Total Heptachlorodibenzo-p-dioxin (HpCDD, Total)	NPW	MN	
RCRP	EPA 8290A	Total Heptachlorodibenzofuran (HpCDF, Total)	SCM	MN	
RCRP	EPA 8290A	Total Heptachlorodibenzofuran (HpCDF, Total)	NPW	MN	
RCRP	EPA 8290A	Total Hexachlorodibenzo-p-dioxin (HxCDD, Total)	SCM	MN	
RCRP	EPA 8290A	Total Hexachlorodibenzo-p-dioxin (HxCDD, Total)	NPW	MN	
RCRP	EPA 8290A	Total Hexachlorodibenzofuran (HxCDF, Total)	SCM	MN	
RCRP	EPA 8290A	Total Hexachlorodibenzofuran (HxCDF, Total)	NPW	MN	
RCRP	EPA 8290A	Total HpCDD	TISSUE	MN	
RCRP	EPA 8290A	Total HpCDF	TISSUE	MN	
RCRP	EPA 8290A	Total HxCDD	TISSUE	MN	
RCRP	EPA 8290A	Total HxCDF	TISSUE	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8290A	Total PeCDD	TISSUE	MN	
RCRP	EPA 8290A	Total PeCDF	TISSUE	MN	
RCRP	EPA 8290A	Total Pentachlorodibenzo-p-dioxin (PeCDD, Total)	NPW	MN	
RCRP	EPA 8290A	Total Pentachlorodibenzo-p-dioxin (PeCDD, Total)	SCM	MN	
RCRP	EPA 8290A	Total Pentachlorodibenzofuran (PeCDF, Total)	NPW	MN	
RCRP	EPA 8290A	Total Pentachlorodibenzofuran (PeCDF, Total)	SCM	MN	
RCRP	EPA 8290A	Total TCDD	TISSUE	MN	
RCRP	EPA 8290A	Total TCDF	TISSUE	MN	
RCRP	EPA 8290A	Total Tetrachlorodibenzo-p-dioxin (TCDD, Total)	SCM	MN	
RCRP	EPA 8290A	Total Tetrachlorodibenzo-p-dioxin (TCDD, Total)	NPW	MN	
RCRP	EPA 8290A	Total Tetrachlorodibenzofuran (TCDF, Total)	SCM	MN	
RCRP	EPA 8290A	Total Tetrachlorodibenzofuran (TCDF, Total)	NPW	MN	

EPA 9095B

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 9095B	Paint Filter Liquids Test	SCM	MN	

EPA 8015B

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic; Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8015B	Diesel range organics (DRO)	NPW	MN	
RCRP	EPA 8015B	Diesel range organics (DRO)	SCM	MN	
RCRP	EPA 8015B	Gasoline range organics (GRO)	NPW	MN	
RCRP	EPA 8015B	Gasoline range organics (GRO)	SCM	MN	

EPA 8015C

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic; Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8015C	Diesel range organics (DRO)	SCM	MN	
RCRP	EPA 8015C	Diesel range organics (DRO)	NPW	MN	
RCRP	EPA 8015C	Gasoline range organics (GRO)	SCM	MN	
RCRP	EPA 8015C	Gasoline range organics (GRO)	NPW	MN	

EPA 8015D

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE);

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8015D	Diesel range organics (DRO)	SCM	MN	
RCRP	EPA 8015D	Diesel range organics (DRO)	NPW	MN	

EPA 8021B

Preparation Techniques: Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8021B	1,2,4-Trimethylbenzene	SCM	MN	
RCRP	EPA 8021B	1,2,4-Trimethylbenzene	NPW	MN	
RCRP	EPA 8021B	1,3,5-Trimethylbenzene	SCM	MN	
RCRP	EPA 8021B	1,3,5-Trimethylbenzene	NPW	MN	
RCRP	EPA 8021B	Benzene	SCM	MN	
RCRP	EPA 8021B	Benzene	NPW	MN	
RCRP	EPA 8021B	Ethylbenzene	NPW	MN	
RCRP	EPA 8021B	Ethylbenzene	SCM	MN	
RCRP	EPA 8021B	m+p-xylene	SCM	MN	
RCRP	EPA 8021B	m+p-xylene	NPW	MN	
RCRP	EPA 8021B	Methyl tert-butyl ether (MTBE)	SCM	MN	
RCRP	EPA 8021B	Methyl tert-butyl ether (MTBE)	NPW	MN	
RCRP	EPA 8021B	o-Xylene	SCM	MN	
RCRP	EPA 8021B	o-Xylene	NPW	MN	
RCRP	EPA 8021B	Toluene	NPW	MN	
RCRP	EPA 8021B	Toluene	SCM	MN	
RCRP	EPA 8021B	Xylene (total)	NPW	MN	
RCRP	EPA 8021B	Xylene (total)	SCM	MN	

EPA 8260B

Preparation Techniques: Extraction, EPA 1312 SPLP, zero headspace (ZHE); Extraction, EPA 1311 TCLP, zero headspace (ZHE); Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,1,2-Tetrachloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,1-Trichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,1-Trichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	SCM	MN	
RCRP	EPA 8260B	1,1,2,2-Tetrachloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	NPW	MN	
RCRP	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	SCM	MN	
RCRP	EPA 8260B	1,1,2-Trichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1,2-Trichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloroethane	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloroethane	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	1,1-Dichloropropene	SCM	MN	
RCRP	EPA 8260B	1,1-Dichloropropene	NPW	MN	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,2,3-Trichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2,3-Trichloropropane	NPW	MN	
RCRP	EPA 8260B	1,2,3-Trichloropropane	SCM	MN	
RCRP	EPA 8260B	1,2,3-Trimethylbenzene	NPW	MN	
RCRP	EPA 8260B	1,2,3-Trimethylbenzene	SCM	MN	
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2,4-Trichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	SCM	MN	
RCRP	EPA 8260B	1,2,4-Trimethylbenzene	NPW	MN	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	NPW	MN	
RCRP	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)	SCM	MN	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	NPW	MN	
RCRP	EPA 8260B	1,2-Dibromoethane (EDB, Ethylene dibromide)	SCM	MN	
RCRP	EPA 8260B	1,2-Dichlorobenzene	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	1,2-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	NPW	MN	
RCRP	EPA 8260B	1,2-Dichloroethane (Ethylene dichloride)	SCM	MN	
RCRP	EPA 8260B	1,2-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	1,2-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	NPW	MN	
RCRP	EPA 8260B	1,3,5-Trimethylbenzene	SCM	MN	
RCRP	EPA 8260B	1,3-Dichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,3-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,3-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	1,3-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	1,4-Dichlorobenzene	SCM	MN	
RCRP	EPA 8260B	1,4-Dichlorobenzene	NPW	MN	
RCRP	EPA 8260B	1,4-Dioxane (1,4- Diethyleneoxide)	SCM	MN	
RCRP	EPA 8260B	1,4-Dioxane (1,4- Diethyleneoxide)	NPW	MN	
RCRP	EPA 8260B	2,2-Dichloropropane	SCM	MN	
RCRP	EPA 8260B	2,2-Dichloropropane	NPW	MN	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	SCM	MN	
RCRP	EPA 8260B	2-Butanone (Methyl ethyl ketone, MEK)	NPW	MN	
RCRP	EPA 8260B	2-Chloroethyl vinyl ether	NPW	MN	
RCRP	EPA 8260B	2-Chloroethyl vinyl ether	SCM	MN	
RCRP	EPA 8260B	2-Chlorotoluene	NPW	MN	
RCRP	EPA 8260B	2-Chlorotoluene	SCM	MN	
RCRP	EPA 8260B	2-Hexanone	SCM	MN	
RCRP	EPA 8260B	2-Hexanone	NPW	MN	
RCRP	EPA 8260B	2-Methylnaphthalene	SCM	MN	
RCRP	EPA 8260B	2-Nitropropane	NPW	MN	
RCRP	EPA 8260B	4-Chlorotoluene	SCM	MN	
RCRP	EPA 8260B	4-Chlorotoluene	NPW	MN	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	NPW	MN	
RCRP	EPA 8260B	4-Isopropyltoluene (p-Cymene)	SCM	MN	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	SCM	MN	
RCRP	EPA 8260B	4-Methyl-2-pentanone (MIBK)	NPW	MN	
RCRP	EPA 8260B	Acetone	NPW	MN	
RCRP	EPA 8260B	Acetone	SCM	MN	
RCRP	EPA 8260B	Acetonitrile	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Acrolein (Propenal)	SCM	MN	
RCRP	EPA 8260B	Acrolein (Propenal)	NPW	MN	
RCRP	EPA 8260B	Acrylonitrile	SCM	MN	
RCRP	EPA 8260B	Acrylonitrile	NPW	MN	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	SCM	MN	
RCRP	EPA 8260B	Allyl chloride (3-Chloropropene)	NPW	MN	
RCRP	EPA 8260B	Benzene	SCM	MN	
RCRP	EPA 8260B	Benzene	NPW	MN	
RCRP	EPA 8260B	Bromobenzene	SCM	MN	
RCRP	EPA 8260B	Bromobenzene	NPW	MN	
RCRP	EPA 8260B	Bromochloromethane	NPW	MN	
RCRP	EPA 8260B	Bromochloromethane	SCM	MN	
RCRP	EPA 8260B	Bromodichloromethane	NPW	MN	
RCRP	EPA 8260B	Bromodichloromethane	SCM	MN	
RCRP	EPA 8260B	Bromoform	NPW	MN	
RCRP	EPA 8260B	Bromoform	SCM	MN	
RCRP	EPA 8260B	Carbon disulfide	SCM	MN	
RCRP	EPA 8260B	Carbon disulfide	NPW	MN	
RCRP	EPA 8260B	Carbon tetrachloride	NPW	MN	
RCRP	EPA 8260B	Carbon tetrachloride	SCM	MN	
RCRP	EPA 8260B	Chlorobenzene	SCM	MN	
RCRP	EPA 8260B	Chlorobenzene	NPW	MN	
RCRP	EPA 8260B	Chlorodibromomethane	NPW	MN	
RCRP	EPA 8260B	Chlorodibromomethane	SCM	MN	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	SCM	MN	
RCRP	EPA 8260B	Chloroethane (Ethyl chloride)	NPW	MN	
RCRP	EPA 8260B	Chloroform	SCM	MN	
RCRP	EPA 8260B	Chloroform	NPW	MN	
RCRP	EPA 8260B	Chloroprene (2-Chloro-1,3-butadiene)	NPW	MN	
RCRP	EPA 8260B	cis & trans-1,2-Dichloroethene	SCM	MN	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	cis-1,2-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	NPW	MN	
RCRP	EPA 8260B	cis-1,3-Dichloropropene	SCM	MN	
RCRP	EPA 8260B	cis-1,4-Dichloro-2-butene	NPW	MN	
RCRP	EPA 8260B	Cyclohexane	SCM	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Cyclohexane	NPW	MN	
RCRP	EPA 8260B	Di-isopropylether (DIPE)	SCM	MN	
RCRP	EPA 8260B	Di-isopropylether (DIPE)	NPW	MN	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	SCM	MN	
RCRP	EPA 8260B	Dibromomethane (Methylene bromide)	NPW	MN	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	NPW	MN	
RCRP	EPA 8260B	Dichlorodifluoromethane (Freon-12)	SCM	MN	
RCRP	EPA 8260B	Diethyl ether	SCM	MN	
RCRP	EPA 8260B	Diethyl ether	NPW	MN	
RCRP	EPA 8260B	Ethanol	SCM	MN	
RCRP	EPA 8260B	Ethanol	NPW	MN	
RCRP	EPA 8260B	Ethyl acetate	NPW	MN	
RCRP	EPA 8260B	Ethyl methacrylate	NPW	MN	
RCRP	EPA 8260B	Ethyl-t-butylether (ETBE) (2-Ethoxy-2-methylpropane)	NPW	MN	
RCRP	EPA 8260B	Ethyl-t-butylether (ETBE) (2-Ethoxy-2-methylpropane)	SCM	MN	
RCRP	EPA 8260B	Ethylbenzene	NPW	MN	
RCRP	EPA 8260B	Ethylbenzene	SCM	MN	
RCRP	EPA 8260B	Hexachlorobutadiene	NPW	MN	
RCRP	EPA 8260B	Hexachlorobutadiene	SCM	MN	
RCRP	EPA 8260B	Iodomethane (Methyl iodide)	SCM	MN	
RCRP	EPA 8260B	Iodomethane (Methyl iodide)	NPW	MN	
RCRP	EPA 8260B	Isobutyl alcohol (2-Methyl-1-propanol)	SCM	MN	
RCRP	EPA 8260B	Isobutyl alcohol (2-Methyl-1-propanol)	NPW	MN	
RCRP	EPA 8260B	Isopropyl alcohol (2-Propanol, Isopropanol)	NPW	MN	
RCRP	EPA 8260B	Isopropylbenzene	SCM	MN	
RCRP	EPA 8260B	Isopropylbenzene	NPW	MN	
RCRP	EPA 8260B	m+p-xylene	SCM	MN	
RCRP	EPA 8260B	m+p-xylene	NPW	MN	
RCRP	EPA 8260B	Methacrylonitrile	NPW	MN	
RCRP	EPA 8260B	Methyl acetate	NPW	MN	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	NPW	MN	
RCRP	EPA 8260B	Methyl bromide (Bromomethane)	SCM	MN	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	SCM	MN	
RCRP	EPA 8260B	Methyl chloride (Chloromethane)	NPW	MN	
RCRP	EPA 8260B	Methyl methacrylate	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	NPW	MN	
RCRP	EPA 8260B	Methyl tert-butyl ether (MTBE)	SCM	MN	
RCRP	EPA 8260B	Methylcyclohexane	NPW	MN	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	SCM	MN	
RCRP	EPA 8260B	Methylene chloride (Dichloromethane)	NPW	MN	
RCRP	EPA 8260B	n-Butyl alcohol (1-Butanol, n-Butanol)	NPW	MN	
RCRP	EPA 8260B	n-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	n-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	n-Hexane	SCM	MN	
RCRP	EPA 8260B	n-Hexane	NPW	MN	
RCRP	EPA 8260B	n-Propylbenzene	SCM	MN	
RCRP	EPA 8260B	n-Propylbenzene	NPW	MN	
RCRP	EPA 8260B	Naphthalene	NPW	MN	
RCRP	EPA 8260B	Naphthalene	SCM	MN	
RCRP	EPA 8260B	o-Xylene	SCM	MN	
RCRP	EPA 8260B	o-Xylene	NPW	MN	
RCRP	EPA 8260B	Propionitrile (Ethyl cyanide)	NPW	MN	
RCRP	EPA 8260B	sec-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	sec-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	Styrene	SCM	MN	
RCRP	EPA 8260B	Styrene	NPW	MN	
RCRP	EPA 8260B	T-amylmethylether (TAME)	SCM	MN	
RCRP	EPA 8260B	T-amylmethylether (TAME)	NPW	MN	
RCRP	EPA 8260B	tert-Butyl alcohol	SCM	MN	
RCRP	EPA 8260B	tert-Butyl alcohol	NPW	MN	
RCRP	EPA 8260B	tert-Butylbenzene	NPW	MN	
RCRP	EPA 8260B	tert-Butylbenzene	SCM	MN	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	NPW	MN	
RCRP	EPA 8260B	Tetrachloroethylene (Perchloroethylene)	SCM	MN	
RCRP	EPA 8260B	Tetrahydrofuran (THF)	SCM	MN	
RCRP	EPA 8260B	Tetrahydrofuran (THF)	NPW	MN	
RCRP	EPA 8260B	Toluene	SCM	MN	
RCRP	EPA 8260B	Toluene	NPW	MN	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	SCM	MN	
RCRP	EPA 8260B	trans-1,2-Dichloroethylene	NPW	MN	
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	NPW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA 8260B	trans-1,3-Dichloropropylene	SCM	MN	
RCRP	EPA 8260B	trans-1,4-Dichloro-2-butene	NPW	MN	
RCRP	EPA 8260B	trans-1,4-Dichloro-2-butene	SCM	MN	
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	NPW	MN	
RCRP	EPA 8260B	Trichloroethene (Trichloroethylene)	SCM	MN	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	SCM	MN	
RCRP	EPA 8260B	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	NPW	MN	
RCRP	EPA 8260B	Vinyl acetate	NPW	MN	
RCRP	EPA 8260B	Vinyl acetate	SCM	MN	
RCRP	EPA 8260B	Vinyl chloride	NPW	MN	
RCRP	EPA 8260B	Vinyl chloride	SCM	MN	
RCRP	EPA 8260B	Xylene (total)	NPW	MN	
RCRP	EPA 8260B	Xylene (total)	SCM	MN	

EPA RSK-175 (GC/FID)

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
RCRP	EPA RSK-175 (GC/FID)	Ethane	NPW	MN	
RCRP	EPA RSK-175 (GC/FID)	Ethene	NPW	MN	
RCRP	EPA RSK-175 (GC/FID)	Methane	NPW	MN	

Safe Drinking Water Program

EPA 180.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 180.1	Turbidity	DW	MN	

EPA 300.0

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 300.0	Bromide	DW	MN	
SDWP	EPA 300.0	Chloride	DW	MN	
SDWP	EPA 300.0	Fluoride	DW	MN	
SDWP	EPA 300.0	Nitrate	DW	MN	
SDWP	EPA 300.0	Nitrite	DW	MN	
SDWP	EPA 300.0	Sulfate	DW	MN	

EPA 353.2

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 353.2	Nitrate	DW	MN	
SDWP	EPA 353.2	Nitrite	DW	MN	

SM 2320 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 2320 B-97	Alkalinity as CaCO3	DW	MN	

SM 2340 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 2340 B-97	Hardness	DW	MN	

SM 2510 B-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 2510 B-97	Conductivity	DW	MN	

SM 2540 C-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 2540 C-97	Residue-filterable (TDS)	DW	MN	

SM 4500-Cl G-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-Cl G-93	Total chlorine	DW	MN	

SM 4500-Cl⁻ E-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-Cl⁻E-97	Chloride	DW	MN	

SM 4500-CN⁻ E-97

Preparation Techniques: Distillation, micro;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-CN⁻E-97	Cyanide	DW	MN	

SM 4500-F⁻ C-97

Preparation Techniques: N/A;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-F⁻C-97	Fluoride	DW	MN	

SM 4500-H+ B-96

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-H+ B-96	pH	DW	MN	

SM 4500-$NO2^-$B-93

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 4500-$NO2^-$B-93	Nitrite	DW	MN	

EPA 200.8

Preparation Techniques: Digestion, hotplate or HotBlock;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 200.8	Aluminum	DW	MN	
SDWP	EPA 200.8	Antimony	DW	MN	
SDWP	EPA 200.8	Arsenic	DW	MN	
SDWP	EPA 200.8	Barium	DW	MN	
SDWP	EPA 200.8	Beryllium	DW	MN	
SDWP	EPA 200.8	Cadmium	DW	MN	
SDWP	EPA 200.8	Chromium	DW	MN	
SDWP	EPA 200.8	Copper	DW	MN	
SDWP	EPA 200.8	Lead	DW	MN	
SDWP	EPA 200.8	Manganese	DW	MN	
SDWP	EPA 200.8	Nickel	DW	MN	
SDWP	EPA 200.8	Selenium	DW	MN	
SDWP	EPA 200.8	Silver	DW	MN	
SDWP	EPA 200.8	Thallium	DW	MN	
SDWP	EPA 200.8	Uranium	DW	MN	
SDWP	EPA 200.8	Zinc	DW	MN	

EPA 245.1

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 245.1	Mercury	DW	MN	

SM 9215 B (R2A)-94

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 9215 B (R2A)-94	Heterotrophic plate count	DW	MN	

SM 9223 B (Colilert®)-97

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	SM 9223 B (Colilert®)-97	Escherichia coli	DW	MN	
SDWP	SM 9223 B (Colilert®)-97	Total coliforms	DW	MN	

EPA 1613

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, automated soxhlet; Extraction, solid phase (SPE);

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 1613	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	DW	MN	

EPA 1613 mod GCMS

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 1613 mod GCMS	2,3,7,8-Tetrachlorodibenzo- p-dioxin (2,3,7,8-TCDD)	DW	MN	

EPA 524.2

Preparation Techniques: Purge and trap;

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 524.2	1,1,1,2-Tetrachloroethane	DW	MN	
SDWP	EPA 524.2	1,1,1-Trichloroethane	DW	MN	
SDWP	EPA 524.2	1,1,2,2-Tetrachloroethane	DW	MN	
SDWP	EPA 524.2	1,1,2-Trichloroethane	DW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 524.2	1,1-Dichloroethane	DW	MN	
SDWP	EPA 524.2	1,1-Dichloroethylene	DW	MN	
SDWP	EPA 524.2	1,1-Dichloropropene	DW	MN	
SDWP	EPA 524.2	1,2,3-Trichlorobenzene	DW	MN	
SDWP	EPA 524.2	1,2,3-Trichloropropane	DW	MN	
SDWP	EPA 524.2	1,2,4-Trichlorobenzene	DW	MN	
SDWP	EPA 524.2	1,2,4-Trimethylbenzene	DW	MN	
SDWP	EPA 524.2	1,2-Dichlorobenzene	DW	MN	
SDWP	EPA 524.2	1,2-Dichloroethane (Ethylene dichloride)	DW	MN	
SDWP	EPA 524.2	1,2-Dichloropropane	DW	MN	
SDWP	EPA 524.2	1,3-Dichlorobenzene	DW	MN	
SDWP	EPA 524.2	1,4-Dichlorobenzene	DW	MN	
SDWP	EPA 524.2	2,2-Dichloropropane	DW	MN	
SDWP	EPA 524.2	2-Chlorotoluene	DW	MN	
SDWP	EPA 524.2	4-Chlorotoluene	DW	MN	
SDWP	EPA 524.2	Benzene	DW	MN	
SDWP	EPA 524.2	Bromobenzene	DW	MN	
SDWP	EPA 524.2	Bromochloromethane	DW	MN	
SDWP	EPA 524.2	Bromodichloromethane	DW	MN	
SDWP	EPA 524.2	Bromoform	DW	MN	
SDWP	EPA 524.2	Bromomethane	DW	MN	
SDWP	EPA 524.2	Carbon tetrachloride	DW	MN	
SDWP	EPA 524.2	Chlorobenzene	DW	MN	
SDWP	EPA 524.2	Chlorodibromomethane	DW	MN	
SDWP	EPA 524.2	Chloroethane (Ethyl chloride)	DW	MN	
SDWP	EPA 524.2	Chloroform	DW	MN	
SDWP	EPA 524.2	cis-1,2-Dichloroethylene	DW	MN	
SDWP	EPA 524.2	cis-1,3-Dichloropropene	DW	MN	
SDWP	EPA 524.2	Dibromomethane (Methylene bromide)	DW	MN	
SDWP	EPA 524.2	Dichlorodifluoromethane (Freon-12)	DW	MN	
SDWP	EPA 524.2	Ethylbenzene	DW	MN	
SDWP	EPA 524.2	Hexachlorobutadiene	DW	MN	
SDWP	EPA 524.2	Isopropylbenzene	DW	MN	
SDWP	EPA 524.2	Methyl chloride (Chloromethane)	DW	MN	
SDWP	EPA 524.2	Methyl tert-butyl ether (MTBE)	DW	MN	
SDWP	EPA 524.2	Methylene chloride (Dichloromethane)	DW	MN	

Program	Method	Analyte	Matrix	Primary	SOP
SDWP	EPA 524.2	n-Butylbenzene	DW	MN	
SDWP	EPA 524.2	n-Propylbenzene	DW	MN	
SDWP	EPA 524.2	Naphthalene	DW	MN	
SDWP	EPA 524.2	sec-Butylbenzene	DW	MN	
SDWP	EPA 524.2	Styrene	DW	MN	
SDWP	EPA 524.2	tert-Butylbenzene	DW	MN	
SDWP	EPA 524.2	Tetrachloroethylene (Perchloroethylene)	DW	MN	
SDWP	EPA 524.2	Toluene	DW	MN	
SDWP	EPA 524.2	Total Trihalomethanes	DW	MN	
SDWP	EPA 524.2	trans-1,2-Dichloroethylene	DW	MN	
SDWP	EPA 524.2	trans-1,3-Dichloropropylene	DW	MN	
SDWP	EPA 524.2	Trichloroethene (Trichloroethylene)	DW	MN	
SDWP	EPA 524.2	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	DW	MN	
SDWP	EPA 524.2	Vinyl chloride	DW	MN	
SDWP	EPA 524.2	Xylene (total)	DW	MN	

Underground Storage Tank Program

AK102 DRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	AK102 DRO	Diesel range organics (DRO)	NPW	MN	
USTP	AK102 DRO	Diesel range organics (DRO)	SCM	MN	

AK102 DRO-SV

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	AK102 DRO-SV	Diesel range organics (DRO)	NPW	MN	
USTP	AK102 DRO-SV	Diesel range organics (DRO)	SCM	MN	

AK103 RRO

Preparation Techniques: Extraction, separatory funnel liquid-liquid (LLE); Extraction, ultrasonic;

Program	Method	Analyte	Matrix	Primary	SOP
USTP	AK103 RRO	Residual Range Organics (RRO) - Oil Range Organics	SCM	MN	

WI(95) DRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) DRO	Diesel range organics (DRO)	NPW	MN	
USTP	WI(95) DRO	Diesel range organics (DRO)	SCM	MN	
USTP	WI(95) DRO	Diesel range organics (DRO)	SCM	MN	User Defined S-MN-O-466 Rev. Rev.23
USTP	WI(95) DRO	Diesel range organics (DRO)	NPW	MN	User Defined S-MN-O-466 Rev. Rev.23

AK101 GRO-MS

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	AK101 GRO-MS	Gasoline range organics (GRO)	SCM	MN	
USTP	AK101 GRO-MS	Gasoline range organics (GRO)	NPW	MN	

EPA TO-15

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	1,1,1-Trichloroethane	AIR	MN	
USTP	EPA TO-15	1,1,2,2-Tetrachloroethane	AIR	MN	
USTP	EPA TO-15	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	AIR	MN	
USTP	EPA TO-15	1,1,2-Trichloroethane	AIR	MN	
USTP	EPA TO-15	1,1-Dichloroethane	AIR	MN	
USTP	EPA TO-15	1,1-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	1,2,4-Trichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,2,4-Trimethylbenzene	AIR	MN	
USTP	EPA TO-15	1,2-Dibromoethane (EDB, Ethylene dibromide)	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)	AIR	MN	
USTP	EPA TO-15	1,2-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,2-Dichloroethane (Ethylene dichloride)	AIR	MN	
USTP	EPA TO-15	1,2-Dichloropropane	AIR	MN	
USTP	EPA TO-15	1,3,5-Trimethylbenzene	AIR	MN	
USTP	EPA TO-15	1,3-Butadiene	AIR	MN	
USTP	EPA TO-15	1,3-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,4-Dichlorobenzene	AIR	MN	
USTP	EPA TO-15	1,4-Dioxane (1,4- Diethyleneoxide)	AIR	MN	
USTP	EPA TO-15	1-Propene	AIR	MN	
USTP	EPA TO-15	2-Butanone (Methyl ethyl ketone, MEK)	AIR	MN	
USTP	EPA TO-15	2-Hexanone	AIR	MN	
USTP	EPA TO-15	4-Ethyltoluene	AIR	MN	
USTP	EPA TO-15	4-Methyl-2-pentanone (MIBK)	AIR	MN	
USTP	EPA TO-15	Acetone	AIR	MN	
USTP	EPA TO-15	Benzene	AIR	MN	
USTP	EPA TO-15	Benzyl chloride	AIR	MN	
USTP	EPA TO-15	Bromodichloromethane	AIR	MN	
USTP	EPA TO-15	Bromoform	AIR	MN	
USTP	EPA TO-15	Carbon disulfide	AIR	MN	
USTP	EPA TO-15	Carbon tetrachloride	AIR	MN	
USTP	EPA TO-15	Chlorobenzene	AIR	MN	
USTP	EPA TO-15	Chlorodibromomethane	AIR	MN	
USTP	EPA TO-15	Chloroethane (Ethyl chloride)	AIR	MN	
USTP	EPA TO-15	Chloroform	AIR	MN	
USTP	EPA TO-15	cis-1,2-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	cis-1,3-Dichloropropene	AIR	MN	
USTP	EPA TO-15	Cyclohexane	AIR	MN	
USTP	EPA TO-15	Dichlorodifluoromethane (Freon-12)	AIR	MN	
USTP	EPA TO-15	Ethanol	AIR	MN	
USTP	EPA TO-15	Ethyl acetate	AIR	MN	
USTP	EPA TO-15	Ethylbenzene	AIR	MN	
USTP	EPA TO-15	Hexachlorobutadiene	AIR	MN	
USTP	EPA TO-15	Isopropyl alcohol (2-Propanol, Isopropanol)	AIR	MN	
USTP	EPA TO-15	m+p-xylene	AIR	MN	

Program	Method	Analyte	Matrix	Primary	SOP
USTP	EPA TO-15	Methyl bromide (Bromomethane)	AIR	MN	
USTP	EPA TO-15	Methyl chloride (Chloromethane)	AIR	MN	
USTP	EPA TO-15	Methyl tert-butyl ether (MTBE)	AIR	MN	
USTP	EPA TO-15	Methylene chloride (Dichloromethane)	AIR	MN	
USTP	EPA TO-15	n-Heptane	AIR	MN	
USTP	EPA TO-15	n-Hexane	AIR	MN	
USTP	EPA TO-15	Naphthalene	AIR	MN	
USTP	EPA TO-15	o-Xylene	AIR	MN	
USTP	EPA TO-15	Styrene	AIR	MN	
USTP	EPA TO-15	Tetrachloroethylene (Perchloroethylene)	AIR	MN	
USTP	EPA TO-15	Tetrahydrofuran (THF)	AIR	MN	
USTP	EPA TO-15	Toluene	AIR	MN	
USTP	EPA TO-15	trans-1,2-Dichloroethylene	AIR	MN	
USTP	EPA TO-15	trans-1,3-Dichloropropylene	AIR	MN	
USTP	EPA TO-15	Trichloroethene (Trichloroethylene)	AIR	MN	
USTP	EPA TO-15	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	AIR	MN	
USTP	EPA TO-15	Vinyl acetate	AIR	MN	
USTP	EPA TO-15	Vinyl chloride	AIR	MN	
USTP	EPA TO-15	Xylene (total)	AIR	MN	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Gasoline range organics (GRO)	NPW	MN	
USTP	WI(95) GRO	Gasoline range organics (GRO)	SCM	MN	

WI(95) GRO

Preparation Techniques: N/A

Program	Method	Analyte	Matrix	Primary	SOP
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	NPW	MN	
USTP	WI(95) GRO	Petroleum Volatile Organic Compounds (PVOC)	SCM	MN	

Note: Method beginning with "SM" refer to the approved editions of Standard methods for the Examination of Water and Wastes. Approved methods are listed in the applicable parts of Title 40 of the Code of Federal Regulations (including its subsequent Federal Register updates), MN Statutes and Rules, and state-issued permits.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

JUN 29 2018

REPLY TO THE ATTENTION OF:
WG-15J

Mr. Paul Moyer,
Minnesota Department of Health
Division of Public Laboratories
601 Robert Street North
P.O. Box 64899
St. Paul, Minnesota 55164-0899

Dear Mr. Moyer,

It is the U.S. Environmental Protection Agency (US EPA) Region 5's responsibility to evaluate the capabilities of the Principle Lab for the State of Minnesota drinking water program for analyzing samples pursuant to the National Primary Drinking Water Regulations as implemented by 40 CFR Parts 141 and 142. The Region 5 Laboratory Certification Program (R5 LCP) performed an on-site audit of Minnesota Department of Health (MDH), Division of Public Laboratories (DPL) on September 18-22, 2017. The certification by our office for the MDH - DPL located at 601 Robert Street North, expired on April 21, 2018, before completion of the audit report which is in the final stages of preparation.

Based on the observations from the September 18-22, 2017 on-site visit and the most recent Proficiency Testing (PT) results, the US EPA grants to the MDH-DPL Interim Certification for the analytes and methods identified in Enclosure A for chemistry, microbiology, and radiochemistry. Interim Certification is effective from April 22, 2018 to October 22, 2018, or until a final certification decision is issued, which will be based upon completion of the audit report and review of MDH responses.

If you have any questions concerning this letter, please contact Frank Lagunas, Region 5's Laboratory Certification Program Manager, at (312) 886-4466 or by e-mail at lagunas.frank@epa.gov.

Sincerely,

Thomas Poy

for Linda Holst
Acting Director, Water Division

Enclosure: A

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Enclosure A
MDH, Division of Public Health Laboratories
Parameters List of Interim Certification
April 18, 2018

Inorganic Contaminants (IOCs)	Method, 40 CFR	Certification Status
Antimony	200.8	Interim Certified
Arsenic	200.8	Interim Certified
Barium	200.8	Interim Certified
Beryllium	200.8	Interim Certified
Cadmium	200.8	Interim Certified
Chromium	200.8	Interim Certified
Copper	200.8	Interim Certified
Lead	200.8	Interim Certified
Selenium	200.8	Interim Certified
Thallium	200.8	Interim Certified

Parameter/Analyte	Method, 40 CFR	Certification Status
Cyanide (free)	SM4500-CN-F	Interim Certified
Cyanide (total)	335.4	Interim Certified
Fluoride	SM4500F-C	Interim Certified
Mercury	245.2	Interim Certified
Nitrate + Nitrite	SM4500 NO_3 F	Interim Certified
Nitrite	SM4500 NO_2 B	Interim Certified

Metals- ICP	Method, 40 CFR	Certification Status
Calcium	200.7	Interim Certified
Magnesium	200.7	Interim Certified

Volatile Organic Contaminants (VOCs)	Method, 40 CFR	Certification Status
1,1,1-Trichloroethane	524.2	Interim Certified
1,1,2-Trichloroethane	524.2	Interim Certified
1,1-Dichloroethylene	524.2	Interim Certified
1,2,4-Trichlorobenzene	524.2	Interim Certified
1,2-Dichlorobenzene	524.2	Interim Certified
1,2-Dichloroethane	524.2	Interim Certified
1,2-Dichloropropane	524.2	Interim Certified
1,4-Dichlorobenzene	524.2	Interim Certified
Benzene	524.2	Interim Certified
Carbon tetrachloride	524.2	Interim Certified
Chlorobenzene (mono)	524.2	Interim Certified
Cis 1,2-Dichloroethylene	524.2	Interim Certified
Dichloromethane	524.2	Interim Certified
Ethylbenzene	524.2	Interim Certified
Styrene	524.2	Interim Certified

Tetrachloroethylene	524.2	Interim Certified
Toluene	524.2	Interim Certified
trans-1,2-Dichloroethylene	524.2	Interim Certified
Trichloroethylene	524.2	Interim Certified
Vinyl chloride	524.2	Interim Certified
Xylenes (total)	524.2	Interim Certified

Total Trihalomethanes (TTHM)	**Method, 40 CFR**	**Certification Status**
Bromodichloromethane	524.2	Interim Certified
Bromoform	524.2	Interim Certified
Chloroform	524.2	Interim Certified
Dibromochloromethane	524.2	Interim Certified

Synthetic Organic Contaminants (SOCs)	**Method, 40 CFR**	**Certification Status**
1,2-Dibromo-3-chloropropane (DBCP)	504.1	Interim Certified
1,2-Dibromoethane Ethylene dibromide (EDB)	504.1	Interim Certified

Synthetic Organic Contaminants (SOCs)	**Method, 40 CFR**	**Certification Status**
2,4,5-TP (Silvex)	515.4	Interim Certified
2,4-D	515.4	Interim Certified
Dalapon	515.4	Interim Certified
Dinoseb	515.4	Interim Certified
Pentachlorophenol	515.4	Interim Certified
Picloram	515.4	Interim Certified

Synthetic Organic Contaminants (SOCs)	**Method, 40 CFR**	**Certification Status**
Glyphosate	547	Interim Certified

Synthetic Organic Contaminants (SOCs)	**Method, 40 CFR**	**Certification Status**
Alachlor	508.1	Interim Certified
Atrazine	508.1	Interim Certified
Endrin	508.1	Interim Certified
Heptachlor	508.1	Interim Certified
Heptachlor epoxide	508.1	Interim Certified
Hexachlorobenzene	508.1	Interim Certified
Hexachlorocyclopentadiene	508.1	Interim Certified
Lindane	508.1	Interim Certified
Methoxychlor	508.1	Interim Certified
Simazine	508.1	Interim Certified
Technical Chlordane	508.1	Interim Certified
Toxaphene	508.1	Interim Certified

Synthetic Organic Contaminants (SOCs)	**Method, 40 CFR**	**Certification Status**
Carbofuran	531.1	Interim Certified
Oxamyl (Vydate)	531.1	Interim Certified

Haloacetic Acids (HAA5)	Method, 40 CFR	Certification Status
Dibromoacitic acid	552.2	Interim Certified
Dichloroacetic acid	552.2	Interim Certified
Monobromoacetic acid	552.2	Interim Certified
Momochloroacetic acid	552.2	Interim Certified
Trichloroacetic acid	552.2	Interim Certified

Radiochemical Contaminants	Method, 40 CFR	Certification Status
Gamma Emitters, Gamma Ray Spectrometry	(EPA80, 901.1)	Interim Certified
Gross Alpha, Evaporation	(EPA80, 900.0)	Interim Certified
Gross Beta, Evaporation	(EPA80, 900.0)	Interim Certified
Radium-226	(EPA80, 903.0)	Interim Certified
Radium-228	(EPA80, 904.0)	Interim Certified
Strontium-89/90	(EPA80, 905.0)	Interim Certified
Tritium, Liquid Scintillation	(EPA80, 906.0)	Interim Certified
Uranium	(EPA80, 200.8)	Interim Certified

Disinfected Byproducts	Method, 40 CFR	Certification Status
Bromide	300.1	Interim Certified

Microbiological Contaminants	Method, 40 CFR	Certification Status
Total Coliforms, *Escherichia coli*	SM 9223B, Colilert	Interim Certified
Total Coliforms, *Escherichia coli*	SM 9223B, Colilert 18	Interim Certified
Total Coliforms, *Escherichia coli* (P/A)	SM 9223B, Colisure	Interim Certified
Escherichia coli (Enumeration)	SM 9223B, Colilert QT	Interim Certified
Escherichia coli (Enumeration)	SM 9223B, Colilert 18 QT	Interim Certified
Heterotrophic Plate Count	SM 9215B, SimPlate	Interim Certified
Enterococci	SM 9223B, Enterolert	Interim Certified
Enterococci (P/A)	SM 9223B, Enterolert QT	Interim Certified

Parameter/Analyte	Method, 40 CFR	Certification Status
Alkalinity	SM2320	Interim Certified
Orthophosphate	365.1	Interim Certified
Silica	SM4500-SiO2 C	Interim Certified
Total Organic Carbon	SM5310 C	Interim Certified
Dissolved Organic Carbon	SM5310 C	Interim Certified
SUVA UV Absorbance at 254 NM	SM5310 B	Interim Certified

Appendix D

Barr Data Review Standard Operating Procedures

Standard Operating Procedure

Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 6

January 15, 2016

Approved By:

Michael Dupay		01/15/16
Print Technical Reviewer	Signature	Date

Terri Olson		01/15/16
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Routine Level Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of VOC, GRO, and TPH data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine VOC (including BTEX), GRO, and TPH (in the approximate gasoline carbon range, C_6-C_{10}) data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, WI GRO (GRO)
- Gas Chromatography/Photoionization Detector (GC/PID)
  - Method example: EPA 8021, WI GRO (PVOC)
- Gas Chromatography/Electrolytic Conductivity Detector (GC/ELCD)
  - Method example: EPA 8021
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 624, EPA 8260
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8260
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
VOC/PVOC	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Aqueous	≤ 6 °C	Unpreserved	7 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	14 days
GRO (WI Method)	Aqueous	≤ 6 °C	HCl < 2 pH	14 days
	Sediment/Soil	≤ 6 °C	1:1 soil:solvent (e.g., 10 g soil:10 mL MeOH in lab pre-weighed vial)	21 days

(Table 1 continued on next page)

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Table 1 – Recommended Holding Times and Preservation				
Compound	**Matrix**	**Temp.**	**Preservative**	**Maximum Hold Time**
TPH	Aqueous	≤ 6 °C	HCl or H_2SO_4 < 2 pH	7 day extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6 °C	Zero headspace*	14 days extraction/ addl. 40 days analysis
TCLP	Various	≤ 6 °C	No preservative	14 days TCLP extraction/ addl. 14 days analysis

* = Alternatively, samples may be collected as per the VOC analysis.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG) – laboratories should analyze a method blank at least once every 12 hours. Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Trip blanks should be placed in each transport cooler containing VOC sample containers prior to shipment into the field and remain with the associated VOC samples submitted to the laboratory for VOC analysis; including sample storage through analysis.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination	
Sample Result	**Recommended Action for Associated Data**
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

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Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the VOC GC/MS analysis. *Table 3* presents the recommended DMCs with their associated target compounds.

Table 3 –DMC and Associated Target Compounds

DMC (alphabetical)	Associated Target Compounds	
1,1,2,2-Tetrachloroethane-d_2	1,1,2,2-Tetrachloroethane	1,2-Dibromo-3-chloropropane
1,1-Dichloroethane-d_2	trans-1,2-Dichloroethene 1,1-Dichloroethene	cis-1,2-Dichloroethene
1,2-Dichlorobenzene-d_4	Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene	1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
1,2-Dichloroethane-d_4	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether	1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
1,2-Dicloropropane-d_6	Cyclohexane Methylcyclohexane	1,2-Dichloropropane Bromodichloromethane
1,4-Dioxane-d_8	1,4-Dioxane	
2-Butanone-d_5	Acetone	2-Butanone
2-Hexanon-d_5	4-Methyl-2-pentanone	2-Hexanone
Benzene-d_6	Benzene	
Chloroethane-d_5	Dichlorodifluoromethane Chloromethane Bromomethane	Chloroethane Carbon disulfide
Chloroform-d	1,1-Dichloroethane Bromochloromethane Chloroform	Dibromochloromethane Bromoform
Toluene-d_8	Trichloroethene Toluene Tetrachloroethene Ethylbenzene	o-Xylene m,p-Xylene Styrene Isopropylbenzene
trans-1,3-Dichloropropene-d_4	cis-1,3-Dichloropropene trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Vinyl Chloride-d_3	Vinyl chloride	

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Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMCs or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMCs or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI GRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of the DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

For the WI GRO analysis, surrogates are not required for GRO but are required for PVOC. The method minimum surrogate recovery is 80%; there is no method maximum recovery. Use professional judgment when evaluating surrogates for WI GRO samples.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

Table 4 – Guidelines for Single DMC or Surrogate

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met
'**' = reported value is unusable and QA/QC criteria were not met

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

Table 5 – Guidelines for Multiple DMC or Surrogates

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with '*'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met
'**' = reported value is unusable and QA/QC criteria were not met

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4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix - WI GRO requires an additional LCSD analyzed at the end of 20 samples)
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset (see *Table 6* for guidance) and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 6 – Number of Suggested Target Compounds - LCS/LCSD and MS/MSD

Number of Target Parameters	Number of Spiked Compounds
1-10 analytes	Spike all compounds
11-20 analytes	At least 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike at least 16 compounds

Table 7 – Guidelines for Laboratory Control Samples

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are

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not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 8 – Guidelines for Laboratory Duplicates

% RPD	Recommended Action for Associated Data
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

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4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset (see *Table 6*) and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to GRO in the WI method)
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 9 – Guidelines for Matrix Spikes

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

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4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3*.

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	06/17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
		IX	Added statement regarding multiple qualifiers
6.0	01/15/16	Document Wide	SOP restructuring, new format

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Standard Operating Procedure

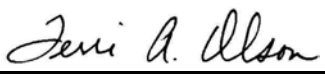
Routine Level Volatile Organic Compounds (VOC) Air Data Evaluation

Revision 0

April 6, 2016

Approved By:

Dana Pasi		04/06/16
Print Technical Reviewer	Signature	Date

Terri Olson		04/06/16
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Routine Level Volatile Organic Compounds (VOC) Air Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of VOC air data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine VOC air data evaluation for analyses by the following technologies:

- Gas Chromatography/Mass Spectrometry (GC/MS)
    - Method example: TO-15

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the

laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

The methods and regulatory guidance documents (e.g., Minnesota Pollution Control Agency (MPCA) *Vapor Intrusion Assessments Performed During Site Investigations* and Michigan Department of Environmental Quality (MDEQ) *For the Vapor Intrusion Pathway* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
VOC (TO-15)	Air	NA	Certified clean* canister or Bottle-Vac®	14 days (MPCA), 30 days (TO-15, MDEQ)

* Certified clean (<0.2 ppbv) can be batch or individual

If samples do not meet analysis recommendations for holding time in *Table 1*, consider qualification with an "**h**". Professional judgment should be applied (considering matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- One method blank is analyzed per batch (typically 20 or less samples) and consists of an unused canister or Bottle-Vac® that has not left the laboratory and has been carried through the same analytical procedure as a field sample.

Table 2 – Guidelines for Blank Contamination

Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 10x blank concentration	Qualify with 'b'
≥ 10x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other factors of the blank contamination may be used based on professional judgment (reporting the MDL, common lab contaminant, etc.)

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Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Surrogates

Surrogates are not required for the TO-15 analysis.

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- One LCS every batch (typically 20 or less samples)

Laboratory control samples percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use applicable regulatory guidance, if available. Percent recoveries are calculated for accuracy and the relative difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 3 – Guidelines for Laboratory Control Samples

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed:

- One in every ten samples (10%)

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Laboratory or project specific requirement are used to evaluate RPDs. If criteria are not available, use applicable regulatory guidance or professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 4 – Guidelines for Laboratory Duplicates	
% RPD	**Recommended Action for Associated Data**
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 40% are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples are not required for TO-15.

4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other

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supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

Some regulatory agencies have additional requirements within their guidance documents on what data shall be included with each laboratory report. The report should be reviewed for completeness based on these items. Examples of additional data/items that shall be within the report include but are not limited to: chemical abstract service (CAS) number of each reported compound, results reported in µg/m^3, results of the top five or ten tentatively identified compounds >5 ppbv), narrative stating if initial calibration curves, calibration checks, and internal standards met method requirements, assigned regulator, flow rate, and labeled chromatograms. In addition to these items, if the canister or Bottle-Vac® was individually certified clean (< 0.2 ppbv), the results of the testing should be included.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

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Minnesota Pollution Control Agency, Petroleum Remediation Program. October 2010. *Guidance Document 4-01a, Vapor Intrusion Assessments Performed During Site Investigations*.

Michigan Department of Environmental Quality, Remediation and Redevelopment Division. May 2013. *Guidance Document For the Vapor Intrusion Pathway*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
0	04/06/16	Entire SOP	New document

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Standard Operating Procedure

Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

Revision 6

January 19, 2016

Approved By:

Michael Dupay		01/19/16
Print Technical Reviewer	Signature	Date

Terri Olson		01/19/16
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Routine Level Semivolatile Organic Compounds (SVOC), Polycyclic Aromatic Hydrocarbons (PAH), Diesel Range Organics (DRO), and Total Petroleum Hydrocarbons (TPH) Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of semivolatile organic compounds data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine SVOC (including PAHs and phenols), TPH at various carbon ranges (e.g., TPH as fuel oil, TPH as motor oil, TPH as jet fuel), and DRO data evaluation for analyses by the following technologies:

- Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Method examples: EPA 8015, EPA 8100, WI DRO
- Gas Chromatography/Mass Spectrometry (GC/MS)
  - Method example: EPA 625, EPA 8270
- Gas Chromatography/Mass Spectrometry-Selective Ion Monitoring (GC/MS-SIM)
  - Method example: EPA 8270
- High Performance Liquid Chromatography (HPLC)
  - Method example: EPA 610, EPA 8310
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136, WI GRO method, and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
SVOC/PAH/TPH	Aqueous	≤6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis
DRO	Aqueous	≤ 6° C	Ice, HCl < 2 pH	7 days extraction/ 47 days collection to analysis
	Sediment/Soil	≤ 6° C	Ice	10 days solvent addition/ 47 days collection to extraction and analysis
TCLP SVOC	Various	--	NA	14 days TCLP extraction/ 7 days extraction/ addl. 40 days analysis

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If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination

Sample Result	Recommended Action for Associated Data
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Deuterated Monitoring Compounds (DMC) and Surrogates

DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are only used for the SVOC GC/MS analysis. *Table 3* presents the recommended DMCs with their associated target compounds.

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Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	**Associated Target Compounds**	
2,4-Dichlorophenol-d_3	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol	2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
2-Chlorophenol-d_4	2-Chlorophenol	
2-Nitrophenol-d_4	Isophorone	2-Nitrophenol
4-6-Dinitro-2-methylphenol-d_2	4,6-Ditritro-2-methylphenol	
4-Chloroaniline-d_4	4-Chloroaniline Hexachlorocyclopentadiene	3,3′-Dichlorobenzidine
4-Methylphenol-d_8	2-Methylphenol 4-Methylphenol	2,4-Dimethylphenol
4-Nitrophenol-d_4	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol	4-Nitrophenol 4-Nitroaniline
Acenaphthylene-d_8	Naphthalene 2-Methylnaphthalene 2-Chloronapthalene	Acenaphthylene Acenaphthene
Anthracene-d_{10}	Hexachlorobenzene Atrazine	Phenanthrene Anthracene
Benzo(a)pyrene-d_{12}	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
Bis-(2-chloroethyl) ether-d_8	Bis-(2-chloroethyl) ether 2,2′-oxybis(1-chloropropane)*	bis(2-Choloethoxy) methane
Dimethylphthalate-d_6	Caprolactum 1,1′-Biphenyl Dimethylphthalate Diethylphthalate	Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d_{10}	Dibenzofuran Fluorene 4-Chlorophenyl-phenylether	4-Bromophenyl-phenylether Carbazole
Nitrobenzene-d_5	Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene	2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosdiphenylamine

(Table 3 continued on next page)

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Table 3 – DMC and Associated Target Compounds		
DMC (alphabetical)	**Associated Target Compounds**	
Phenol-d_5	Benzaldehyde	Phenol
Pyrene-d_{10}	Fluoranthrene Pyrene	Benzo(a)anthracene Chrysene
SIM DMC and Associated Target Compounds		
Fluoranthene-d_{10}	Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthene	Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
2-Methylnaphthalene-d_{10}	Naphthalene 2-Methylnaphthalene Acenaphthylene Acenaphthene	Fluorene Pentachlorophenol Phenanthrene Anthracene

* = Bis(2-chloroisopropyl)ether

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other DMC or surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. All samples (blanks, spiked samples, project samples, QC samples) should contain DMC or surrogates. If a sample does not contain DMC or surrogates or the method does not require surrogates (WI DRO), professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of DMC or surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each DMC or surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

For the WI DRO analysis, surrogates are not required by the method. If used, the method requires that the surrogates must not elute within the WI DRO window (C_{10}-C_{28}). If the laboratory report includes a surrogate spike recovery for WI DRO, use professional judgment to assess the data.

Table 4 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

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Table 4 – Guidelines for Single DMC or Surrogate		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met
'**' = reported value is unusable and QA/QC criteria were not met

Table 5 includes guidance where multiple surrogates are analyzed per analytical fraction.

Table 5 – Guidelines for Multiple DMC or Surrogates		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with '*'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met
'**' = reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (20 or less samples of the same matrix - WI DRO requires an additional LCSD analyzed at the end of 20 samples).
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset (see *Table 6* for guidance) and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

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Table 6 – Number of Suggested Target Compounds - LCS/LCSD and MS/MSD	
Number of Target Parameters	**Number of Spiked Compounds**
1-10 analytes	Spike all compounds
11-20 analytes	At least 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike at least 16 compounds

Table 7 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable

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concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 8 – Guidelines for Laboratory Duplicates	
% RPD	**Recommended Action for Associated Data**
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset (see *Table 6*) and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples (does not apply to DRO in the WI method)
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

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If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 9 – Guidelines for Matrix Spikes

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the

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sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3*.

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
3.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		IX	Added Table 10
		Attachments	Added Attachment 3
3.2	04/2011	Document Wide	Added analytical methods to applicability section.
		Attachments	Updated Attachment 1 and 2 to include current forms.
4.0	04/06/12	Document Wide	Major revision
5.0	0601//17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI, VII	Added 'project specific requirements' as possible criteria source
		VI	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VI	Clarified field duplicate criteria as < one value and not a range
		IX	Added statement regarding multiple qualifiers
6.0	01/19/16	Document Wide	SOP restructuring, new format

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Standard Operating Procedure

Routine Level Radium 226 and 228 Data Evaluation

Revision 0

September 28, 2017

Approved By:

Andrea Nord		09/28/17
Print Technical Reviewer	Signature	Date

Terri Olson		09/28/17
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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Routine Level Radium 226 and Radium 228 Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of Radium 226 and Radium 228 data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG)* and applies to Radium 226 and Radium 228 data evaluation for analyses by the following methods:

- EPA 903.1, EPA 904.0, EPA 9315, EPA 9320, EPA EMSL-19, SM 7500-Ra B, SM7500-Ra D, Georgia Technical Research Institute

In the case of specific methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data validation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136 and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
Radium 226, Radium 228	Aqueous	---	HNO_3 < 2 pH*	6 months
Radium 226, Radium 228	Solid	≤ 6 °	NA	14 days

* = Per SM 7010B, chemical preservative should be added at the time of collection but not delayed beyond 5 days from collection. At least sixteen (16) hours must elapse between acidification and analysis.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**".

Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Assessment of Detections

Prior to review of the QC data, determine if a result was detected or not detected by comparing the result to the Minimum Detectable Concentration (MDC) and the uncertainty.

The MDC is the minimum detectable activity (MDA) expressed in concentration units relative to the sample weight or volume and is the smallest concentration of radioactivity in a sample that can be detected with a 5 % probability of erroneously detecting radioactivity, when in fact none was present and also, a 5 % probability of not detecting radioactivity when in fact it is present.

Uncertainty is the degree of inaccuracy and imprecision associated with a measured quantity. It must be reported to determine if the result was detected or not detected. It may also be called counting uncertainty and is defined as the statistical sample standard deviation, which is an approximation of the population standard. Units for counting uncertainty should be the same as for the reported result and the MDC. The uncertainty is typically reported at 2 standard deviation (95% confidence level). If the uncertainty confidence level is not provided in the laboratory report, it should be confirmed with the lab. The uncertainty used below assumes 2s (95% confidence level).

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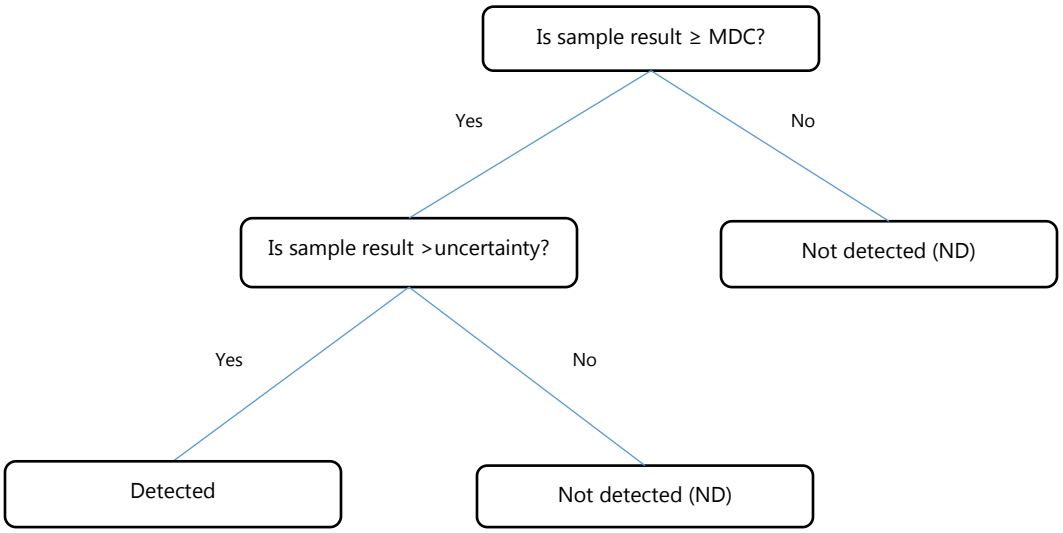
Reporting of results can vary by laboratory. The laboratory report should include:

- Minimum Detectable Concentration (MDC)
- Sample result concentration and sample result uncertainty
- QC data (e.g., method blank, laboratory control sample (LCS), matrix spike (MS), matrix spike duplicate (MSD), and/or laboratory duplicate sample results

The test for detection includes two distinct steps:

1. Is the sample result ≥ MDC?
2. Is the sample result > uncertainty?

See flow chart below:



- If the sample result is < MDC, accepting probability of a 5% false negative result (assuming MDC at 95%).
- If the sample result is < uncertainty, the radionuclide is not different than zero at the 95% confidence level.

Examples:

Sample Result ± Uncertainty	MDC	Unit	Detected or Not Detected?
0.5 ± 0.2	0.3	pCi/L	Detected
0.5 ± 0.6	0.3	pCi/L	Not detected
0.5 ± 0.2	0.6	pCi/L	Not detected

If the MDC was not included, but a reporting limit was provided, use this value to determine if the result was detected or not detected. Without this information, the determination of detected or not detected (ND) cannot be performed.

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4.3 Blank Samples

Blank sample evaluation is conducted after assessment of detections to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each batch. Evaluation pertains to the samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank evaluation is performed by calculating the normalized absolute difference between the highest detected blank concentration associated with a group of samples and the detected sample concentration.

$$Normalized\ Absolute\ Difference\ (NAD) = \frac{|S - B|}{\sqrt{U_S^2 + U_B^2}}$$

Where:
S = Sample result
B = Blank result
U = Uncertainty

The method blank result should include the uncertainty. If any of the equation variables are missing, the NAD equation cannot be used. Qualify samples results < 2x the blank concentration.

Table 2 – Guidelines for Blank Contamination

Result	Recommended Action for Associated Data
Sample or MB not detected	No action required
NAD < 1.96 or < 2x blank concentration	Qualify with 'b'
NAD ≥ 1.96 or ≥ 2x blank concentration	No action required

b = Reported value may be a potential false positive based on blank data evaluation procedures

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix).

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent

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recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 3 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.5 Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for laboratory duplicate analysis.

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific.

Duplicate evaluation is performed by calculating the NAD (sometimes referred to as Relative Error Ratio (RER) in laboratory reports) using the equation under the blank section but substituting the duplicate result for the blank sample result. The NAD is typically only evaluated where both the native and duplicate sample results are detected and where data are not already qualified with b, U, <, or **. In cases where either of the samples (native or duplicate) is not detected and the other corresponding sample has a detectable concentration, the NAD may still be calculated but professional judgment should be used to determine if qualification is appropriate.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates when evaluating precision.

Table 4 – Guidelines for Duplicates	
NAD	**Recommended Action for Associated Data**
NAD ≤ 1.96	No action is required
NAD > 1.96	Qualify laboratory source or native and field duplicate with '*'

* = Reported value is estimated and QA/QC criteria were not met

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4.6 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset (see *Table 6*) and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 5 – Guidelines for Matrix Spikes

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the NAD for MS and MSD pairs using the equation as provided under duplicate samples.

4.7 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

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5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with the data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

J.G. Paar, University of TN, Knoxville/Oak Ridge National Laboratory and D. R. Porterfield, Chemical Science and Technology Division Los Alamos National Laboratory. April 1997. *Evaluation of Radiochemical Data Usability.*

Environmental Protection Agency, *National Functional Guidelines.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Standard Operating Procedure

Routine Level Polychlorinated Biphenyl (PCB), Aroclor, Pesticide, and Herbicide Data Evaluation

Revision 4

January 22, 2016

Approved By:

Michael Dupay		01/22/16
Print Technical Reviewer	Signature	Date

Terri Olson		01/22/16
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

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Routine Level Polychlorinated Biphenyl (PCB), Aroclor, Pesticide, and Herbicide Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of polychlorinated biphenyl (PCB), Aroclor, pesticide, and herbicide data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine level PCB, Aroclor, pesticide, and herbicide data evaluation for analyses by the following technologies:

- Gas Chromatography/Electron Capture Detector (GC/ECD)
  - Method examples: EPA 608, EPA 8081, EPA 8082, EPA 8151
- Gas Chromatography/Electrolytic Conductivity Detector (GC/ELCD)
  - Method examples: EPA 8081, EPA 8082
- Gas Chromatography/Flame Photometric Detector (GC/FPD)
  - Method example: EPA 1657, EPA 8141
- Gas Chromatography/Nitrogen Phosphorus Detector (GC/NPD)
  - Method example: EPA 8141
- GC/ECD for Herbicides
  - Method example: EPA 8151
- Methods above with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136 and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
PCBs (EPA 608)	Aqueous	≤ 6° C	Ice	1 year extraction/ addl. 1 year analysis
Organochlorine Pesticides (EPA 608)	Aqueous	≤ 6° C	Ice (if >72 hrs. to extraction, preserve to pH 5-9 with NaOH and/or H_2SO_4)	72 hrs. extraction unpreserved, 7 days extraction preserved/ addl. 40 days analysis
Organochlorine Pesticides (EPA 8081)	Aqueous	≤ 6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis

(Table 1 continued on next page)

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Table 1 – Recommended Holding Times and Preservation				
Compound	**Matrix**	**Temp.**	**Preservative**	**Maximum Hold Time**
Organochlorine Pesticides (EPA 8081)	TCLP	--	NA	14 days TCLP extraction/ 7 days extraction/ addl. 40 days analysis
PCBs/Aroclor (EPA 8082)	Aqueous	≤ 6° C	Ice	None
	Sediment/Soil	≤ 6° C	Ice	None
Organophosphorus Compounds (EPA 8141)	Aqueous and Sediment/Soil	≤ 6° C	Ice	7 days extraction/ addl. 40 days analysis
Herbicides (EPA 8151)	Aqueous	≤ 6° C	Ice	7 days extraction/ addl. 40 days analysis
	Sediment/Soil	≤ 6° C	Ice	14 days extraction/ addl. 40 days analysis

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. It may be easier to work with the raw data and/or convert the data to the same units for comparison purposes.

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Table 2 – Guidelines for Blank Contamination	
Sample Result	**Recommended Action for Associated Data**
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Surrogates

Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. All samples (blanks, spiked samples, project samples, QC samples) should contain surrogates. If a sample does not contain surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not reported, use guidance found in the NFG, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 3 includes guidance to evaluate the surrogate recovery where a single surrogate is analyzed.

Table 3 – Guidelines for Single Surrogate		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R within Limits	No qualification	

'*' = reported value is estimated and QA/QA criteria were not met

'**' = reported value is unusable and QA/QC criteria were not met

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Table 4 includes guidance where multiple surrogates are analyzed.

Table 4 – Guidelines for Multiple Surrogates		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Two or more %R > Upper Limit	Qualify fraction with '*'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'*' = Reported value is estimated and QA/QA criteria were not met
'**' = Reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix).
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset (see *Table 5* for guidance) and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 5 – Number of Suggested Target Compounds - LCS/LCSD and MS/MSD	
Number of Target Parameters	**Number of Spiked Compounds**
1-10 analytes	Spike all compounds
11-20 analytes	At least 10 compounds or 80% of all analytes, whichever is greater
More than 20 analytes	Spike at least 16 compounds

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Table 6 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

'*' = Reported value is estimated and QA/QA criteria were not met
'**' = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 7 – Guidelines for Laboratory Duplicates	
% RPD	**Recommended Action for Associated Data**
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

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4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples may contain all target compounds or a subset (see *Table 5*) and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

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Table 7 – Guidelines for Matrix Spikes		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

'*' = Reported value is estimated and QA/QA criteria were not met
'**' = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

Note: Pesticides, herbicides, PCBs and Aroclors require additional ECD or GC/MS confirmation of tentatively identified compounds (TIC), using a separate column. This may occur at the same time as the initial analysis using a dual-column GC with an additional detector; or a second, separate analysis via EPA 8270 (see Barr SOP for Routine Level SVOC Data Evaluation if positive detections occur). Herbicides are sufficiently identified by a single column if a GC/MS is used for analysis. If there is indication that conformational analysis was not performed for the remaining parameters, professional judgment should be used to critically evaluate the usability of the data as reported.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

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The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3*.

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
1.1	02/2009	Document Wide	Edits to references, formatting; minor language additions and corrections
		Attachments	Added Attachment 3
1.2	04/2011	Attachments	Updated Attachment 1 and 2 to current forms.
2.0	04/06/12	Document Wide	Major revision
3.0	06/17/13	Cover page	Added Calgary office
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI	Added 'project specific requirements' as possible criteria source
		V	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		V	Clarified field duplicate criteria as < one value and not a range
		VIII	Added statement regarding multiple qualifiers
4	01/22/16	Document Wide	SOP restructuring, new format

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Standard Operating Procedure

Routine Level Polycyclic Aromatic Hydrocarbons (PAH) Air Data Evaluation

Revision 0

April 6, 2016

Approved By:

Michael Dupay		04/06/16
Print Technical Reviewer	Signature	Date

Terri Olson		04/06/16
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

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Routine Level Polycyclic Aromatic Hydrocarbons (PAH) Air Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of PAH air data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data* and applies to routine PAH air data evaluation for analyses by the following technologies:

- Gas Chromatography/Mass Spectrometry (GC/MS)
    - Method example: TO-13A

In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the

laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

The methods are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Hold Time
PAH (TO-13A)	Air	≤ 4 °C	Blue ice, gel ice, or dry ice, protect from light	7 days extraction/ addl. 40 days analysis

If samples do not meet holding time, preservation and extraction/analysis recommendations in *Table 1*, consider qualification with an "**h**".

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- One method blank is prepared and analyzed per batch (typically 20 or less samples) and consists of a filter and cartridge that has not left the laboratory and has been carried through the same analytical procedure as a field sample. Evaluation pertains to the batch of samples analyzed with the method blank.
- One solvent blank is carried through the same analytical procedure as the method blank except it does not contain a filter and cartridge.
- Field blank collection and analysis frequency is project specific but typically consists of one blank filter and cartridge that was shipped to the field and returned to the laboratory with each group of samples, without drawing air through the sampler. Evaluation pertains to the field samples associated with the field or equipment, blank.

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Table 2 – Guidelines for Blank Contamination	
Sample Result	**Recommended Action for Associated Data**
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other factors of the blank contamination may be used based on professional judgment (reporting the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Deuterated Monitoring Compounds (DMC)

DMCs are isotopically labeled (deuterated) analogs of native target compounds. For TO-13A, DMCs are used as surrogates. Surrogates are similar to analytes of interest in chemical composition, extraction, and chromatography but are not typically found in environmental samples. Other surrogates may be used by a laboratory based on their experience provided adequate chromatographic separations can be demonstrated. TO-13A uses both field and laboratory surrogate compounds. Field surrogates are added to each cartridge prior to sending out in the field to monitor matrix effects, breakthrough, etc. Laboratory surrogates are used to monitor for extraction effects, unusual matrix effects, gross sample processing errors, etc. *Table 3* presents the surrogate type with their associated surrogate compounds.

Table 3 – DMC/Surrogate Type		
Surrogate Type	**Associated Surrogate Compounds**	
Field	Benzo(a)pyrene-d12	Fluoranthene-d10
Laboratory	Fluorene-d10	Pyrene-d10

All samples (blanks, project samples, QC samples) should contain surrogates. If a sample does not contain surrogates, professional judgment should be used to determine if the reported results are useable or not. Acceptable evaluation of the surrogate spikes may not be applicable if dilution of the sample was required. Percent recoveries are calculated for each surrogate and these are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use applicable regulatory guidance, if available. Percent recoveries are calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 4 includes guidance where multiple surrogates are analyzed.

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Table 4 – Guidelines for Multiple DMCs/Surrogates		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
One %R < Lower Limit	No qualification may be necessary, use professional judgment	
Two or more %R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Two or more %R > Upper Limit	Qualify with '*'	No qualification
One %R > Upper Limit	No qualification may be necessary, use professional judgment	No qualification
All %R within Limits	No qualification	

'*' = Reported value is estimated and QA/QA criteria were not met
'**' = Reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- One LCS every batch (typically 20 or less samples)

Laboratory control samples percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use applicable regulatory guidance, if available. Percent recoveries are calculated for accuracy and the relative difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 5 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.5 Laboratory Duplicate Samples

Laboratory duplicate samples are not required for TO-13A.

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4.6 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 50% are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.7 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples are not required for TO-13A.

4.8 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

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6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *National Functional Guidelines for Superfund Organic Methods Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
0	04/06/16	Entire SOP	New document

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Standard Operating Procedure

Routine Level Metals Data Evaluation

Revision 7

April 24, 2018

Approved By:

Dana Pasi		04/24/18
Print Technical Reviewer	Signature	Date

Terri Olson		04/24/18
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

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Routine Level Metals Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance document for the routine level evaluation of metals data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Inorganic Data* and applies to routine metals data evaluation for analyses by the following technologies:

- Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES)
    - Method examples: EPA 200.7, EPA 6010
- Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
    - Method examples: EPA 200.8, EPA 6020
- Cold Vapor Atomic Absorption (CVAA)
    - Method examples: EPA 245.1, EPA 7470, EPA 7471, SM 3112 B
- Cold Vapor Atomic Fluorescence Spectrometry (CVAF)
    - Method examples: EPA 245.7, EPA 1631 (low-level mercury), EPA 7474
- Thermal Decomposition / Atomic Absorption Spectrophotometer
    - EPA 7473
- Graphite Furnace Atomic Absorption (GFAA)
    - Method examples: EPA 7010, SM 3113 B
- Methods above in conjunction with Toxicity Characteristic Leachate Procedure (TCLP), EPA 1311
- Methods above in conjunction with Synthetic Precipitation Leachate Procedure (SPLP), EPA 1312

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards, post digestion, serial dilution).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136 and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation

Compound	Matrix	Temp.	Preservative	Maximum Holding Time
Mercury	Aqueous	--	HNO_3 < 2 pH	28 days
	Aqueous (low level)	--	Pre-tested hydrochloric acid or bromine chloride	48 hours preserve or analyze if not oxidized in sample bottle/28 days preserve if oxidized in sample bottle 90 days analysis (from collection) if preserved
	Sediment/Soil	Cool, ≤ 6 °C	Ice	28 days
	Wipe/Air	--	NA	28 days

(Table 1 continued on next page)

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Table 1 – Recommended Holding Times and Preservation				
Compound	**Matrix**	**Temp.**	**Preservative**	**Maximum Holding Time**
Mercury	TCLP	--	NA	28 days TCLP Extraction/ 28 days analysis
All other metals	Aqueous	--	HNO_3 < 2 pH	180 days
	Sediment/Soil	Cool, ≤ 6 °C	Ice	180 days
	Wipe/Air	--	NA	180 days
	TCLP	--	NA	180 days TCLP Extraction/ 180 days analysis

Note: When analyzing boron or silica, do not collect samples in borosilicate glass bottles.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

Special considerations for low-level mercury

Low-level mercury must be collected directly into a specially cleaned, pretested, fluoropolymer or glass bottle using sample handling techniques specially designed for collection of mercury at trace levels and preserved with pre-tested hydrochloric acid (required for methyl mercury) or bromine chloride. Samples not collected in the correct type of container may be qualified with an "**h**". These samples may be shipped unpreserved provided:

- Sample is collected in a fluoropolymer or glass bottle.
- Bottle contains no headspace and is capped tightly.
- Sample temperature was maintained at ≤ 6 °C.
- Samples are preserved or analyzed within 48 hours or oxidized in the bottle within 28 days.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG), or each batch digested (whichever is more frequent). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment, blank.

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- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. Data reviewers may have to obtain raw data and/or convert the data to the same units for comparison purposes.
- Low-level mercury method requires *at least* three method blanks per run per analytical batch.

Table 2 – Guidelines for Blank Contamination	
Sample Result	**Recommended Action for Associated Data**
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix).
- Once for each matrix.
- For low-level mercury, ongoing precision and recovery (OPR) samples are run before and after each analytical batch - quality control samples (QCS) should be from a different source and analyzed once per analytical batch.

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

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Table 3 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.4 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use guidance found in NFG or use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 4 – Guidelines for Laboratory Duplicates	
% RPD	**Recommended Action for Associated Data**
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

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4.5 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.6 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples contain a known amount of a target compound and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

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Table 5 – Guidelines for Matrix Spikes		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

4.7 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

4.8 Total vs. Dissolved

Occasionally, the measurements for dissolved metals are equivalent to or greater than the associated results reported for the total metals analysis. When this occurs, the variation between the total and dissolved results may indicate that the majority of the target metals present in the sample were in the dissolved phase and normal analytical variability may account for the difference. Professional judgment should be used to determine if the variation is significant enough to be qualified.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

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The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, *National Functional Guidelines for Inorganic Superfund Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1
Revision History

Revision Number	Date of Revision	Section	Revision Made
5.0	06/17/13	Cover page	Added Calgary office
		Applicability	Added US to EPA reference
		I	Added waste rock and drill cores to examples of product sample
		III, IV, V, VI	Added 'project specific requirements' as possible criteria source
		V	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		V	Clarified field duplicate criteria as < one value and not a range
		VIII	Added statement regarding multiple qualifiers
6.0	01/07/16	Document Wide	SOP restructuring, new format
7	04/24/18	1.0	Added letter indicator for the various EPA method revisions was intentional omitted; multiple versions of the approved methods would be applicable for review under this SOP.
			Added laboratories may not provide all the review elements in this SOP, review only those that are provided.
		4.2, third bullet	Clarified that data reviewers would have to obtain raw data since not provided with Level II report.

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Standard Operating Procedure

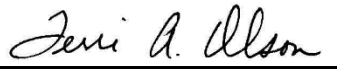
Routine Level General Chemistry Data Evaluation

Revision 7

April 24, 2018

Approved By:

Dana Pasi		04/24/18
Print Technical Reviewer	Signature	Date

Terri Olson		04/24/18
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Initials: _____ Date: _____

Routine Level General Chemistry Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance document for the routine level evaluation of general chemistry data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on the recommendations of the associated approved analytical methods from USEPA, ASTM, and *Standard Methods for the Examination of Water and Wastewater* and applies to routine general chemistry data evaluation including a variety of approved methods not limited to the following parameters:

Alkalinity as $CaCO_3$	Nitrate (or Nitrite) only
Ammonia, total (NH_3 + NH_4^-)	Nitrate + Nitrite
Biological Oxygen Demand (BOD)	pH – *in lab*
Chemical Oxygen Demand (COD)	Phosphorus, total
Chloride	Sulfate
Chromium VI (Hexavalent Chromium)	Sulfide
Conductance, Specific – *in lab*	Total Dissolved Solids (TDS)
Cyanide (as CN^-)	Total Kjeldahl Nitrogen (TKN)
Fluoride	Total Organic Carbon (TOC)
Hardness	Total Suspended Solids (TSS)
Oil and Grease (as HEM)	

In the case of specific parameters not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with project specific requirements.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards, post digestion, serial dilution).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

40 CFR Part 136 and the *Test Methods for Evaluating Solid Waste (SW-846)* are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 - Recommended Holding Times and Preservation

Parameter *(Alternate Name)*	**Recommended Hold Time**						**Preservation**					
	24 Hour	48 Hour	7 Day	14 Day	28 Day	180 Day	Ice Only (< 6 °C)	HCl	HNO_3	H_2SO_4	NaOH	ZnAc + NaOH
Alkalinity, as $CaCO_3$				X			X					
Ammonia as N					X		X			X		
Biochemical Oxygen Demand *(BOD)*		X					X					
Chemical Oxygen Demand *(COD)*					X		X			X		
Chloride					X		X					
Chromium, hexavalent	X				a		X					
Conductance, specific - in lab					X		X					
Cyanide				X			X				X	
Dissolved Organic Carbon *(DOC)*					X		X	X[c]		X[c]		
Fluoride					X		X					
Hardness						X			X[c]	X[c]		

(Table 1 continued on next page)

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Table 1 - Recommended Holding Times and Preservation												
Parameter *(Alternate Name)*	**Recommended Hold Time**						**Preservation**					
	24 Hour	48 Hour	7 Day	14 Day	28 Day	180 Day	Ice Only (< 6 °C)	HCl	HNO_3	H_2SO_4	NaOH	ZnAc + NaOH
Nitrate or Nitrite		X					X					
Nitrate + Nitrite as N					X		X			X		
Oil & Grease, HEM					X		X	X[c]		X[c]		
pH[b] - in lab			X				X					
Phosphorus, total					X		X			X		
Sulfate					X		X					
Sulfide			X				X					X
Total Dissolved Solids *(TDS)*			X				X					
Total Kjeldahl Nitrogen *(TKN)*					X		X			X		
Total Organic Carbon *(TOC)*					X		X	X[c]		X[c]		
Total Suspended Solids *(TSS)*			X				X					

a = Per 40 CFR Part 136.3, a 28-day holding time may be achieved if the ammonium sulfate buffer solution specified in EPA Method 218.6 is used. This footnote supersedes preservation and holding time requirements in approved hexavalent chromium methods, unless this would compromise the measurement and then the method must be followed.
b = Method recommends pH should be measured in the field.; however, for confirmation measurements in the laboratory, a maximum holding time of 7 days from sample collection will be used as a guideline for qualification.
c = Either preservative may be used (pH < 2) - for hardness, HNO_3 only if calculated from Ca and Mg.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- While not required for all methods, method blanks are recommended for all but the pH analysis. Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. Data reviewers may have to obtain raw data and/or convert the data to the same units for comparison purposes.

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Table 2 – Guidelines for Blank Contamination	
Sample Result	**Recommended Action for Associated Data**
Non-detect	No action required
< 5x blank concentration	Qualify with 'b'
≥ 5x blank concentration	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

Professional judgment regarding the usability of the data should be used in cases where gross detections of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (typically 20 or less samples of the same matrix).
- Once for each matrix.

Laboratory control samples contain a known amount of each target compound and the percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Table 3 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

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4.4 Laboratory Duplicate Samples

Laboratory duplicate samples are separate aliquots of field samples analyzed to demonstrate acceptable method precision by the laboratory at the time of analysis. Field blanks and proficiency testing (PT) samples should not be used for duplicate analysis. The RPDs are calculated using the equation as provided in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data are already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Duplicates should be analyzed (whichever is more frequent):

- One from each matrix (soil or water)
- One from each SDG

The MS/MSD duplicate pairs may be substituted for laboratory duplicates.

Laboratory acceptance criteria or project specific requirement are used to evaluate RPDs. If criteria are not available, use professional judgment when considering qualification of associated results.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

Table 4 – Guidelines for Laboratory Duplicates

% RPD	Recommended Action for Associated Data
RPD < Upper Limit	No action is required
RPD > Upper Limit	Both results are ≤ 5x RL, no action is required
RPD > Upper Limit	Both results are > 5x RL, consider qualifying with '*****'

* = Reported value is estimated and QA/QC criteria were not met

4.5 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample

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concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.6 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples contain a known amount of a target compound and provide information about the effect of each samples' matrix on the sample preparation procedures and analytical results. Matrix spikes are typically analyzed at the following frequencies:

- 1 (MS/MSD pair) in every 20 samples
- 1 per preparation batch per matrix
- 1 per SDG

However, the frequency may be project specific and the documents outlining the needs of the project (SAP, QAPP, etc.) should be reviewed. In some cases, MS/MSD analysis is not required.

The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If a matrix spike recovery does not meet acceptance criteria and is not associated with a project sample, no further action is required unless other systematic evidence warrants qualification.

If the native concentration of a spiked sample is significantly greater than the spike added (>4x), spike recovery cannot be accurately evaluated, therefore the criteria do not apply. Professional judgment should be used for percent recoveries nominally outside laboratory acceptance criteria prior to qualifying data.

If criteria are not available, use guidance found in the NFG. Percent recoveries of matrix spike (and matrix spike duplicate) samples should be calculated using the equation provided under accuracy in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

Solid samples may have highly variable concentrations of target analytes and percent recoveries (%R) may be influenced by the sampling precision and inherent sample homogeneity. Professional judgment should be used for difficult matrices and the acceptance criteria adjusted accordingly.

Table 5 – Guidelines for Matrix Spikes

Criteria	Recommended Action for Associated Data	
	Detect	Non-Detect
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

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While matrix spike duplicates are not required by all methods, if results for MSD analyses are reported, evaluate the RPD for MS and MSD pairs using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

4.7 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency. *Title 40 of the Code of Federal Regulations, Part 136.3.*

Environmental Protection Agency, *National Functional Guidelines for Inorganic Superfund Data Review.*

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
5.0	06/17/13	Cover page	Added Calgary office
		Applicability	Added US to EPA reference
		I	Added waste rock and drill cores to examples of product sample
		III	Added LCSD information
		III, IV, V, VI	Added 'project specific requirements' as possible criteria source
		V	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		V	Clarified field duplicate criteria as < one value and not a range
		VIII	Added statement regarding multiple qualifiers
6.0	01/07/16	Document Wide	SOP restructuring, new format
7	04/24/18	1.0	Added laboratories may not provide all the review elements in this SOP, review only those that are provided.
		4.2, third bullet	Clarified that data reviewers would have to obtain raw data since not provided with Level II report.

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Standard Operating Procedure

Routine Level Dioxin/Furan (CDD/CDF) Data Evaluation

Revision 6

March 16, 2018

Approved By:

Marta Nelson		03/16/18
Print Technical Reviewer	Signature	Date

Terri Olson		03/16/18
Print QA Manager	Signature	Date

Review of the SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____

Initials: _____ Date: _____

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Initials: _____ Date: _____

Routine Level Routine Level Dioxin/Furan (CDD/CDF) Data Evaluation

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1.0 Scope and Applicability

This SOP is intended as a guidance SOP for the routine level evaluation of poly-chlorinated dibenzo-p-dioxin (CDDs or dioxins) and chlorinated dibenzofurans (CDFs or furans) data provided by laboratories to be used in Barr Engineering Company (Barr) projects.

This SOP is based on quality assurance elements, not the specific criteria, of *USEPA Contract Laboratory Program National Functional Guidelines (NFG) for* Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review and applies to routine CDD and CDF data evaluation for analyses by the following technologies:

- HRGC/HRMS
    - Method examples: EPA 1613, EPA 8290

The letter indicator for the various EPA method revisions have been intentional omitted. Multiple versions of the approved methods would be applicable for review under this SOP. In the case of specific technologies and/or methods not listed above, the guidelines within this document will provide the basis upon which to make adequate professional judgment in the evaluation of data submitted for review. Laboratories may not provide all the review elements in this SOP, review only those that are provided.

The recommended procedures in this SOP should be followed unless conditions make it impractical or inappropriate to do so. Modifications should be noted in the applicable documentation and communicated to appropriate personnel. Significant changes may result in a revision or newly created SOP.

2.0 Limitations

- Level IV data evaluation is not covered in this SOP and should be performed in accordance with NFG or project specific requirements and by a reviewer with CDD/CDF experience.
- Due to the complex nature of the analysis, Level IV data is most commonly reported; however, in some cases of well characterized sites, routine data are received. In these cases, this SOP applies.

3.0 Responsibilities

The laboratory is responsible for generating data from the samples submitted for analysis. In instances where QC criteria are not met for the analysis of samples, the laboratory is responsible for reanalysis of the samples, provided reanalysis is possible (considering matrix interference, holding times and sample volume, etc.), or documenting the impact to the data.

The Data Quality Specialist is responsible for evaluating the data in accordance with this document, in addition to using professional judgment where necessary or appropriate. Project specific requirements, such as those specified in a Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP), may differ from these recommendations and professional judgment should be applied before qualifying any data.

4.0 Procedure

The Quality Assurance/Quality Control (QA/QC) data detailed below are the most typical found in a routine level laboratory report. Other QA/QC data may be provided by the laboratory within the laboratory report case narrative, data qualifiers, or cover sheet and should be evaluated using professional judgment (e.g., initial calibration, calibration verification, internal standards).

Definitions to common QA/QC terms and terms used within this SOP along with a list of Barr 'Data Qualifiers/Footnotes' that may be applied during review can be found in Barr's "Compendium of Data Quality Assessment Documentation".

4.1 Holding Time and Preservation

The purpose of holding time and preservation evaluation is to ascertain the validity of the analytical results based on the sample condition, preservation, and time elapsed between the date of sample collection and date of analysis.

Individual methods and NFG are used as guidance for the recommended holding time and preservation acceptance criteria listed in *Table 1*.

Table 1 – Recommended Holding Times and Preservation^

Matrix	Temp.	Maximum Hold Time
Aqueous	≤6° C Protected from light sources	30 days extraction/ addl. 45 days extraction to analysis
Soil	≤6° C Protected from light sources	30 days extraction/ addl. 45 days extraction to analysis
Fish/Adipose Tissue	≤-10° C Protected from light sources	30 days extraction/ addl. 45 days extraction to analysis

^ *Preservation Notes*

- All samples should be stored in the dark. Amber sampling containers are recommended where applicable.
- If samples are suspected of containing residual chlorine (from potable supplies), those samples should be further preserved with sodium thiosulfate ($Na_2S_2O_3$) in a concentration of 80 mg/L. The pH must be adjusted afterward to a pH between 7 and 9.
- Due to the stability of CDD/CDF compounds in a variety of matrices, holding times may be up to one year if stored properly as noted above. Use the project-specific requirements when they deviate from the above guidelines.
- Per NFG, aqueous and soil samples must be extracted and analyzed within 35 days of the last sample receipt date in the SDG per contract requirements. However, technical holding time requirements allow that samples may be stored for up to one year under the conditions above. Sample extracts may be stored at < -10 °C in the dark for up to one year also.
- Fish and tissue samples must be extracted within 24 hours of thawing. Thawing time is considered cumulative after initial freezing period (upon receipt at the laboratory). For example, if the fish is

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thawed for two hours to be sub-sampled, refrozen and then thawed for two additional hours in subsequent sub-samplings, the total thaw time is four hours. Use professional judgment for exceedances beyond 24 hours of thaw time, if indicated in the report.

If samples do not meet holding time, preservation and analysis recommendations in *Table 1*, consider qualification with an "**h**". Other matrices, such as product samples (e.g. oil, waste rock, drill cores) may not be subjected to the same holding time recommendations.

If the sample was stored on ice upon collection and delivered to the laboratory the same day, the sample may exceed recommended temperature at the time of laboratory receipt. Professional judgment should be applied (considering temperature, matrix, magnitude of the exceedance, etc.) when evaluating the application of qualifiers when criteria are not met.

4.2 Blank Samples

Blank sample evaluation is conducted to determine the existence and magnitude of target analyte contamination as a result of activities in the field during collection and transport or from inter-laboratory sources.

- For each matrix, at least one method blank should be prepared and analyzed with each sample delivery group (SDG). Evaluation pertains to the batch of samples analyzed with the method blank.
- Field or equipment blank collection and analysis frequency is project specific. Evaluation pertains to the field samples associated with the field or equipment blank.
- Blank analyses may not have involved the same weights, volumes, or dilution factors as the associated samples. Data reviewers may have to obtain raw data and/or convert the data to the same units for comparison purposes.

Table 2 – Guidelines for Blank Contamination

Method Blank Result	Sample Result	Recommended Action for Associated Data
Less than (<) the estimated detection limit (EDL) or reporting limit (RL)	Non-detect	No qualification
Greater than (>) the EDL or RL	≤ 5x blank concentration (≤ 10x OCDD/OCDF)	Consider qualifying with 'b' or '<' (project specific)
	> 5x blank concentration (> 10x OCDD/OCDF)	Use professional judgment

b = Reported value may be a potential false positive based on blank data evaluation procedures

Note: Other multipliers of the blank contamination may be used based on professional judgment (reporting to the MDL, common lab contaminant, etc.)

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Professional judgment regarding the usability of the data should be used in cases where gross detections (equal to or greater than the RLs) of target analytes are found in the blank sample. A number of factors may be considered including historical data, prior knowledge of the site conditions, target analytes involved, type of blank sample, etc. In such cases, it may be appropriate to qualify the affected data with '*****' (estimated value, QA/QC criteria not met) or '******' (unusable value, QA/QC criteria not met).

4.3 Relative Retention Times (RRT)

Relative retention times (RRT) must be calculated by the laboratory for all identified 2,3,7,8-substituted isomers. The RRT is a comparison of the identified 2,3,7,8-substituted isomers to the isotopically-labeled counterpart or internal standard. These should fall within the defined ranges as published in the methods. The RRT values may or may not be provided in the non-CLP reports.

4.4 Ion Abundance Ratios (IAR)

Calculated ion abundance ratios are calculated for all positively identified native and isotopically-labeled compounds detected in each analysis. The ion abundance ratios for native concentrations may be reported on Form I and for the labeled-compound concentrations on Form II. Calculated ion abundance ratios should be within the guidelines in *Table 3* below (±15% of the theoretical ion abundance ratio) or within 10% of the third calibration standard. Samples meeting the RRT and signal to noise criteria but not meeting these IAR criteria would be qualified with the notation "EMPC" (Estimated Maximum Potential Concentration) depending on the method (8290 requires EMPCs; 1613 does not). Following method protocols, laboratory required calculations of Toxic Equivalents (TEQs) would exclude EMPC; however, recalculation of TEQs occur in most projects and scaling non-detect (ND) results or EMPC to 0, ½, or 1 needs to be decided by the project team.

Table 3 – Recommended QC limits for Ion Abundance Ratios (IAR)

# of Chlorine Atoms	Compounds in Group		QC Limits[1]	
	Dioxins	Furans	Lower	Upper
4[2]	2,3,7,8-TCDD $^{13}C_{12}$-2,3,7,8-TCDD	2,3,7,8-TCDF $^{13}C_{12}$-2,3,7,8-TCDF	0.65	0.89
5	1,2,3,7,8-PeCDD $^{13}C_{12}$-1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF $^{13}C_{12}$-1,2,3,7,8-PeCDF	1.32	1.78
6	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD $^{13}C_{12}$-1,2,3,6,7,8-HxCDD	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 2,3,4,6,7,8-HxCDF	1.05	1.43
6[3]	–	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	0.43	0.59
7	1,2,3,4,6,7,8-HpCDD $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	0.88	1.20
7[4]	–	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	0.37	0.51
8	OCDD $^{13}C_{12}$-OCDD	OCDF	0.76	1.02

[1] QC Limits ±15% windows around the theoretical IAR.

[2] Does not apply to ^{37}Cl -2,3,7,8-TCDD (cleanup standard).

[3] Use for $^{13}C_{12}$-HxCDF only.

[4] Use for $^{13}C_{12}$-HpCDF only.

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4.5 Labeled Compounds

Isotopically-labeled compounds, not expected to be present in the sample, are added to quantify sample concentrations and measure the analytical and extraction efficiency for each sample. The labeled compounds should be added to all QC samples as well as the study samples. Labeled compounds should be associated with their non-labeled counterparts when evaluating their effect on the reported results, per the number of chlorine atoms in the congener. In other words, $^{13}C_{12}$-2,3,7,8-**T**CDD would be applied to 2,3,7,8-**T**CDD, $^{13}C_{12}$-1,2,3,6,7,8-**Hx**CDD would apply to 1,2,3,4,7,8-**Hx**CDD, 1,2,3,6,7,8-**Hx**CDD and 1,2,3,7,8,9-**Hx**CDD.

If criteria are not reported, use guidance found in the NFG appendix table 'Labeled compound Recovery in Samples When All CDDs/CDFs are Tested'.

Table 4 – Labeled Compound Recovery Guidance

Criteria	Recommended Action for Associated Data	
	Detect	**Non-Detect**
%R > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
Note: If labeled compound recoveries are outside acceptance criteria, but the signal-to-noise ratio is greater than 10:1, the data is deemed acceptable and no qualification is required.		

'*' = reported value is estimated and QA/QA criteria were not met

'**' = reported value is unusable and QA/QC criteria were not met

4.6 Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicate Samples (LCSD)

The laboratory control sample is used to monitor the overall performance of each step during analysis, including sample preparation. The LCS should be analyzed:

- Once every preparation batch (20 or less samples of the same matrix)
- Once for each matrix.

Laboratory control samples may contain all target compounds or a subset depending on the analytical method. The percent recoveries are evaluated based on the criteria within the laboratory report or project specific requirements. If criteria are not available, use guidance found in the NFG. Percent recoveries are calculated for accuracy and the relative percent difference (RPD) is calculated for precision (when an LCSD was analyzed). Accuracy and precision equations can be found in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation".

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Table 5 – Guidelines for Laboratory Control Samples		
Criteria	**Recommended Action for Associated Data**	
	Detect	**Non-Detect**
%R and RPD > Upper Limit	Qualify with '*'	No qualification
%R < Lower Limit	Qualify with '*' or '**', use professional judgment	
%R and RPD within Limits	No qualification	

* = Reported value is estimated and QA/QC criteria were not met
** = Reported value is unusable and QA/QC criteria were not met

4.7 Field Duplicate Samples

Field duplicate samples (also known as "masked" or "blind" duplicate samples) are used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. Frequency of collection is project specific. The RPDs are calculated using the equation as provided under precision in 'Definitions' from Barr's "Compendium of Data Quality Assessment Documentation" and are not calculated where data is already qualified with b, U, <, or **. RPD results are dependent on the homogeneity of the samples.

Acceptance criteria for field duplicate samples are subject to the professional judgment of the Data Quality Specialist but typically RPDs ≤ 30% for aqueous samples and ≤ 40% for soil and sediment samples are considered acceptable unless other project specific requirements are defined.

Higher RPDs are expected when results are at or near the reporting limits and are not always indicative of poor precision. RPDs are typically only evaluated for samples where both the native and duplicate sample concentrations are greater than five times (>5x) the RL. In cases where either of the samples (native or field duplicate) is non-detect for a parameter and the other corresponding sample has detectable concentrations much greater than five times (>5x) the RL, professional judgment should be used to determine if qualification is appropriate.

4.8 Matrix Spikes (MS) and Matrix Spike Duplicate (MSD) Samples

Matrix spike samples are not required for this analysis due to isotope dilution quantitation method resulting in every sample effectively being an MS/MSD.

4.9 Overall Assessment

The chain-of-custody should be reviewed to determine if the laboratory report matches the requested analyses and that project specific parameters were analyzed as requested. The narrative and other supporting documentation should be evaluated to ensure that sample condition was appropriately documented by the laboratory upon receipt. If available, historical data should be used to assist with data evaluation. Any additional anomalies should be documented and evaluated, if necessary.

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5.0 Quality Control and Quality Assurance (QA/QC)

Depending on the project objectives, the data review may include the completion of a Routine Level Quality Control Report (see Barr's "Compendium of Data Quality Assessment Documentation") as part of the evaluation process. Within each QC data section, the reviewer should include references to whether the QC data met or exceeded the acceptance criteria. The qualifiers, added, removed, or retained, should be documented also. Where multiple qualifiers may be applicable to a sample/analyte result, professional judgment should be used to determine if all qualifiers are necessary or if one qualifier would be sufficient to represent the deviations. A statement as to whether the data are acceptable as reported or acceptable with qualification(s) should also be included. If revised reports are required and the revision affects the sample results, notification should be given to the appropriate data management personnel and/or project team members.

The Data Quality Specialist will verify that the qualifiers associated with data tables match the Routine Level Quality Control Report.

6.0 Records

The Routine Level Quality Control Report should be saved to the appropriate internal Barr file and the link uploaded to the tracking system. Periodically, Data Quality staff should check for missing Routine Level Quality Control Reports in the tracking system to help maintain the most current information.

Documentation specific to this SOP are listed below and are available in Barr's "Compendium of Data Quality Assessment Documentation".

- Definitions
- Barr Qualifiers/Footnotes
- Routine Level Quality Control Report

Additional records information can be found in Barr's "Records Management System Manual".

7.0 References

Environmental Protection Agency, *National Functional Guidelines for Superfund Organic Methods Data Review*.

Analytical methods listed under the 'Scope and Applicability' section of this SOP.

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Attachment 1

Revision History

Revision Number	Date of Revision	Section	Revision Made
2.0	06/2009	Document Wide	Complete revision to reflect current practices
3.0	04/2011	Document Wide	Corrected holding time and sample preservation requirements. Updated attachments.
4.0	04/06/12	Document Wide	Major revision
5.0	06/17/13	Cover page	Added Calgary office
		V, VII	Added 'project specific requirements' as possible criteria source
		VII	Added 'field and laboratory procedures' to clarify that it's not only a laboratory item
		VII	Clarified field duplicate criteria as < one value and not a range
		X	Added statement regarding multiple qualifiers
6	03/16/18	Document Wide	SOP restructuring, new format

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Compendium

Of

Data Quality Assessment Documentation

Minneapolis, MN ● Hibbing, MN ● Duluth, MN ● Ann Arbor, MI ● Jefferson City, MO ● Bismarck, ND ● Calgary, AB, Canada ● Grand Rapids, MI ● Salt Lake City, UT

Barr DQ Assessment Definitions

Accuracy: Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy measures the bias in a measurement system. Accuracy of laboratory results may be assessed using the analytical results of method blanks, field blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples and laboratory control samples. The percent recovery for (%R) matrix spikes and laboratory control samples will be calculated using the following equation:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where: %R = % recovery
SSR = spiked sample result
SR = sample result
SA = spike added to native sample

NOTE: In the case of LCS and other laboratory-prepared samples, SR is zero.

Batch: Group of samples of the same matrix prepared for single or multiple analyses that will be analyzed during one operation at a given specific time frame. Typical size is 1-20 samples.

Blank: A sample designed to assess specific sources of contamination.

Calibration: Calibration is the process of checking, adjusting or determining by comparison under specified conditions an instrument's response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data.

Contamination: A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Data Quality Specialist: An individual that is part of the Data Quality group at Barr Engineering and may be referred to as a Quality Assurance Manager, Quality Assurance Officer, or Quality Manager within Quality Assurance Project Plans or other project documentation.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Equipment (Rinsate) Blank: A sample of analyte-free water collected when rinsing sampling equipment. It measures the potential for sample cross contamination due to insufficient decontamination of sampling equipment.

Field Blank: A sample of analyte-free water exposed to environmental conditions at the sampling site by transferring from one sample container to another or by removing the lid and exposing a container filled with analyte-free water to the atmosphere for the time equivalent necessary to fill a container. It measures the potential for sample cross contamination due to site conditions.

Field Duplicate: A duplicate sample generated in the field that is used to demonstrate acceptable precision and reproducibility of the field and laboratory procedures. The sample identification is typically kept blind (masked) from the laboratory.

Holding Time: The maximum recommended amount of time samples may be held before they are processed**.**

Instrument Blank**:** A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover. It measures laboratory sources of contamination.

Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD): A sample of analyte-free media spiked with known concentrations of target analytes that is carried through the same sample preparation and analytical procedures. LCS recoveries are used to estimate overall analytical method accuracy independent of sample matrix effects. The RPD between the LCS and LCSD is used to assess the overall analytical method precision. Also referred to as a Laboratory Fortified Blank.

Matrix: The predominant material of which the sample to be analyzed is composed (e.g. water, soil, sediment, etc.).

Matrix Effect**:** In general, the effect of a particular matrix on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike **(MS)** ***and Matrix Spike Duplicate (MSD):*** A sample spiked with known concentrations of target analytes that is carried through the sample preparation and analysis procedures in order to assess the accuracy of a method in a given sample matrix. The RPD between the MS and MSD is used to assess the precision of a method in a given sample matrix. Also referred to as a Laboratory Fortified Matrix.

Method Detection Limit (MDL): The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. EPA procedures for determining the MDL are given at 40 CFR 136, Appendix B.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. It measures laboratory sources of contamination.

Narrative: The portion of the data package which includes laboratory, contact, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Precision. Precision measures the reproducibility of measurements under a given set of conditions. Precision of analytical laboratory data may be assessed by comparing the analytical results between matrix spike/matrix spike duplicates (MS/MSD), laboratory duplicates, or masked field samples (field duplicates). Field duplicate samples, when collected, processed, and analyzed by the same organization, provide intralaboratory precision information for the entire measurement system, including: sample acquisition, sample constituent heterogeneity, handling, shipping, storage, preparation, and analysis. Field duplicate samples are submitted to the laboratory as blind or mask samples. The relative percent difference (%RPD) will be calculated using the equation below for each pair of duplicate analysis.

$$RPD = \frac{|S - D|}{(S + D)/2} x\,100$$

Where: RPD = relative percent difference
S = original sample result
D = duplicate sample result

Quality Assurance Project Plan (QAPP): A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

Reporting Limit (RL): The RL is the lowest reported concentration, provided on the sample-analysis data report, after corrections have been made for sample dilution, sample weight, and (for soils and sediments) amount of moisture in the sample.

Sample Delivery Group (SDG): Identifies a group of samples for delivery, A sample delivery group is defined by the following, whichever is most frequent:

- Each set of field samples received; or
- Each 20 field samples within a sampling event; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples are received.

Synthetic Precipitation Leaching Procedure (SPLP): A test designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes. It can be used to assess the risk of groundwater contamination posed by the land application of granular solid wastes.

Toxicity Characteristic Leaching Procedure (TCLP): A test designed to determine whether a waste is hazardous or requires treatment to become less hazardous; also can be used to monitor treatment techniques for effectiveness.

Barr Qualifiers/Footnotes

Qualifier	Definition
a	Estimated value, calculated using some or all values that are estimates.
b	Potential false positive value based on blank data validation procedures.
c	Coeluting compound.
e	Estimated value, exceeded the instrument calibration range.
f	Sample was collected at a flowrate exceeding the recommended rate of 200 mL/minute.
h	EPA recommended sample preservation, extraction or analysis holding time was exceeded.
i	Indeterminate value based on failure of blind duplicate data to meet quality assurance criteria.
j	Estimated detected value. The reported value is less than the stated laboratory quantitation limit but greater than the laboratory method detection limit.
p	Relative percent difference is >40% (25% CLP pesticides) between primary and confirmation GC columns.
pp	Small peak in chromatogram below method detection limit.
r	The presence of the compound is suspect based on the ID criteria of the retention time and relative retention time obtained from the examination of the chromatograms.
t	Sample positive for total coliforms but negative for *E. coli*.
v	Sample was collected under a vacuum of greater than XX inches of mercury.
*	Estimated value, QA/QC criteria not met.
**	Unusable value, QA/QC criteria not met.
AT	Sample chromatogram is noted to be atypical of a petroleum product.
EMPC	Estimated maximum possible concentration.

Barr Engineering Company
Routine Level Quality Control Report

Project #		**Project Name**	
Laboratory		**COC(s)/Event**	
Lab Report #		**Matrix**	
Report Date		**Review Date**	
Holding Times Met	Yes No	**Reviewed By**	
If no, comments		**Posted to QC Track?**	☐
		Revised Report?	☐ **Rev #**
Temps on Receipt (°C)		**Data Report Request #**	

Method Blanks	**LCS/LCSD**
Field Blanks	**MS/MSD**
Trip Blanks (VOCs Only)	
Field Duplicates (if applicable)	**Surrogates (if applicable)**
	Lab Duplicates (if applicable)

Master Worksheet located at:
W:\Business Units\AR\Subunit Admin\Practices-Processes\QCTRACK\MASTER SOP File\Data Val Wrksht Rev 2011 locked.xlsx

Additional Notes (include historical comparison, if appropriate)

Qualifier Summary		Qualifier Changes		
Sample Name	Parameter	Added	Removed	Retained

Additional Notes to DM

For DM Use Only	
Equis Y / N	Data Tech Init:
Facility ID:	Date entered into Equis:

Appendix E

Pace Field Division Standard Operating Procedures and Quality Assurance Manual

Soil Vapor Collection

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I. Soil Vapor General Procedures

The proper collection of soil vapor samples is a critical step in producing reliable concentration data. A number of factors are important in ensuring the reliability of the data; each is discussed below. Note that some regulatory agencies have specific guidelines for soil vapor collection. These guidelines should be followed in the design of a soil vapor collection plan.

Prior to beginning a soil vapor sampling program, it is important to obtain the correct sampling equipment and to write a site-specific sampling plan. Written documentation of the equipment used and the sampling processes employed is critical. Consistency in equipment and sampling processes between probe locations and between multiple sampling events is important in order to minimize potential discrepancies in soil vapor concentration data. Make sure all equipment has been decontaminated before beginning sampling activities.

II. Sampling equipment

Numerous types and combinations of tubing, connectors, valves, and pumps have been used for soil vapor sampling. The tubing, gauges, and pump (if any) should be connected by tubing that is flexible, air-tight, and has a low capacity for adsorption of VOC's. Teflon® or Nylon tubing (marketed under the NylaFlow® name) with ⅛ or ¼-inch OD recommended. Tygon®, rubber, and polyethylene tubing should be avoided. Swagelok® type connectors should be used for all connections between tubing and other sampling components. These connectors are air-tight and reliable. The lack of an air-tight seal can allow oxygen to enter the sample, thus diluting the vapor concentrations and compromising the integrity of the sample. Leak testing is used to ensure the integrity of soil vapor samples.

A vacuum must be created in order to draw the soil vapor to the land surface. The vacuum can be created by a battery powered pump, a syringe, or a sampling container that is under a vacuum (such as a Summa® canister, discussed below). If a pump is used, it is important to ensure that the sample collection point is on the intake side of the pump. This will prevent any contaminants present in the pump from being drawn into the vapor sample. A three-way valve can be used to isolate the pump from a separate tube that is connected to a vapor sample container (see Figure 11).

Summa® canisters are strongly recommended for soil vapor samples. Tedlar® bags are not recommended. Soil vapor samples are collected in syringes in on-site soil vapor analyses *only* where the sample is immediately injected into a gas chromatograph (GC). Containers range in size from < 1 L to 15 L and are provided by the analytical laboratory. The canister cleaning process utilizes dilution, heat, and high vacuum. The canister will hold a vacuum of < -25 in Hg for more than 30 days. Check the locally applicable regulations to determine the maximum holding time for the site in question. The soil vapor sample flows into the canister due to the pressure gradient between the vadose zone and the canister. A flow controller/particulate filter controls the vapor flow rate into the canister.

III. Sample Collection

Make certain all connections between the Summa® canister, flow controller, and all other portions of the sampling equipment are tight. Leak testing should be performed concurrently with sampling. To begin

sampling, open the valve on the Summa® canister. As the canister fills, observe the pressure gauge on the flow controller to ensure that the vacuum in the canister is decreasing over time. If the flow controller is working correctly, the planned sampling completion time will be reached when the pressure has decreased to 5 in Hg. Note that low permeability soils characterized by low soil vapor flow rates may require sampling to cease before the canister pressure has decreased to 5 in Hg.

Quality control (QC) of soil vapor samples must be addressed through the collection of field blanks and field duplicates, and the transport trip blanks. A field blank should be collected at the site during sampling activities from a certified air source. At least one trip blank should be obtained from the analytical laboratory for each sampling day (event). The trip blank contains laboratory grade ultra-pure air and is intended to provide evidence of contaminants entering the sample containers during handling and shipping.

At least one duplicate sample should be obtained each day of sampling. A duplicate sample should be collected by using a splitter located upgradient of the flow controller, with separate sampling tubes connecting the splitter to two Summa® canisters.

After collection, canisters must not be chilled since contaminants may condense in the canister at low temperatures. Make certain that all samples are correctly and clearly labeled. Follow standard chain-of-custody procedures. Document all procedures, sampling times, conditions, problems, etc.

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Soil Sampling

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I. General Procedures

A. The locations of surface soil samples, soil borings, and excavation areas should be identified and documented on a site map. If applicable, obtain soil boring logs prepared by qualified personnel (e.g., geologist, engineer, etc.) or Site Investigation Report. The appropriate method for obtaining a soil sample is determined by site conditions. Soil samples can be obtained using the following methods:

1. Hand auger, trowel or spatula for collecting surface samples and composite samples or stockpiled soils;
2. Slit spoon sampler when drilling well boreholes and constructing depth profiles; and
3. Backhoe for collecting samples from an excavation area.

II. Surface Sampling

A. All soil sampling equipment must be decontaminated prior to each use. A shovel, trowel or scoop can be used for sample collection of surface soils. Soil samples taken at depths greater than three inches should be collected with a hand auger or a tube sampler. Stainless steel sampling equipment should be used whenever possible.

III. Subsurface Sampling

A. Borings for subsurface sampling should be advanced with a hollow-stem, continuous flight auger. Other drilling methods may be used as dictated by site specific conditions and approved in advance by appropriate personnel.

B. Soil samples should be obtained using a split spoon sampler. The sampling method as prescribed by ASTM:D 1586-84 may be used. Samples should, at a minimum, be taken every five feet or as often as necessary to accurately describe the stratigraphy and any zones of contamination. The sampling device should be decontaminated between each sampling event.

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PROCEDURE MANUAL

WATER MONITORING

WASTEWATER, SURFACE WATER, DRINKING WATER, AND GROUNDWATER

Document Number:	PM-FSD-004-rev.06
Effective Date:	Date of Final Signature
Supersedes:	PM-FSD-004-rev.05

APPROVALS

Donald B. Stock, QEP, General Manager — Date: 6/15/17

Richard A. Smith, Quality Assurance Officer — Date: 6/15/17

DOCUMENTATION OF PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1.0 INTRODUCTION

Pace Analytical Services, LLC – Field Services Division (Pace FSD) employs a rigorous quality program to ensure its clients receive the highest quality data. Proper collection and analysis of field samples are essential to producing quality field and laboratory results. All field personnel performing testing projects are trained in the proper use of sampling equipment, sampling procedures, and sample analysis.

Field technicians performing sampling and field testing procedures have primary responsibility for the quality of sample analyzed. Selection of sample points, collection of representative samples, sample preservation, transportation and storage, along with equipment and methods selected to collect and analyze samples are made based on many factors. These factors include regulatory requirements, reference methods, training and experience, documented procedures, process information, data quality objectives, and site conditions. All water surveys are performed by trained field personnel under the supervision of a project administrator, knowledgeable in the applicable regulatory programs, EPA Safe Drinking Water Standards, and National Pollutant Discharge Elimination System (NPDES). Documentation on field data sheets is essential to demonstrate appropriate sampling procedures and record ancillary information. Documentation shall be sufficient to enable a second party to reconstruct the events of a project.

Pace FSD ensures, to the extent practical, incompatible activities or environmental conditions do not invalidate results or adversely affect the quality of the work performed. Sample preparation, collection, recovery, and analysis are shielded from environmental conditions, including photo reactivity, which may bias sample results. Pace FSD employs good housekeeping practices in locations where testing or analytical activities are conducted and will institute special procedures as necessary to ensure valid sample collection, preservation, and analysis.

1.1 Procedure Manual

The purpose of this procedure manual is to provide consistency throughout the sample and field data collection process. It contains information on equipment, field testing procedures, flow monitoring, and sample collection. Examples of forms used for collecting survey data are also included.

It is Pace FSD policy to make safety and health an integral part of its daily operations. All employees receive training in OSHA safety requirements for performing applicable job duties. Pace FSD provides each employee with the means to create a safe working environment, including all necessary personal protective equipment and training. Each employee is responsible for his or her own safety by complying with established safety programs and any site-specific safety requirements. Applicable safety procedures are to be followed during all sampling preparation and sampling events discussed in this manual.

1.2 Wastewater Project Task Chronology

Each wastewater project is accomplished by performing a series of tasks. Field technicians follow the chronology of events listed in Section 1.2, when applicable, to accomplish project objectives in the most efficient and accurate manner possible. Project administration tasks are performed by a project administrator or designee. A Project Completion Record (Attachment 1) is initiated for each project and is stapled to the front of a project folder. A Project Completion Record provides a checklist of frequently performed project tasks.

1.2.1 Project Initiation

The project administrator contacts clients and schedules projects. The project administrator determines pertinent regulatory and analytical laboratory requirements to meet client needs. Projects are assigned to a specific technician and posted on the scheduling board in a timely manner. Multiple projects for a given technician are scheduled within the same geographical

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region when possible.
For most projects, the project administrator provides the following in a project folder to the designated technician:

- FSD project number.
- Project Completion Record stapled to project folder (Attachment 1)
- Client Data Sheet (Attachment 2) and/or Project Specific Site Information Form (Attachment 3)
- Chain-of-Custody (Attachment 4)
- Sample Labels (Attachment 5)
- Project Expense Form (Attachment 6)

1.2.2 Project Preparation

The lead technician reviews the project folder and is responsible for overseeing required activities. As needed, the lead technician meets with the project administrator to discuss and clarify aspects of the project. Applicable data sheets dictated by the project scope are obtained by the lead technician. The technician(s) prepares all sampling equipment necessary for the project, including icing the sampler base prior to deployment, when needed. Appropriate vehicles for project needs are signed out and loaded with project equipment.

1.2.3 Set-up Procedure

1. Contact client with estimated time of arrival when required by project.
2. When onsite, perform project specific requirements.
   a. obtain all necessary information from client
      i. location of sampling point(s)
      ii. water meter(s), testing parameters
      iii. hours of operation
      iv. employee count
   b. If required, set-up automatic/programmable sampler. Refer to Sections 3.1.1, 3.2, and 3.5 for equipment use and sampling procedures.
   c. If applicable, install continuous pH meter. Refer to Sections 6.2.2 or 6.2.3.
   d. If applicable, collect grabs. Refer to Section 3.6 and 5.0.
   e. If applicable, initiate flow monitoring.
      i. **For projects requiring hourly facility water meter readings**: Take first reading on Hourly Water Meter Reading Record Sheet, when possible, and inform client to continue on an hourly basis. Record any additional facility water meters specified by project and confirm meter units. Refer to Section 7.6.
      ii. **For Projects requiring flow-monitoring equipment**: Only a 24-hour reading (one at the beginning and ending of the sampling time frame) is necessary, unless otherwise specified by project. Record beginning reading and confirm meter units. Refer to Section 7.6.
3. Upon completion of setup, fill out required data sheets and initiate chain-of-custody if samples are obtained.
4. Review client data sheet prior to leaving the site to verify all project tasks have been completed.
5. Review with project administrator any deviations encountered during setup.

1.2.4 Take-Down Procedure

1. When onsite, perform project specific requirements.
   a. obtain all necessary information from client not obtained during set-up.

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i. location of sampling point(s)
ii. water meter(s), testing parameters
iii. hours of operation
iv. employee count

b. If required, take-down automatic/programmable sampler. Refer to Section 3.2.4 for general sampler take-down procedure.
   i. **If one 24-hour sample period required:** Verify representative sample and flow data were obtained before removing equipment. Refer to Section 8.0 for criteria of representative sample.
   ii. **If multiple 24-hour sample periods required:** Verify representative sample and flow data were obtained before removing sampler base. Change over sampler following setup procedures. Refer to Section 8.0 for criteria of representative sample.

2. If applicable, complete flow monitoring.
   a. **Projects requiring hourly facility water meter readings**: Record last reading on Hourly Water Meter Reading Record Sheet, when possible. Review sheet for completion and accuracy and address any anomalies with client. Record ending readings of any additional water meters specified by project.
   b. **Projects requiring flow-monitoring equipment**: Record ending water meter readings as close to 24-hours after set-up reading as possible.
3. If applicable, remove continuous pH meter. Refer to Sections 6.2.2 or 6.2.3.
4. If applicable, collect grabs. Refer to Section 3.6 and 5.0.
5. Upon completion of take-down, fill out required data sheets noting problems or abnormalities.
6. Review with project administrator any deviations encountered during take-down. Upon return to Pace, proceed to Section 1.2.5 and 1.2.6.

1.2.5 Demobilization

1. Place sampler control sections in area designated for cleaning.
2. Place entire sampler base and any other samples in walk-in cooler pending compositing or sample check-in as temperature is a preservation method for most analytes. Make every effort to keep samples chilled.
3. Clean equipment used in sampling, such as flow meters, when necessary and return to proper storage areas.
4. Charge depleted batteries for adequate recharge time.
5. Clean, restock, and reorganize vehicle when necessary.

1.2.6 Compositing and Sample Check-In

Refer to Sections 8.0 and 9.0.

1.3 Groundwater Monitoring Task Chronology

Each groundwater project is accomplished by performing a series of tasks. Technicians follow the chronology of events listed in Section 1.3, when applicable, to accomplish project objectives in the most efficient and accurate manner possible. Project administration tasks are performed by a project administrator or designee.

1.3.1 Project Initiation

The project administrator is responsible for meeting pertinent regulatory and analytical laboratory requirements for client needs. Projects are assigned to a specific technician and

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posted on the scheduling board in a timely manner. The lead technician obtains a client data sheet, client sampling plan, or Pace proposal from the project administrator regarding project details and objectives. The lead technician reviews a project's associated binder or folder containing client, site, and historical information when available. The lead technician is responsible for overseeing required activities and will meet with the project administrator to discuss and clarify aspects of the project as needed.

1.3.2 Project Preparation

1. The lead technician obtains applicable documents and data sheets as dictated by project scope including but not limited to:

    - Client data sheet, client sampling plan, or Pace proposal.
    - Other information necessary to properly identify scope of work. This may include maps, historical field and laboratory data, or other special instructions.
    - Field Data Sheets (Attachment 11).
    - Field Instrument Calibration and Performance Log (Attachment 14A-F).
    - Quick Reference Table (Attachment 12).
    - Sample labels (Attachment 5).
    - Chain-of-Custody Record (Attachment 4).

2. Technician(s) prepares all sampling equipment and performs decontamination and calibration requirements for project objectives. Refer to Section 2.0 and 6.0. Document calibration on Field Instrument Calibration and Performance Log (Attachment 14A-F).
3. QA/QC samples are prepared, if necessary. Refer to Section 1.4.
4. Technician obtains keys required for entry into well(s) or sampling point(s), if necessary.
5. Technician signs out vehicle appropriate for project tasks and loads equipment and supplies. Refer to Attachment 13 for general equipment list.

1.3.3 Field Activities

Begin activities at the monitoring well with the least expected contamination and proceed to increasingly contaminated wells. If contaminant distribution is unknown, begin with wells considered to be up gradient from likely sources of contamination and work down gradient. Complete the entire purging and sampling requirements for one well before moving to subsequent wells. Take appropriate measures to minimize contamination of sampling equipment.

The following is a basic chronology used to sample a well for a typical groundwater monitoring project. Refer to client data sheet, client sampling plan, or Pace proposal for specific project requirements.

1. Unlock well and record observations regarding its security such as cracks in the casing or seal. Record observations regarding well conditions including soil depressions, vegetation, and unusual debris or odors.
2. Measure and record the static water level and total well depth. Refer to Section 4.3.1.
3. Calculate the volume of water in the monitoring well (one casing volume) using the Well Volume Multiplication Factors Table (Attachment 12). Refer to Section 4.3.2.
4. Purge well and monitor stabilization criteria. Refer to Section 4.4 and 4.3.3.
5. Collect required samples. Refer to Section 4.5.
6. Proceed to demobilization and post sampling activities. Refer to Sections 1.3.4 and 1.3.5.

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1.3.4 Demobilization

1. Maintain cooling process for samples requiring thermal preservation pending sample check-in. Submit samples to analytical laboratory accompanied by a chain-of-custody. Refer to Section 9.0.
2. Clean and/or decontaminate equipment used in the field as necessary.
3. Return equipment to proper storage areas and recharge depleted batteries.
4. Clean, restock, and reorganize vehicle when necessary.

1.3.5 Post Sampling Activities

1. Review sampling event with project administrator and discuss any deviations encountered during sampling.
2. Enter field data into electronic format as required by client or project scope.
3. Once obtained from laboratory, review Sample Receipt Form to ensure complete and correct analysis of samples.
4. Original calibration and field data sheets are retained for the client report; copies of calibration and field data sheets are retained in a project folder at Pace FSD for future reference.

1.4 Quality Assurance/Quality Control Samples

Quality assurance/quality control samples are collected to facilitate accurate and consistent procedures and data. The type and number of QA/QC samples are dictated by the respective project scope. The project administrator will provide instructions as to whether the quality assurance/quality control samples will be analyzed for a specific project. If QA/QC samples are not to be analyzed, indicate "HOLD" on the chain-of-custody.

1.4.1 Travel/Trip Blanks

Travel/trip blanks are used to detect contamination associated with sample handling and transport. Travel blanks are prepared in the laboratory with laboratory grade, organic-free water, or equivalent, and preserved using the same sampling methods used to collect field samples. Travel blanks will be kept with associated sample bottles at all times.

1.4.2 Ambient Air Field Blanks

Ambient air field blanks are collected to detect background contamination. Field blanks are filled with appropriate grade water by the same personnel, in the same manner, and at the same location and approximate time as the samples are collected. Expose the blank water in each container to the air for an amount of time equivalent to the time required for filling and sealing sample containers.

1.4.3 Equipment Blanks

Equipment blanks are used to detect equipment or sampling method contamination. Equipment blank samples are collected in the field using the same procedures and equipment that will be used to obtain samples. All efforts will be made to have blank sample water contact all interfaces that sample water will contact during the collection process.

1.4.4 Field Duplicates

Field duplicates are collected and analyzed to verify the reproducibility of both the sample collection procedure and the analytical methodology. Field duplicates are collected using the

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same sampling equipment following the same procedures that will be used to collect primary samples but are collected during a separate sampling operation. When submitted to an analytical laboratory, the true identity of samples will be recorded on the field data sheet only; aliases will be assigned for chain-of-custody purposes.

1.4.5 Split Samples

Split samples are used to verify the accuracy of laboratory analysis. Split samples are collected by filling multiple sample containers from a single sample producing replicate samples for desired parameters. Sample splits may be sent to two separate analytical laboratories.

1.4.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Additional sample volumes are sometimes collected for MS/MSD samples. MS/MSD samples are collected to satisfy routine analytical quality control requirements of the analytical laboratory. MS/MSD samples verify the recovery rates of specific compounds in a variety of matrixes for a given analytical method.

2.0 DECONTAMINATION

Equipment used to collect samples must be decontaminated before use. These procedures are applicable to most sampling equipment components; refer to manufacturer specifications for compatibility concerns.

Pace FSD uses certified clean sample containers. Cleaning of sample containers is not necessary if sample is collected directly into new, certified sample container for desired analyte.

2.1 Wastewater, Surface Water, and Drinking Water Decontamination

2.1.1 Sampling for Inorganic Analytes

1. Remove any gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Final rinse with deionized (DI) water
5. Drain or allow components to air dry when possible.

2.1.2 Sampling for Semi-Volatile Organic Analytes

1. Remove any gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Triple rinse with DI water.
5. Rinse with acetone or methanol.
6. Optional final rinse with methylene chloride.
7. Wrap cleaned equipment in aluminum foil shiny side out.

2.1.3 Sampling for Volatile Organic Analytes

1. Remove any gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Triple rinse with DI water.

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5. Place components in oven for a minimum of 1 hour at 105°C or greater.
6. Wrap component in aluminum foil shiny side out.

2.2 Groundwater Decontamination

All pumps used to obtain groundwater samples are decontaminated before use in the field. Permanently installed sampling equipment is exempt from decontamination procedures. New or dedicated pump tubing is used for each sampling location. Dedicated tubing is stored between sampling events in a sealed, chemically inert plastic bag labeled with the site location. Pump bladders are dedicated in this same manner. Field cleaning procedures are performed on equipment used at multiple sampling locations to minimize cross-contamination. Technicians take appropriate measures to minimize potential contamination during transport and handling of sampling equipment. Avoid introducing surface or ambient air contamination into monitoring well.

2.2.1 Pump Decontamination Before Field Use

1. Scrub and flush pump exterior with laboratory grade soap and hot tap water.
2. Fill tank with hot tap water containing laboratory grade soap.
3. Place pump in tank and pump approximately 10 gallons through pump.
4. Fill tank with DI water.
5. Place pump in tank and pump approximately 10 gallons through pump.

2.2.2 Pump Cleaning Onsite

1. Scrub and flush pump exterior with laboratory grade soap and water.
2. Rinse pump exterior with DI water.
3. Place pump in DI tank and pump approximately 1 gallon of DI water through pump.

3.0 WASTEWATER, SURFACE WATER, DRINKING WATER MONITORING

3.1 Automatic/Programmable Monitoring Equipment

3.1.1 Isco Automatic Wastewater Samplers

Automatic/Programmable Isco model numbers 2700, 3700, and 6700 are portable devices designed to collect up to 24 separate sequential samples or a single composite sample from a liquid source. The samples may be collected at equal time intervals using the sampler's internal timing circuitry or at equal flow volume intervals using flow pulse inputs from an external flow meter.

In the time mode, the interval between samples can be set from 1 to 9999 minutes in 1-minute intervals. In the flow mode, the interval can be set from 1 to 9999 flow pulses in one-pulse intervals. A manual sample collection can also be initiated.

In the sequential mode, samples can be collected in either 350-mL glass or 1000-mL polypropylene sample bottles. In the composite mode, samples can be collected in a 9500 mL glass or polyethylene sample container. Sample volumes can be selected in 10-mL increments with a maximum volume of 990 mL.

Samplers are powered by using a 110-volt AC charger or an external 12-volt battery. Basic programming steps for conventional sampling routines are listed below for each model. For non-routine sampling programs, review the appropriate manufacturer instruction manual before deployment in the field.

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3.1.1.1 Isco Model 2700

To Program:

1. Press "ON" button.
2. Press "PROGRAM/STEP PROGRAM" button.
3. Using the numeric keypad, enter desired setting for program quantity indicated by light.
4. Press "ENTER VALUE" button.
5. Press "PROGRAM/STEP PROGRAM" button to advance to next feature.
6. Repeat steps 3, 4, and 5 until all program features are set.
7. Press "START PROGRAM/RESET DISTRIBUTOR" button to start sampling program.

3.1.1.2 Isco Model 3700

To Program:

1. Turn unit on. The "STANDBY" message will appear above the time and date. An option is selected when it is flashing.
2. To set sample tubing length:
    a. Press the "ENTER/PROGRAM" button.
    b. Use the arrow key to select "CONFIGURE" and press the "ENTER/PROGRAM" button.
    c. Use the arrow key to scroll through the list of options and select "SUCTION LINE" and press "ENTER/PROGRAM."
    d. Select appropriate suction line ID and press "ENTER/PROGRAM."
    e. Select "VINYL" or "TEFLON" depending on type of sample tubing.
    f. Enter suction line length in feet (3-99) and press "ENTER/PROGRAM." This is the estimated combined length of sample and pump tubing.
    g. Press the "EXIT PROGRAM" button to return to "STANDBY" window.
3. To set sample program, press the "ENTER/PROGRAM" key, select "PROGRAM," and press "ENTER/PROGRAM."
4. To set time or flow dependent sampling:
    a. For time dependent sampling, enter desired hour and minute interval (0 hr. and 15 min. for four samples per hour).
    b. For flow paced sampling, enter desired sample frequency in pulses (1-9999).
5. To set multiplex sample option, select [YES] if using multiple bottles and [NO] if using only one bottle.
6. To set sample volume, select [SAMPLES PER BOTTLE].
    a. Enter number of samples per bottle (1-50).
    b. Enter sample aliquot volume in milliliters (10-25).
    c. Enter suction head in feet (3-30).
7. Select start time [YES or NO]:
    a. If yes, select [YES] and enter time and date of first sample. Press the "START SAMPLING" button. Screen will display sample schedule, program start time, and the current time if the sampling program is initiated.
    b. If no, select [NO] and press the "START SAMPLING" button to start program immediately.
8. Press the "EXIT PROGRAM" button at any time to return to "STANDBY" display.

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3.1.1.3 Isco Model 6700

To program: 24-1000 mL bottles.

1. Turn unit on using the ON/OFF button. The main menu screen will appear with four options:
    a. Run
    b. Program
    c. View Report
    d. Other Functions
2. An option is selected when it is flashing. Select "Program" using the arrow keys. Press the "↵□" key in the lower right hand corner of the keypad to enter selection. Select program features as prompted.
3. Site Description Change: Select "NO" and "ENTER."
4. Select units for length: Select "ft" and "ENTER."
5. Number of bottles: Select "24" and "ENTER."
6. Bottle Volume (mL): Select "1000 mL."
7. Suction Line Length is (3-99) ft.: Select appropriate line length in ft.
8. Time or Flow Paced:
    a. Time Paced: enter desired hour and minute interval (0 hr. and 15 min. for four samples per hour).
    b. Flow Paced: enter desired pulse interval (1-9999).
9. Sequential or Bottles/Sample or Samples/Bottle: select sequential for flow paced and samples/bottle for time paced.
10. Select appropriate samples per bottle (1-50) for time paced.
11. Run Continuously?: select "YES" for flow paced and "NO" for time paced.
12. Sample Volume (10-250) mL: Select desired sample volume.
13. No Delay to Start or Delayed Start or Clock Time: Select appropriate starting time.
14. Programming Complete: Run this Program Now? YES or NO.
    a. If YES, distributor arm will move to first bottle and begin sampling.
    b. If NO, a list of options will appear and selecting "Resume Program" will begin sampling.
15. Stop program by pressing the red STOP button at any time (circle inside of a triangle located in the upper right side of the keypad) and "Resume Program" to begin sampling again.

3.1.1.4 Isco Model 2900 and 2910

Automatic/Programmable Isco model numbers 2900 and 2910 are small portable devices designed to collect up to 24 separate sequential samples or a single composite sample from a liquid source. The samples can be collected at equal time intervals using the sampler's internal timing circuitry or at equal flow volume intervals using flow pulse inputs from an external flow meter.

In the time mode, the interval between samples can be set from 1 to 9999 minutes in 1-minute intervals. In the flow mode, the interval can be set from 1 to 9999 flow pulses in one-pulse intervals. A manual sample collection can also be initiated. Sample volumes can be selected in 10-mL increments with a maximum volume of 500 mL and a single sample collection can also be manually initiated.

Samplers are powered by using a 110-volt AC charger or an external 12-volt battery. Basic programming steps for conventional sampling routines are listed below. For non-routine sampling programs, review the appropriate manufacturer instruction manual before deployment in the field.

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To Program:

1. Press "ON" button. The viewing window flashes a number indicating the active program step and then flashes a number indicating the choice selected for the active step. To enter a setting for the active program step, press the desired number and then press "ENTER VALUE" button.
2. Press "PROGRAM/STEP PROGRAM" button.
3. Program step 1 – Mode is selected. Enter desired sampling mode. (Ex: Press 1 and "ENTER VALUE" button to select 24 bottle sequential time mode).
4. Press "PROGRAM/STEP PROGRAM" button to advance to next step.
5. Program step 2- Interval btw Samples. Select desire time between sample aliquots. (Ex: For four samples per hour enter 15 and press "ENTER VALUE" button).
6. Press "PROGRAM/STEP PROGRAM" button to advance.
7. Program step 3 – Delay to first/next sample. Enter value to delay start of program from 1-9999 min.
8. Press "PROGRAM/STEP PROGRAM" button to advance.
9. Program step 4 – Nominal Sample volume. Set aliquot volume. Volume restrictions listed on sampler.
10. Press "PROGRAM/STEP PROGRAM" button.
11. Program step 5 – Type of suction line. Select sample tubing type used.
12. Press "PROGRAM/STEP PROGRAM" button.
13. Program step 6 – Suction Head. Select suction length from 1 to 20 ft.
14. Press "PROGRAM/STEP PROGRAM" button to advance.
15. Program step 7–Calibrate sample volume. Skip step.
16. Press "PROGRAM/STEP PROGRAM" button to advance.
17. Program step 8 – Multiplex mode. Select collection method for sequential mode.
18. Press "PROGRAM/STEP PROGRAM" button.
19. Program step 9 – Multiplex Number (sequential). This step only used in the sequential mode. Enter the number of samples to be taken per bottle.
20. Press "PROGRAM/STEP PROGRAM" button to advance.
21. Program step10 – Number of Samples (composite). This step only used when in composite mode. Enter the desired number of samples in the composite.
22. Press "PROGRAM/STEP PROGRAM" button.
23. "Press "START PROGRAM/RESET DISTRIBUTOR" button to start sampling program. The viewing window will flash between the bottle number, the sample number, and the time remaining until sample taken.

3.1.1.5 Isco Model 2710 and 3710

Automatic/Programmable Isco model numbers 2710 and 3710 are portable devices designed to collect a single composite sample from a liquid source. The samples can be collected at equal time intervals using the sampler's internal timing circuitry or at equal flow volume intervals using flow pulse inputs from an external flow meter.

In the time mode, the interval between samples can be set from 1 to 9999 minutes in 1-minute intervals. In the flow mode, the interval can be set from 1 to 9999 flow pulses in one-pulse intervals. A single sample collection can also be manually initiated. Samples are collected in a 9500 mL glass or polyethylene sample container.

The sampler is powered by a 110-volt AC charger or an external 12-volt battery. The basic programming steps for conventional sampling routines are listed below. For non-routine sampling programs, review the manufacturer's instruction manual before deployment in the field.

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I. Isco Model 2710:

1. Press "ON" button.
2. Press "PROGRAM/STEP PROGRAM" button. A light indicates the program step to be set. The numeric number displayed in the viewing window corresponds to the function listed below the indicated program step.
3. Enter numeric value desired for program step indicated by light.
4. Press "ENTER VALUE" button to set value.
5. Press "PROGRAM/STEP PROGRAM" button to advance to next program step.
6. Repeat Steps 3, 4, and 5 until all program features are set.
7. Press "START PROGRAM/RESET DISTRUBTOR" button to start sampling program.

II. Isco Model 3710:

1. Press "ON/OFF" key. The "STANDBY" message will appear above the time and date. An option is selected when it is flashing. Press the "EXIT PROGRAM" button at any time to return to the standby screen.
2. To set sample program press the "ENTER/PROGRAM" key and select "PROGRAM" by using the arrow keys and pressing the "ENTER/PROGRAM" button.
3. Time or Flow dependent sampling:
   a. For Time dependent sampling: enter sampling frequency in hours and minutes. Enter appropriate hour and minute interval (0 hr. and 15 min. for four samples per hour)
   b. For Flow Paced sampling, enter appropriate sample frequency in pulses (1-9999) when prompted.
4. Enter number of samples desired in composite (0-750).
5. Enter desired sample volume in milliliters (10-990) for each sample aliquot.
6. Enter suction head in feet.
7. Setting start time:
   a. If yes, select [YES] and enter time and date of first sample. Press "START SAMPLING". Screen will read "1 of 4, bottle 1 at (Programmed start time) (Current Time)"
   b. If no, select [NO] and press the "START SAMPLING" button to start program immediately.

3.1.1.6 Isco Model GLS

Automatic/Programmable Isco model GLS is a portable device designed to collect a single composite sample from a liquid source. Samples may be collected in a 9600 mL polyethylene or glass sample container. The samples may be collected at equal time intervals using the sampler's internal timing circuitry or at equal flow volume intervals using flow pulse inputs from an external flow meter.

In the time mode, the interval between samples may be set from 1 to 9999 minutes in one-minute intervals. In the flow mode, the interval may be set from 1 to 9999 flow pulses in one-pulse intervals. A single sample collection may also be manually initiated.

The sampler is powered by a 110-volt AC charger or an external 12-volt battery. The basic programming steps for conventional sampling are listed below. For non-routine sampling programs, review the manufacturer's instruction manual before going into the field.

To Program:

1. Turn on sampler. The standby viewing screen displays: "Program", "View Log" and

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current time and date.

2. An item is selected when it is flashing. Press the four-arrow button to move selection. Press the ENTER button which is pictured as "↵" to choose option selected.
3. Choose "Program." (Choosing View Log will allow user to scroll through past event log).
4. Choose "Time Paced" or "Flow Paced."
5. For "Time Paced", enter time in minutes between samples and press "↵□".
6. For "Flow Paced", enter the number of flow pulses between samples.
7. Enter the volume of the bottle used in the base Section and press " ↵ ".
8. Enter number of samples desired between 0-480. For example, when collecting four-100mL samples per hour, enter "96".
9. Enter appropriate sample volume to obtain necessary composite volume.
10. Enter time in minutes to first sample. An entered value of '0' indicates collection will begin immediately after sampling program is started.
11. Suction line size and length will be displayed. To change size or length, press the button with the picture of a ruler. Make desired changes to size or length. When asked to calibrate volume, select YES or NO.
12. Press the green diamond button to start sampling program.

To take grab sample:

1. Press button displaying a picture of a hand holding a test tube.
2. The size and length of suction tubing selected will be displayed. Make any desired changes and press "Enter".
3. The volume to be sampled will be displayed. Make any desired changes and press "Enter" to begin grab sample.

To manually run pump:

1. Forward:
   a. Press "3" while in the standby viewing screen.
   b. The screen will display for 4 seconds a message to press "Enter" to pump forward.
   c. Press "Enter."
2. Reverse:
   a. Press "1" while in the standby viewing screen.
   b. The screen will display for 4 seconds a message to press "Enter" to pump reverse.
   c. Press "Enter."

3.1.2 Isco Model Flow Meters

Pace FSD uses Isco 4230 and 4210 Model flow meters primarily as flow data acquisition devices. It is most commonly used for hourly flow rate data determination for the calculation of flow weighted sample aliquot compositing. Hourly, daily, weekly or monthly flow rate data may also be collected for process optimization or sewer capacity studies. A flow meter is also used to initiate sample collection for an automatic sampler programmed to equal flow volume intervals.

The Isco 4230 and 4210 flow meters are programmed for flow data acquisition using the unit's control panel or by the aid of a computer installed with Flowlink® software. The majority of site parameters can be programed in the shop rather than at the sampling location with the exception of adjusting level/parameters. When using the unit's control panel, refer to

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relevant instrument manual for specifics on keypad options. Select options from the displayed menu; a flashing item indicates that choice is currently selected. Press [Enter] to confirm the selection or use the arrow keys to move across the display to another selection. Use the Go To Program Step key to go directly to the program step needed.

NOTE: If "NOT MEASURED" is chosen for any selection, the flow meter will not display that value or function for the rest of the program (i.e., Value or function cannot be activated unless Step 1 is reprogramed). If a feature or option is needed but does not appear on the display, return to Step 1 to check that it has not been turned off in either the Program or Setup menus.
Flow data is obtained from the paper printout generated by the flow meter's onboard printer. Data can also be retrieved electronically using Flowlink® software in the event the paper printout is compromised, such as in the case of mechanical failure. A Rapid Transfer Device (RTD) can be used to transfer data from a flow meter to a computer (i.e., when a client's flow meter is used to measure flow).

3.1.2.1 Isco Model 4230 Bubbler Flow Meter

When measuring flow rate, the 4230 is used with a primary measuring device (e.g., weir or flume) or other open channel flow arrangement where a known relationship exists between level and flow rate, including flow calculation using the Manning Equation or a non-standard equation or data points. The level measuring device is a bubbler which measures the liquid level in the flow stream. As the liquid level increases, the pressure required to release the bubble also increases. The flow meter electronically converts the level reading using built-in standard level-to-flow conversions into a properly-scaled flow rate value.

A. Basic Programming using Unit Panel

1. Turn on unit. The prompt PROGRAM will be highlighted and flashing. Use the arrow keys to move to selection.
2. Press "ENTER/PROGRAM STEP" for Enter.
3. The following prompts will appear. The input responses for a typical project appear in "( )".
    a. Level Units of Measure (FT).
    b. Flow Rate Units of Measure (GPM).
    c. Totalized Volume Units of Measure (GAL).
    d. Rainfall Units of Measure (Not Measured).
    e. pH Units of Measure (Not Measured).
    f. Dissolved Oxygen Units of Measure (Not Measured).
    g. Temperature Units of Measure (Not Measured).
    h. YSI 600 Connected (No).
    i. Flow Conversion Type (Weir/Flume).
    j. Type of Device (choose weir or flume).
    k. Depending on the type of device selected, input the specific device information (size, configuration etc.).
    l. Maximum Head (1.000).
    m. Flow Rate Maximum Head (flow meter calculates and displays a flow rate value).
    n. Parameter to Adjust (None) level is adjusted only after the bubbler line is secured in the waste stream.
    o. Flow Totalizer (Enter).
    p. Reset Flow Totalizer (Yes).
    q. Enable Totalize (Enter).
    r. Reset Sampler Enable Totalizer (No).

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s. Sampler Pacing (Disabled).
t. Sampler Enable Mode (Disabled).
u. Plotter Speed (Off).
v. Report Generator A (On).
w. Report Duration (1 Hours).
x. Print First Report At (enter desired date and time- typically programmed during field deployment of unit to correlate with sampler start time).
y. Report Generator B (Off).
z. Print Flow Meter History (No).
aa. Clear History (No).
bb. Unit is now programmed.

B. Preparation, Installation and Removal

See Section 7.2.

3.1.2.2 Isco Model 4210 Ultrasonic Flow Device

When measuring flow rate, the 4210 is used with a primary measuring device (e.g., weir or flume) or other open channel flow arrangement where a known relationship exists between level and flow rate, including flow calculation using the Manning Equation or a non-standard equation or data points. The flow meter's ultrasonic level sensor emits a pulse several times a second. The time between the transmitted pulse and received signal is proportional to the distance between the sensor and the liquid surface. By comparing this distance with a referenced "zero" level for the flow stream, the flow meter converts the level reading using built-in standard level-to-flow conversions into a properly-scaled flow rate value.

A. Basic Programming using Unit Panel

1. To program the 4210 flow meter:
2. Connect flow meter to battery or other power source.
3. Turn on flow meter by pressing [ON] located on the keypad and wait for the display to settle. A clicking sound and level reading will appear if an ultrasonic level sensor is attached to the flow meter. If a sensor is not attached, a zero level reading with an asterisk will display, indicating an error reading.
   a. **Step 1, Operating Mode:** After turning on the flow meter, the following menu will appear:

SELECT OPTION • PROGRAM • • SETUP •

   i. Use the arrow keys to select SETUP, press [Enter]. Setup selects various basic "housekeeping" features for the flow meter such as setting the internal clock, site identification, measurement setup, hysteresis, report contents, operation of the display backlight, and program lock.
   ii. Selecting PROGRAM option allows selection of parameters correlated to the flow stream (e.g., units of measure for level, calculations, and reports). Press [Enter] to select PROGRAM to advance to the next display:

UNITS OF LEVEL MEASUREMENT • FT • • IN • • M • • MM • • NOT MEASURED •

   iii. Select appropriate units. Press [Enter]. The display will indicate:

FLOW RATE UNITS OF MEASURE

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• GPS • • GPM • • GPH • • MGD • • CFS • • CFM •

珏Continually press the right arrow key to see more options. Select appropriate units. Press

iv. Continually press the right arrow key to see more options. Select appropriate units. Press [Enter]. The display will indicate:

TOTALIZER VOLUME UNITS • GAL • • MGAL • • CF • • L • • M3 • • AF •

Select appropriate units. Press [Enter]. Continue through the remaining menus to set all flow parameters.

b. **Step 2, Flow Conversion Type** determines how the flow meter calculates flow rate and total flow. Select Step 2 to display the menu:

FLOW CONVERSION TYPE • WEIR/FLUME• • EQUATION• • MANNING• • DATA POINTS •

i. When the primary measure device is a weir or flume, select WEIR/FLUME to display the following menu:

TYPE OF DEVICE • WEIR • • FLUME •

ii. Choose the appropriate primary measuring device and continue through the next steps selecting the characteristics of the site.
iii. When the a non-standard primary device is used or different values from those programmed into the look-up tables of the flow meter are needed, select EQUATION to enter appropriate values for the flow situation.
iv. Select MANNING from the menu to use the Manning formula to calculate flow in open or closed gravity-flow situations based on slope, diameter, and roughness of pipe.
v. Selecting DATA POINTS, allows calculation of flow based on a set of user-entered data points for a flow stream. Similar to the Equation method, this flow conversion is most used with a nonstandard primary measuring device where tables of level and flow rate data are available from the device manufacturer.

c. **Step 3, Port to Adjust** allows calibration of measuring sensors. Select Step 3 to enter the measured level of the flow stream. When selected, the following menu will appear:

PORT TO ADJUST •NONE• •(LEVEL)• •(pH)• •(D.O.)• •(YSI 600)•

i. Select NONE to advance to the next step.
ii. Select LEVEL and enter the current measured level using the number keys and press [Enter]. Make sure the level is entered in the correct units displayed.
iii. To set the flow meter level, measure, as accurately as possible, the level in the flow stream. Enter this value with the numeric keys. Accuracy is important as this measured level provides the basis for calculated flow in the flow meter.

d. **Step 4, Reset Totalizer** provides the option to reset the internal flow totalizers. Typically when using the flow meter as a portable recording unit, moving it from one site to another, the totalizer is reset between sites. Select Step 4 to display the following menu:

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RESET TOTALIZER • YES • • NO •

i. Select NO to advance to the next step.
ii. Select YES to reset the internal totalizer to zero.

e. **Step 5, Sampler Pacing** provides the option for the flow meter to signal an associated wastewater sampler to take a sample after a certain volume has passed or a condition has been met. Select Step 5 to display the following menu:

SAMPLER PACING •DISABLE• •(VOLUME)• •(FLOWLINK)• •CONDITIONAL•

i. Select DISABLE when the flow meter is not required to send flow pulses to the sampler.
ii. Select CONDITIONAL to allow pacing of the sampler by the flow meter when a condition has been met (e.g., level, flow rate, temperature, rainfall).
iii. Select VOLUME to allow the flow meter to signal the sampler whenever a specific flow volume has passed.
iv. Select FLOWLINK to allow the sampler to be signaled from the flow meter as a result of conditions determined by Flowlink®.

f. **Step 6, Sampler Enable** allows the flow meter to control a sampler program when in a combination flow meter/sampler pairing. With sampler enabling, the flow meter can stop operation of the sampler. The sampler is still set up to run its own program, but the inhibit/enable line from the flow meter will determine when and whether the sampler runs its program. The conditions used for sampler enabling are similar to those used for sampler pacing (e.g., level, flow rate, temperature, rainfall).
g. **Step 7, Alarm Dialout Mode** allows the flow meter to signal a remote location, such as to signal alarm conditions, through a telephone line. This feature requires a modem installed and will not appear as an option if one is not present.
h. **Step 8, Printer** option allows the speed for chart advancement to be set from ½" to 4" per hour. Chart speed is set according to the amount of resolution needed – more activity equals a faster printer speed for easier interpretation.
i. **Step 9, Reports/History** programs the flow meter to print regular summary reports, up to two separate reports, on the internal printer. Reports typically include maximum and minimum flow rates, associated times, sample records, etc. Content of the reports are defined in Setup, Step 1. Step 9 merely allows the reports to be turned off and on and set the timing. History provides a record of changes made to the flow meter's program or operation events.

B. Preparation, Installation and Removal

See Section 7.3.

3.1.2.3 Partitioning/Down Loading/Exporting - Using Flowlink® Software

Refer to the Quick Start Procedure or instruction manual of the most recent installed version of Flowlink® software.

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3.1.2.4 Isco 581 Rapid Transfer Device (RTD)

The Isco 581 RTD is designed to transfer flow data from a compatible flow meter in the field to hard copy using Flowlink® software.

To retrieve data from flow meter:

1. Uncover interrogator port on flow meter.
2. Plug the Isco 581 RTD into the interrogator port.
3. The three LED's on the end of the RTD indicate the status of the data transfer.

    - The Yellow LED (Center LED) will blink indicating the unit has power.
    - The Green LED (Bottom) will blink indicating the data transfer is in progress.
    - When the Green LED stops blinking and changes to solid green light, the data transfer is complete.
    - If the Red LED is on, this indicates the memory is full.
    - If the Red LED begins blinking there has been a data transfer error.

4. Unplug the RTD after the data transfer is complete and recap the interrogator port.
5. Download data into the most recent version of Flowlink®.

3.2 General Inorganic Wastewater Sampling

3.2.1 Automatic Sampler Train Requirements

1. Pump head with a pump rate of at least 2 feet per second.
2. Container configuration of one 9600-mL composite container or twenty-four 1000-mL containers typically made of polyethylene.
3. Stainless steel strainer probe or weighted probe.
4. Tygon® sample tubing or equivalent.
5. Silicon pump head tubing.
6. Power source.
7. Ice for sampler base when applicable.

3.2.2 Automatic Sampler Train Cleaning Requirements

Refer to Section 2.1.1 for sampler container and probe cleaning procedure.

For Pump Head and Sample Tubing

1. Use new Tygon® or equivalent sample tubing for each monitoring event.
2. Check suction strength of pump head using a vacuum gauge.
    a. Pump vacuum should quickly reach between 25 and 30 inches of mercury, or equivalent.
    b. Pump pressure should hold after pump is stopped.
    c. If criteria are not met, change tubing.
3. To clean silicon pump head tubing:
    a. Pump hot soapy water through tubing for approximately one minute.
    b. Pump hot tap water through tubing for approximately one minute.
    c. Pump DI water through tubing for approximately 30 seconds.

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4. Secure sampler cover to protect tubing from ambient dust and other contaminants.

3.2.3 Automatic Sampler Quality Control Blanks

An equipment blank is typically not required for inorganic wastewater monitoring unless otherwise specified by particular project scope. Refer to Section 1.4 for Quality Assurance/Quality Control samples.

3.2.4 Automatic Sampler Setup/Takedown

Set-up

1. Position and secure probe in a well-mixed region of the waste stream. Avoid surface and bottom of waste stream when possible. Refer to Section 3.5 for specific site type descriptions.
2. A minimum of one sample aliquot is to be collected every half hour, unless otherwise specified by permit or project objectives. Pace FSD typically programs sampler to collect one aliquot every 15 minutes. Specific site conditions may require more frequent aliquot collection.
3. Collect sufficient sample volume per aliquot to ensure a representative sample volume for required composite parameters.

Take-down

1. Confirm sampler program is complete and a representative composite volume was captured based on facility's discharge information. Refer to Section 8.0. Contact project administrator regarding discrepancies.
2. Samples requiring thermal preservation are cooled to obtain appropriate temperature specified by parameter preservation requirements.
3. Perform necessary sample measurements and compositing as specified by client data sheet. Refer to Section 8.0 for compositing procedure.
4. Record samples on chain-of-custody and submit to laboratory. Refer to Section 9.0 for further detail.

3.2.5 General Grab Sampling

Refer to Section 3.6.

3.3 Priority Pollutant (PP)/Total Toxic Organic (TTO) Sampling

3.3.1 Automatic Sampler Train Requirements

1. Pump head with a pump rate of at least 2 feet per second.
2. Container configuration of one 9600-mL composite container or twenty-four 350-mL containers made of glass.
3. Stainless steel strainer probe.
4. Teflon® sample tubing or equivalent.
5. Silicon pump head tubing.
6. Power source.

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3.3.2 Automatic Sampler Train Cleaning Requirements

For Sampler Container and Stainless Steel Probe

1. Remove gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Triple rinse with organic free DI water.
5. Rinse with acetone and allow equipment to dry before use.
6. Wrap composite container mouth, distributor arm, and probe with aluminum foil (shiny side out).

For Pump Head and Teflon® Sample Tubing

1. Use new silicone pump head tubing and Teflon® sample tubing for each monitoring event.
2. Solvent rinse new tubing:
   a. Compressible Silicone Pump Tubing – thoroughly rinse with methanol.
   b. Teflon® Sample Tubing - thoroughly rinse with acetone.
3. Flush both tubing types with organic free DI water.
4. Cover tubing ends with aluminum foil (shiny side out).

3.3.3 Automatic Sampler Quality Control Blanks

1. After arrival at sampling location, assemble sampler train by attaching Teflon® sample tubing and stainless steel probe using a plastic zip tie.
2. Remove aluminum foil from composite container and distributor.
3. Attach sample tubing to pump head tubing.
4. Pump approximately 5-L of organic free DI water through the sampler train into the 9600-mL composite container, or into a representative number of the 350-mL glass containers.
5. Swirl DI water in container making contact will all surfaces.
6. Fill appropriate sample blank containers.
7. Complete label information and record on chain-of-custody.
8. Discard any remaining water from composite container.

3.3.4 Automatic Sampler Setup/Takedown

Set-up

After collection of equipment blanks, refer to Section 3.2.4.

Take-down

1. Confirm program is complete and a representative composite volume was captured based on the facility's discharge information. Contact project administrator regarding discrepancies.
2. Test composite sample for total residual chlorine following procedure outlined in Section 6.4 for low range analysis.
3. If chlorine is present, preserve each new 1-L unpreserved glass amber sample container with approximately 80 mg (one large pellet), or equivalent liquid measure, of sodium thiosulfate ($Na_2S_2O_3$).
4. Gently slosh composite container and fill labeled sample bottles.
5. Swirl bottles to mix and record samples on chain-of-custody.

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6. Place samples into cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
7. Record samples on chain-of-custody and submit to receiving laboratory.

3.3.5 Grab Sampling

Refer to Sections 3.6 Grab Sampling, 5.0 Parameter Specific Sampling, and applicable decontamination requirements outlined in Section 2.0.

3.4 Perfluorochemical Sampling - National Pollutant Discharge Elimination System (NPDES)

Perfluorochemical (PFC) analysis is performed at very low detection levels. Extreme care must be given to avoid contamination. Do not allow sampling probe or sample containers to come in contact with dirty surfaces. Nitrile gloves must be worn when handling equipment. A new pair of gloves is worn at each sampling location and gloves are changed after contact with possible contaminants.

The chemicals of interest are frequently used in the packaging of prepackaged food and fast food. Avoid the handling of these foods prior to and during equipment preparation, deployment and retrieval.

Teflon® must not be used as a component for any of the equipment that will come in contact with the sample or equipment blank water. Check fittings on DI water source to make certain Teflon® is not used.

The receiving laboratory will typically provide pre-filled trip blank, trip blank spike, sample bottle, pre-spiked sample bottle, and equipment blank. Trip blanks accompany sample bottles at all times. If sample bottles are not provided, bottles made from high-density polyethylene, or equivalent, are prepared according to cleaning procedures in Section 3.4.2.

3.4.1 Automatic Sampler Train Requirements

1. Pump head with a pump rate of at least 2 feet per second.
2. One 9600-mL composite container made from high-density polyethylene or equivalent. Variable grades may be approved for specific sampling locations and projects.
3. Stainless steel strainer probe.
4. Tygon® sample tubing or equivalent.
5. Silicon pump head tubing.
6. Power source.

3.4.2 Automatic Sampler Train Cleaning Requirements

For Sampler Container and Stainless Steel Probe

1. Remove any gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Triple rinse with organic free DI water.
5. Rinse with reagent grade methanol.
6. Rinse a final time with DI water to remove all traces of methanol.
7. Wrap composite container mouth, distributor arm, and probe with aluminum foil (shiny side out).

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For Pump Head Tubing and Tygon® Sample Line

1. New or dedicated silicon pump head tubing will be used at each sampling location. Dedicated tubing may be reused when decontaminated following procedure used for sampler container.
2. New Tygon® or equivalent sample line will be used for each sampling event.
3. Rinse tubing with reagent grade methanol.
4. Rinse with DI water to remove all traces of methanol.
5. Cover tubing ends with aluminum foil (shiny side out).

3.4.3 Automatic Sampler Quality Control Blanks

1. After arrival at the sampling location, assemble the sampler train by connecting the Tygon® or equivalent sample tubing and stainless steel probe using a plastic zip tie.
2. Remove aluminum foil from composite container and distributor.
3. Attach sample tubing to pump head tubing.
4. Place stainless steel probe into the DI blank water source and pump DI water through the system into the composite container.
5. Swirl DI water in composite container making contact with all surfaces.
6. Fill appropriate sample blank bottles.
7. Complete label information and record on chain-of-custody.
8. Discard any remaining water from composite container.

3.4.4 Automatic Sampler Setup/Takedown

Set-up

After collection of equipment blanks, refer to Section 3.2.4.

Take-down

1. Confirm program is complete and a representative composite volume was captured based on facility's discharge information. Contact project administrator regarding discrepancies.
2. Label sample containers.
3. Gently slosh composite container to mix and fill sample bottles in this order when applicable: sample, sample duplicate, sample low spike, and sample high spike. Fill bottles to appropriate fill line if one is provided.
4. Record samples on chain-of-custody and submit samples to receiving laboratory. ***Note:*** Due to the stability of the compounds of interest, chilling of samples is generally not required.

3.4.5 Grab Sampling

Refer to Section 3.6, Grab Sampling.

3.5 Site Type Procedures for Automatic/Programmable Sampling

3.5.1 Manhole Locations

1. Open manhole using proper tools such as a pick ax or pry bar.
2. Observe flow conditions. If more than one waste stream is present, determine appropriate discharge to sample.

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3. With appropriate probe securely attached, lower sample tubing into well-mixed portion of waste stream. Use tape or conduit, if necessary, to hold tubing/probe in position.
4. Program automatic/programmable sampler following appropriate model procedures in Section 3.1.1.
5. If location requires sampler to be hung in manhole, attach harness and hook to sampler. Hang unit from manhole step, ledge, secured 2 X 4, or the edge of manhole if located in lawn.
6. Replace manhole cover.
   a. If sampler does not fit into manhole, the cover may be propped open slightly using a 2X4 secured with lanyard or an additional probe taped to sampler tubing. Take precautions so sample tubing does not get pinched by 2X4 or cover.
   b. Place adequate barriers around the area when necessary.
   c. Upon take-down, open manhole and remove 2X4 from opening before detaching from lanyard to ensure 2X4 does not fall into manhole.

3.5.2 Clean-Out Locations

1. Open cleanout using proper tools.
2. Initiate the pump forward function of the sampler and lower tubing with probe into cleanout until sample is drawn up tubing with minimal air bubbles.
3. Secure tubing in position by taping to floor or other secure location.
4. Program automatic/programmable sampler following appropriate model procedures outlined in Section 3.1.1.
5. Adequately cover cleanout opening with duct tape or other material to prevent sewer gas escape. Be sure material is secure and will not fall into the opening.
6. Place adequate barriers around sampler when necessary.

3.5.3 Sump Locations

1. Open sump using proper tools.
2. Lower sample tubing with probe into sump and secure as close as possible to discharge pipe. Keep probe off bottom solids while being low enough to obtain sample during low flows.
3. Program automatic/programmable sampler following appropriate model procedures in Section 3.1.1.
4. Cover sump. The cover may be propped open slightly using a 2 X 4 secured with lanyard or an additional probe taped to sampler tubing.
5. Place adequate barricades around area when necessary.

3.6 Grab Sampling

3.6.1 Mechanical Sampling Devices

3.6.1.1 Kemmerer Sampler

The Kemmerer Sampler is a long cylindrical tube constructed of metal or plastic with rubber stoppers at each end used to sample water. The stoppers enable the operator to lower the open sampler to a specific depth, close the sampler, and retrieve a sample from that depth. It is designed to collect samples at defined depths within a water column.

All sampling should proceed from the area of lowest expected target analyte concentration to the area of highest expected concentration. At a minimum, remove visible contamination and flush with DI water and/or sample water between sampling locations. Certain projects require

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a higher level of decontamination. Refer to Section 2.1 for analyte specific decontamination procedures.

Metal samplers are suitable for organic and inorganic analytes but not for trace metals. Plastic models are suitable for trace metals and inorganic analytes but are preferably not used for sampling organics. **Note:** If sampling in accordance to EPA 1669 – Trace Metals, sampling equipment and containers must be specially prepared. Pace FSD obtains equipment from a certified vendor when sampling in accordance to EPA 1669.

Sampling Procedure

Before sampling, fasten line to sampler and secure other end to fixed object to prevent loss of sampler. Perform a trial run to ensure the sampler is working properly. A 3/16-inch or 1/4-inch braided nylon sound line is used with a flat-nosed 11-ounce or smaller messenger. Use of any other lines or messengers can result in damage or loss of equipment.

1. Pull open top stopper; this will also lock open the bottom stopper.
2. Rinse sampler with distilled water or sampler water before lowering the device.
3. Lower sampler to desired depth and close sampler by dropping messenger down the line.
4. Retrieve sampler and fill necessary sample containers.

3.6.1.2 Eckman Dredge Sampler

The Eckman Dredge sampler is used to collect sediments from the bottom of streams, lakes, ponds, sumps, storage tanks, or any other type of reservoir with an access point of 18 inches or larger.

The Eckman Dredge is a clamshell-type device constructed of brass. The collection of acidic sludge for metals analysis is avoided due to the potential leaching of copper and zinc from the sampler. A 3/16-inch or 1/4-inch braided nylon sound line is used with an 11-ounce sampler messenger.

All sampling should proceed from the area of lowest expected target analyte concentration to the area of highest expected concentration. At a minimum, remove visible contamination and flush with DI water and/or sample water between sampling locations. Certain projects require a higher level of decontamination. Refer to Section 2.1 for analyte specific decontamination procedures.

Sampling Procedure

1. Pull open the two clamshells and secure wire leads to anchor points.
2. Lower sampler to bottom of reservoir and close sampler by dropping messenger down the line.
3. Retrieve sampler and decant any liquid through the two doors located on the top of the sampler.
4. Fill appropriate sample containers by opening clamshell or scooping sample from the top doors.

3.6.1.3 Pond/Pole Sampler

A pond/pole sampler is a simple sampling device used for collecting liquids and slurries from sampling points that are difficult to access. A pond/pole sampler consists of a glass or plastic sample container or transfer device secured to an aluminum, fiberglass, or PVC pole. The

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pole is typically telescoping or available in multiple sections for extending reach.

All sampling should proceed from the area of lowest expected target analyte concentration to the area of highest expected concentration.

Sampling Procedure

1. Assemble sampler by securing sample container or transfer device into the holding mechanism.
2. Turn sampler so the mouth of the sample container or transfer device faces down, and insert into waste stream.
3. Turn sample container/transfer device right side up when the desired sampling depth is reached. Allow container to fill.
4. Raise pond sampler and cap sample container or transfer liquid to the appropriate sample bottle.

3.6.1.4 Coliwasa

Coliwasa is an acronym for Composite Liquid Waste Sampler. The coliwasa is comprised of a long rod made of glass or PVC with a check ball or plunger type valve on the end. A coliwasas is used to collect a representative cross sectional sample of a liquid contained in a drum or tank.

Sampling Procedure

1. Determine required number of coliwasa samplers needed. Most coliwasa samplers are disposable and designed for one-time use. Discuss with project administrator if multiple location sampling is appropriate as in compositing multiple drum samples into one sample for analysis.
2. Insert coliwasa to the bottom of sample point. Make certain the plunger/valve is open as it passes through the liquid.
3. Close the plunger/valve by pushing down on rod to enclose liquid within the coliwasa; withdraw coliwasa in this position.
4. Hold closed coliwasa over sample container and open plunger/valve to deposit liquid into bottle. A slight upward twisting motion may be needed to open the plunger/valve.
5. Repeat procedure until all required sample bottles are filled.
6. Appropriately dispose of coliwasa.

3.6.1.5 Pump

A pump and sample tubing is used to obtain sample for non-volatile parameters from cleanouts, sumps, tanks, and non-entry manholes. Avoid using a pump in the collection of VOCs and O&G/TPH samples when possible. Draw sample from a well-mixed region of the waste stream. Avoid sampling sludge layers in stagnant areas, such as the bottom of tanks and sumps.

A pump can also be used to collect a representative sample of a stratified liquid by collecting discreet samples from cross-sections of the sample point.

1. Determine total depth of liquid in the tank or sump. Determine characteristics of liquid to be sampled such as bottom solids or floating grease layers. Discuss with project administrator the appropriate number of depth increments to collect discreet samples.

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2. Mark tubing with necessary depth increments (e.g., every 6 inches, 12 inches).
3. Pump an equal volume of sample at each depth increment marked on the sample tubing.
4. Combine equal volume samples into a composite sample for analysis.

3.6.1.6 Bailer

A bailer is used to obtain water samples from difficult to access sample points. Bailers consist of a tube with a one-way check valve at the bottom. Bailers are made in various styles, materials, lengths and widths. Water fills the bailer as it is lowered into the source. The check value closes to contain the sample as it is retrieved. Minimize disturbance of water by gently lowering the bailer into the sample point. Allow the bailer to fill under own weight. Keep bailer away from sides and bottom of sample point; contact with surfaces may disturb particulate matter biasing the water sample. After it fills, use a smooth motion to slowly retrieve bailer.

Stainless Steel Bailer Decontamination

1. Remove any gross visible contamination by scrubbing and flushing with hot tap water.
2. Soak and scrub components in hot tap water containing laboratory grade soap.
3. Triple rinse with hot tap water.
4. Triple rinse with DI water.
5. Place components in oven for a minimum of 1 hour at 105°C or greater.
6. Wrap component in aluminum foil shiny side out.

3.6.2 General Grab Sampling Procedure

1. Determine bottle type and preservation requirements for analytes of interest.
2. Grab samples should be collected at mid-depth in a well-mixed region of waste stream. **Note:** O&G/TPH should be collected directly into sample containers when possible. Refer to Sections 3.6.3 and 5.0 for specific site procedures and collection methods.
3. If the project scope includes automatic wastewater sampling, the same automatic sampler system may be used to collect grabs. Collect all equipment blanks before pumping effluent through system.
4. Label sample bottles. Fill appropriate sample containers for analytes of interest.
5. Samples requiring thermal preservation are placed in cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
6. Log samples on chain-of-custody and submit samples to receiving laboratory. Refer to Section 9.0.

3.6.3 Site Type Procedures for Grab Sampling

3.6.3.1 Faucets and Taps

1. Sample point should be prior to any water conditioning devices such as water softeners or filters.
2. Remove screens from end of faucet when possible.
3. Open tap and purge for a minimum of one minute.
4. Collect sample directly into appropriate sample containers. Avoid overfilling. Refer to Section 5.0 for volatile organic compounds, semi-volatile organic compounds, bacteria, and drinking water metals.

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3.6.3.2 Tanks and Sumps

Grab sampling procedures for tanks and sumps depend on the conditions of the sampling point.

3.6.3.2.1 HOMOGENEOUS: Homogeneous or well-mixed tanks and sumps can be represented by a single grab sample obtained by dipping a transfer device, such as an new, unpreserved sample container or bailer, directly into the water and filling the appropriate sample containers. The project administrator or technician will determine the appropriate transfer device based on the analyte list. If direct access to the sample is difficult, a pump, pole sampler, or coliwasa is used to collect sample (refer to Section 3.6.1).

3.6.3.2.2 HETEROGENEOUS: Potentially heterogeneous (comprised of stratified regions of varied concentrations) tanks or sumps, or those with an unknown composition, are sampled in cross-sections or collected as a composite batch sample during discharge.

A cross-section sample is obtained by collecting equal volume aliquots from equally spaced depth increments using a pump. Cross-section sampling procedure is outlined in Section 3.6.1.5.

A composite batch discharge sample is obtained by collecting equal volume aliquots from a discharge point at equally spaced timed intervals. These equal volume aliquots are then combined and mixed prior to filling the required sample containers. If the discharge rate is varied, it may be more appropriate to base the aliquot collection time on the visual observation of the tank or sump level during discharge.

3.6.3.3 Clean-outs and Non-Entry Manholes

A pump is typically required to collect grabs from clean-outs. A pump can be used to collect samples from extremely deep manholes or from manholes with effluent streams of shallow depth. Avoid using a pump for VOC and SVOC sample collection when possible. A properly cleaned transfer device, such as a pole sampler or bailer can be used to collect sample from manholes with shallow to moderate depths and/or sufficient water depth in channel.

3.6.3.4 Entry Manholes

When a primary flow device is in place, collect grab samples on set-up day, when possible, to avoid biases due to the potential increase of solids upstream over time. For installed weir, fill required sample containers directly from waste stream as it passes over v-notch, or use an appropriate transfer device to avoid the loss of preservative. For installed flume, fill required sample containers directly from well-mixed region of waste stream, or use appropriate transfer device to avoid loss of preservative.

3.6.3.5 Drums

Drum sampling is typically conducted using a coliwasa. Refer to Section 3.6.1.4.

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4.0 GROUNDWATER WELL SAMPLING

4.1 Introduction to Equipment

Purging and sampling of groundwater monitoring wells is accomplished by a dedicated pump, non-dedicated pump, or bailer. Detailed equipment and specific procedures are specified on a project's client data sheet, client sampling plan, or Pace proposal. The technician assesses well characteristics and target parameters to determine appropriate purging and sampling equipment and procedures when not explicitly stated in project objectives. Refer to site history for past conditions and course of action, when available. The most commonly used purging and sampling devices are listed below. For other devices, refer to manufacturer instructions before use in the field.

4.1.1 A **peristaltic pump** uses a motor to drive rollers which compress flexible tubing. This creates suction drawing water up the tubing.

- Minimum I.D. well casing of 3/4 inch
- Maximum depth of 25 feet
- Works well for removing small volumes from shallow wells
- Requires 12V D.C. or 110V AC power source

4.1.2 The **2" submersible pump** is a cylinder with a sealed pump motor in the lower half. When submerged in fluid, a system of mechanical seals drives the fluid to the surface while preventing the fluid from entering the motor. A controller is used to vary the pump speed allowing management of the discharge rate.

- Minimum I.D. well casing of 2 inches
- Maximum depth of 250 feet
- Rapid discharge rate
- Works well for removing medium volumes from shallow to deep wells
- Requires 110 V AC power source

4.1.3 A **bladder pump** uses cycles of compressed air to alternately squeeze and relax a flexible bladder pushing water up the tubing. A check valve prevents back-flow from the discharge tubing. A controller is used to vary the cycle allowing management of the discharge rate.

- Minimum I.D. well casing of 2 inches
- Maximum depth 250 ft; most efficient at depths less than 150 feet
- Surging discharge
- Works well for removing small to medium volumes from shallow to moderate well depths
- Requires 12V car battery

4.1.4 A **bailer** is a hollow cylinder with a one-way check valve at the bottom. Bailers are made in various styles, materials, lengths and widths. Water fills the bailer as it is lowered into the water source. The check valve closes to contain the sample as it is retrieved. A bailer may be used to obtain water samples from difficult to access sample points, such as shallow or narrow diameter wells.

- Minimum I.D. well casing of 1-1/4 inch
- Single use to limit cross-contamination
- Works well for removing small to medium volumes from shallow to medium depth wells
- Requires apparatus to lower and raise

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4.1.5 A **Water Level Meter** is a portable instrument used to measure water levels within a well. A stainless steel probe is attached to a reel-mounted polyethylene coated tape. The sensor uses fluid conductivity to determine the presence of water. An audible signal and visible light activate when in contact with water.

4.2 Equipment Operation

4.2.1 Peristaltic Pump – GeoTech or Masterflex

1. Install new or dedicated tubing into pump for each sampling location. The same pump and tubing is used for purging and sampling when possible.
   a. Separate pump halves.
   b. Place sample tubing around rollers and turn rotor counterclockwise until tubing completely surrounds rotor.
   c. Position second half of pump over the motor shaft and snap shut being careful not to pinch tubing.
2. Connect power source to pump.
3. Slowly lower one end of tubing to the desired depth and secure to top of casing. Do not kink tubing.
4. Determine desired direction of flow and turn toggle switch appropriately before turning on power. If necessary, turn the speed dial to adjust the pumping speed.
5. Refer to Section 4.4 for purging process.
6. When finished sampling, remove suction line while pump is still running. Turn off pump only after suction line is out of well.

4.2.2 ISCO PTP-150 Portable Peristaltic Pump

1. Connect power source to pump.
2. Make sure the PUMP CONTROL switch is in the STOP position and the SPEED CONTROL is in the SLOW position.
3. Install new or dedicated tubing into pump if needed.
4. Slowly lower the intake tubing to the desired depth and secure to top of casing. Do not kink tubing.
5. Place the PUMP CONTROL Switch in the ⇒ (clockwise) position. The tube on the left side of the pump will intake liquid and discharge it from the right side of the pump. Adjust the SPEED CONTROL to a level appropriate for the type and volume of sampling occurring. NOTE: To reverse direction of flow, switch the PUMP CONTROL to the opposite ⇐ (counter- clockwise) direction. The pump will intake from the right side of the pump and discharge it on the left side clearing the line.
6. Refer to Section 4.4 for purging process.
7. When finished sampling, remove suction line while pump is still running. Turn off pump only after suction line is out of well.

4.2.3 2" Submersible Pump

RediFlo Pump Controller used with Grundfos RediFlo 2" Submersible Pump

Use the RediFlo Pump Controller keypad to program parameters, operate motor, and monitor status of control. NOTE: Do not use Grundfos submersible pump for PFC sampling due to Teflon ports.

- [PROG] Press to enter program mode. Parameter settings are edited in program mode.
- Use [▲] and [▼] to change the value of the parameter displayed and to scroll through lists.

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- [ENTER] Press to select operations. Press to save value changes and to move back to previous level in programming menu.
- [JOG] Press to select preprogrammed jog speed while in local mode.
- [FWD] Press to initiate forward motor rotation.
- [REV] Press to initiate reverse motor rotation.
- [STOP] Press to stop motor.
- [DISP] Press to return to display mode. Provides operational status and advances to next menu item. Use to access diagnostic and troubleshooting screens.
- [RESET] Press to clear messages.
- [SHIFT] Press to move cursor while in program mode.

1. Connect tubing to pump. Use new or dedicated tubing for each sampling location. The same pump and tubing is used for both purging and sampling when possible.
2. Connect to power source and slowly lower submersible pump into well to a depth just below the static water level. Reverse the natural bend of the coiled tubing when lowering the pump into the well, so that the tubing will straighten as it descends. Do not kink tubing.
3. Once on, the control will initialize and a display will appear.

    - STP indicates the drive is stopped.
    - V indicates motor volts
    - A indicates motor amps
    - Hz indicates motor frequency
    - LOC indicates the drive is in Local Keypad Mode
    - The default pump Redi-Flo 2 will also be displayed.

4. Operate the pump vertically with the motor always submerged in fluid. If the static water level drops below the pump inlet, continue to lower pump until a sufficient and stable discharge rate is reached or until the well pumps dry.
5. Press [FWD] to start the pump. Start the motor at a minimal speed and gradually increase or decrease as needed using [▲] and [▼].
6. Refer to Section 4.4 for purging process.
7. When finished sampling, pull pump out of well while pump is still running.
8. Stop pump.

4.2.4 Bladder Pump Controllers – Geocontrol Pro or GeoPump Model 5100

A. Geocontrol Pro

The Geocontrol Pro controller is used for operating down well bladder sampling pumps. The fill rate valve controls the pump compression and release cycle allowing control of the pump rate. Adjust the discharge and fill timer knobs to obtain optimal cycle time.

The discharge time knob is used to adjust the time it takes to compress the bladder and push water out of the pump. A longer discharge time is required for deeper wells or larger bladder volumes. Fill time is the time allowed for the bladder to refill. Larger bladder volumes and slower fill rates require longer fill times. Setting the controller fill timer to zero allows for continuous pumping.

1. Use new or dedicated tubing for each sampling location. The same pump and tubing is used for both purging and sampling when possible.

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2. Connect sample tubing to the sample line port and connect air line tubing to the air line port at the top of the bladder pump. The air line port is smaller than the sample line port.
3. Slowly lower the bladder pump into the well to a depth just below the static water level. Reverse the natural bend of the coiled tubing when lowering the pump into the well, so that the tubing will straighten as it descends. Do not kink tubing.
4. Lower pump into well and then connect pump to power source.
5. Turn on controller and adjust the fill time and discharge rate to obtain the desired flow.
   a. If the discharge from the pump stops before the discharge cycle is complete, the fill time is set too low relative to the fill rate, or the discharge time is too high and the pump is empty.
   b. If the discharge cycle ends before the pump is finished discharging, the discharge time should be increased to maximize flow from the pump.
   c. If a quick pressure increase is noticed on the air pressure gauge, turn down the discharge time until the compressor turns off at the time of increase.
6. Refer to Section 4.4 for purging process.
7. When finished sampling, remove pump and tubing from well.

B. GeoPump Model 5100

The GeoPump Model 5100 controller provides a highly portable means to cycle compressed air to a bladder pump. It is a fully self-contained unit supplied in a lightweight and rugged carrying case. The unit includes a panel mounted pressure gauge that indicates the air pressure being delivered to the pump, a flow control adjustment to increase or decrease the air pressure, as well as independent pump discharge and refill timers.
The controller is powered by a rechargeable battery.

1. Use new or dedicated tubing for each sampling location. The same pump and tubing is used for both purging and sampling when possible.
2. Attach the 10-foot air line from the controller to the pump tubing, then attach the 25-foot air hose from the controller to the air supply.
3. Lower pump into well to a depth just below the static water level and then connect pump to power source. Do not kink tubing.
4. Turn on controller and adjust the fill time and discharge rate to obtain the desired flow.
   a. It is suggest to set the discharge-timing knob to the 9-o'clock position, and the refill-timing knob to the 9-o'clock position. This corresponds to approximately 10 seconds for each cycle.
5. Turn on the drive air source and move the toggle switch to the "on" position.
6. Allow the controller go through several full cycles before attempting to re-adjust the timers. Once the pump tubing is primed with water and begins to discharge at the surface then the timers can be reset for optimum operation.

   NOTE: *Verify that the pressure setting is at or above the minimum value of 0.5 psi per foot of depth to allow the pump to discharge properly to the surface.*

   a. If the discharge from the pump stops before the discharge cycle is complete, the fill time is set too low relative to the fill rate, or the discharge time is too high and the pump is empty.
   b. If the discharge cycle ends before the pump is finished discharging, the discharge time should be increased to maximize flow from the pump.
   c. If a quick pressure increase is noticed on the air pressure gauge, turn down the discharge time until the compressor turns off at the time of increase.
7. Refer to Section 4.4 for purging process.
8. When finished sampling, remove pump and tubing from well.

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4.2.5 Mega-Monsoon Stainless Steel Pump

Mega-Monsoon Stainless Steel Pump and Low Flow Controller with Power Booster

CAUTION: The motor module gest extremely hot when in use. Do not touch inside of pump if the pump has been running continuously for over one minute.

WARNING: Do not turn on pump if the outside pump housing is not attached to the motor housing top connector.

1. Check to ensure the controller dial is in the off position.
2. Connect controller to 12 volt power source.
3. Connect pump to controller.
4. If requiring a low-flow rate, connect the PVC Low Flow Sampling Valve to the end of discharge hose. Adjust the dial on the value to obtain flow rates of 10 mL per minute or lower.
5. Slowly lower pump into well until it reaches the designated depth in water.
6. Slowly increase controller voltage until desired water flow is achieved. To obtain the lowest possible flow rate, make sure the dial on the controller is set to the lowest voltage reading to achieve desired flow. Do not use the control flow valve when the discharge rate is greater than 100 mL per minute as it will cause motor damage.
    a. Turn dial clockwise to increase voltage and increase flow rate.
    b. Turn dial counter clockwise to decrease voltage and reduce flow rate.
7. A pulsating pump indicates the battery is low and needs to be charged.

4.2.6 Bailer

1. Anchor desired line or set-up surveyor's tripod and down rigger reel above the center of the well casing.
2. Connect bailer to line or down rigger cable.
3. Keep bailer and line away from sides and bottom of well. Contact with these surfaces may disturb particulate matter biasing the sample. Minimize disturbance and aeration of water by gently lowering the bailer into the sampling point; avoid splashing bailer into water.
4. Allow bailer to fill under own weight.
5. Slowly withdraw bailer from the well using a smooth, steady motion.
6. Measure and record each volume removed by pouring sample into clean, unpreserved container. Perform stabilization readings as specified by project objectives. Refer to Section 4.3.3 and 4.4.
7. Once stabilization criteria have been met, collect required samples. Refer to Section 4.5.

4.2.7 BUuck Controller

The BUuck Controller is used to control water flow from 12V DC submersible pumps during groundwater well purging events. It has variable speed control and can be used for high or low flow rates.

1. With knob in OFF position, connect controller to pump.
2. Then connect to 12V DC source.
3. Turn knob to desired flow rate:
    a. Turn Clockwise to increase water flow
    b. Turn counter-clockwise to decrease flow.
    c. Turn controller OFF by turning knob a full counter-clockwise.

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4. Fault Protection Reset: Identify and correct fault source then push resettable circuit breaker button back in.

4.2.8 Global Water Flow Probe

The Global Water Flow Probe is used to measure the average and maximum water velocities through streams and pipes. It takes one reading per second and averages these readings to calculate velocity. As long as the probe remains in the flow, this continuous average is displayed. For example, after 10 seconds, 10 readings are totaled and then divided by 10 and this average is displayed. Once the average reading becomes steady, the true average velocity of the stream is obtained. Lifting the probe from the water, the average value is frozen on the display until it is reset.

A. Probe Operation:
   1. Check the propeller by blowing strongly on the prop; it should turn freely. If the propeller gets fouled while measuring flow, clean it until the prop turns freely and start over.
   2. Point the propeller directly into the flow to measure. The arrow inside the prop housing should point downstream.
   3. Expand the probe handle rod for correct placement in flow by loosening the locking nut on the handle, pulling out the top piece, and then retightening the nut.
   4. Use the bottom button to scroll through the functions until "AVGSPEED" appears. The top number displays the instantaneous velocity to the nearest .5 ft/second. The lower displays the average velocity.
   5. Place the propeller at the desired measuring point and hold the top button for 3 seconds to clear the value or 5 seconds to clear both average and maximum values. DO NOT submerge the top of the pole handle and the computer.
   6. Hold the probe in place until the reading becomes steady, then remove the probe from the water.
   7. The average and maximum velocities will remain on screen as these values are only updated while the propeller is turning.

B. Flow Type
   1. For small streams and pipes move the probe slowly and smoothly throughout the flow during average velocity measurement by moving the probe smoothly and evenly back and forth from top to bottom of the flow so that the probe stays at each point in the flow for approximately the same amount of time. Keep moving the probe for 20-40 seconds to obtain an accurate average value that accounts for surging.
   2. For larger streams and rivers: If the Flow Probe cannot easily be moved throughout the flow, divide the stream into subsections 2-3 feet wide. Run a measuring tape across the stream for reference. Obtain a vertical flow profile at the center of each subsection: zero the averaging function and move the Flow Probe vertically from the surface to the bottom, up and down, slowly and smoothly for 20-40 seconds to obtain a good average.
   3. The average velocity (obtained with the Flow Probe) times the area of the subsection equals the flow for the subsection (Q=VxA).Once the flow of each subsection is obtained, add all of the subsection flows to obtain the Total Streamflow.

C. Calculation of Flow
   1. Measure or calculate the cross-sectional area of the flow stream in square feet. For round pipes, measure the depth of water and determine the cross-sectional area. For other channels, manually measure water depth at several points across the flow. Drawing a diagram on graph paper with a scale of 1 square foot per graph paper square may be helpful. Cross-sectional area (in square feet) can then be found by

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counting the number of squares in the stream.

2. The average velocity (calculated with the Flow Probe in feet/second) times the cross-sectional area (square feet) equals flow in cubic feet per second (cfs), or Q = V x A.

4.3 Stabilization

A representative sample of groundwater formation is collected by purging stagnant water from the well prior to sample collection. The purging process includes determining the volume of water in the well casing, purging stagnant water from the well, and monitoring this purged water for water quality indicator parameters.

Static water level, the level of water in an undisturbed well, and total well depth are measured and recorded for all wells. The static water level is measured and recorded before purging and after sampling at each well. Total well depth may already be established and provided on the client data sheet, client sampling plan, or Pace proposal. If total well depth is not predetermined, use a water level indicator to obtain measurements.

Static water level and total well depth determine the well purging volume and identify the proper intake depth during purging and sampling procedures. They are also used to identify the direction of groundwater flow. To establish a measurement for water levels, a minimum of two water level measurements are taken with values agreeing within 0.01 foot.

4.3.1 Water Level Determination

Procedure

1. Hold the water level meter vertically above the well case opening and take all measurements from the point marked at the top of the well casing. Do not rub the tape against the top of the casing as it is lowered and raised; cover sharp edges to protect tape if necessary.
2. Thoroughly rinse probe with DI water and perform sensitivity calibration. Setting the meter's sensitivity will avoid false triggering.
    a. Turn instrument on using the ON/SENSITIVITY switch.
    b. Lower probe into well. The light and buzzer will activate when the probe contacts the water surface.
    c. With the probe still in contact with the water, turn the ON/SENSITIVITY dial counter-clockwise until the light/buzzer turns off. Then turn the dial clockwise until the light/buzzer barely activates.
3. Determine the static water level.
    a. Slowly lower the water level indicator probe down into the well until the indicator light comes on and/or the buzzer sounds. Dip the probe in and out of the water several times to confirm the exact point at which the probe is hitting the water.
    b. Take reading from the tape at the appropriate reference point. Measure a second time to confirm initial measurement. Measurements should agree within 0.01 foot. Take additional readings if necessary.
    c. Record static water level to the nearest 0.01 foot (meters x 3.281 = feet) on the Field Data Log Sheet.
4. Determine the total well depth, if necessary.
    a. Lower probe into the well until it hits the bottom and take reading from the appropriate reference point. Measure a second time to confirm initial measurement. Measurements should agree within 0.01 foot. Take additional readings if necessary.

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b. Record total well depth to the nearest 0.01 foot (meters x 3.281 = feet) on the Field Data Log Sheet.
c. After completing required measurements, rewind the tape being careful not to rub it against the casing.

5. Clean meter after use at each well.
   a. For static water level and total well depth measurements, wash probe with soapy water and thoroughly rinse with DI water.
   b. Additionally, after total well depth measurement, wipe any tape having contacted well water with a DI soaked tissue, or equivalent, while reeling in tape.

4.3.2 Well Volume Determination

Determine the well volume for the purging process using static water level, total well depth, and well diameter measurements. Water column length is found by subtracting the static water level from the total well depth.

Water Column length (ft) = Well Depth (ft) (-) Static Water Level (ft)

Water column length is then multiplied by the well diameter multiplication factor to obtain the well water volume. Refer to the Well Volume Determination Table (Attachment 12) to determine the water volume within circular well casings.

Well Volume (gal) = Water Column length (ft) (x) Well Diameter Multiplication Factor

4.3.3 Water Quality Indicator Parameters (WQIP)

Water quality indicator parameters (WQIP) are monitored to determine when formation water has been reached during the purging process. WQIP are measured after each water column volume or partial volume, depending on project specifications, is purged. Purged water is directed through a flow cell to minimize changes in temperature, pressure, and influences of outside elements while WQIP are measured.

The client data sheet, client sampling plan, or Pace proposal specify the parameters and frequency of measurement in determining well stabilization for a given sampling location. Turbidity and ORP measurements are generally not WQIP, although these parameters are measured in the field per client request or project objectives. Monitor WQIP carefully to keep purging to a minimum. Excessive purging can damage the monitored zone.

A well is presumed to be ready for sample collection when at least three successive readings for each WQIP are observed to vary less than the following criteria, unless otherwise specified by project objectives:

Well Stabilization Criteria	
pH	± 0.1 s.u.
Sp. Cond.	± 5%
Temp	± 0.5 °C
DO	± 0.5 mg/L
Turbidity	Project Specific
ORP	Project Specific

From Quick Reference Tables (Attachment 12)

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4.4 Purging

Purging is the process of removing stagnant water from a well before sampling, which then allows a representative groundwater sample to be collected from the adjacent formation water flowing into the well. Typically, purging is completed immediately before sample collection, although samples are acceptable if collected within 24 hours of purging. Project specifications may require sampling to occur within a set time after the purging process.

All attempts are made to avoid purging wells to dryness. Excessive purging can increase or decrease the contaminant concentrations that would otherwise be found in a representative sample from a well. Monitor the static water level throughout the purging process to track drawdown and assist in flow rate adjustments. Reduce pump rate to avoid pumping the well dry and to ensure stabilization criteria are achieved when possible.

For a well that has been purged dry, it is assumed recharge water is fresh from the groundwater formation. Under ideal conditions, a well that has been purged to dryness is permitted to recover 100% before sample collection. At a minimum, allow a well recharge for a period of time sufficient for an adequate water volume to return for the desired sampling parameters. Check the recharge of the well using a water level meter until a sufficient depth is measured to ensure a sufficient amount of water is in the well before sampling. If the recharge time is relatively long (> 1 hour), it is up to the discretion of the technician and client as to whether sampling proceeds.

Procedure

1. Start purging well by withdrawing water from desired depth using appropriate purging procedure for project objectives and well conditions. Use a purge rate dictated by project's sampling plan; if a rate is not indicated, use a purge rate that will minimize drawdown while allowing a reasonable purge time. Check the water level during purging to track drawdown; adjust the pump depth as necessary if the water level drops.
2. Measure and record the purge rate using a bucket and stopwatch.
    a. Collect the purging discharge into a bucket with volumetric markings for a timed interval (15 seconds, 30 seconds, 1 minute).
    b. Determine the purge rate in gallons per minute, or equivalent units, by multiplying the set length of time and the volume collected in the bucket.
3. Inspect flow cell to ensure proper sealing. Connect discharge line to the intake point of the flow cell. Maintain discharge through flow cell(s) at a continuous and steady rate.
4. Immerse applicable field analytical probes into flow cell(s) to measure stabilization criteria. Allow probes to equilibrate for approximately 5 minutes before taking readings.
5. Remove one well volume or partial well volume.
6. Record WQIP readings on appropriate data sheet.
7. Continue purging until another well volume or partial well volume has been removed.
8. Record WQIP on appropriate data sheet.
9. Continue process until 3 or more consecutive measurements within the specified target stabilization criteria are obtained. Refer to Section 4.3.3. If stabilization criteria has not been met after the removal of 5 water column volumes, contact project administrator or client for approval to collect sample. Clearly document that stabilization was not achieved if sampling is to proceed.
10. Collect required samples. Refer to Section 4.5.

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4.5 Sample Collection

Analytical parameters required for sampling are based on regulatory requirements and site history. These analytical parameters are specified on a project's client data sheet, client sampling plan, or Pace proposal. Samples are collected in a manner that minimizes potential contamination. A new pair of gloves is worn at each sampling location and gloves are changed after contact with possible contaminants. If vehicles or generators are running during sample collection, containers are filled upwind from exhaust sources. Samples are placed on ice for cooling immediately upon collection.

Procedure

1. After well stabilization is achieved and documented, complete necessary information on sample labels and adhere to appropriate sample containers.
2. If provided, use the sample rate specified by project's sampling plan. If not provided, the sampling rate is the same as the rate used during the final stage of purging. Sample rate is lowered significantly when sampling for VOCs to approximately 100 mL per minute.
3. Remove discharge line from flow cell maintaining a continuous discharge and collect required QA/QC samples.
4. Collect field samples in the following order if applicable, unless otherwise specified. Do not overfill preserved sample bottles. Refer to Section 4.7 for filtered metals if necessary.

    i. VOCs
    ii. Metals
    iii. SVOCs
    iv. TOC
    v. TOX
    vi. Phenols
    vii. Nitrogen series
    viii. Cyanide
    ix. General Parameters

5. Log all samples on chain-of-custody. Refer to Section 9.0.
6. Samples requiring thermal preservation are placed in cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
7. After all samples have been collected, measure and record the static water level to the nearest 0.01 foot. Refer to Section 4.3.1.
8. Complete relevant paperwork for sampled well.
9. Thoroughly clean all equipment to be used on additional wells before moving to next location. Refer to Section 2.2.2.
10. Review project scope to confirm all requirements have been fulfilled before leaving the site.

4.6 Low-Flow Well Sampling

A well is determined to be a low-flow well when the rate at which water enters the pump intake area from the formation is extremely slow. Low-flow does not refer to the discharge rate of well water during purging or sampling. Wells with extremely slow recharge rates require alternative purging and sampling procedures.

For low-flow wells, the well is not purged, but rather, the water surrounding the pump inlet is continually replaced with fresh formation water without disturbing the remaining water column. The pump remains in the well and is allowed to recover until a sufficient volume returns to permit sample collection. If well recovery is so slow that an adequate water column height is not reached within a reasonable time, a zero submergence bladder pump or bailer is used for sample collection.

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Low-flow purging and sampling is performed from the middle or slightly above the middle of the screened interval within the well. Purging and sampling too close to the bottom of the well can introduce solids into the pump and sample water. Pump in a manner that minimizes stress; monitor changes in the static water level to determine suitable purging and sampling rates. Typical flow rates for low-flow sites are between 100-500 mL per minute. Specific well information is provided on a project's client data sheet, client sampling plan, or Pace proposal.

Procedure

1. Perform necessary water level readings and determine well water volume. Refer to Section 4.3.1 and 4.3.2.
2. When sampling low-flow wells, an adjustable rate controlled submersible or bladder pump is preferred. An adjustable rate peristaltic pump is used when depth to water is 20 ft or less. Refer to Section 4.2 for pump operation.
3. Slowly lower pump into well. The pump intake should be within the screened interval.
    - Teflon or Teflon-lined polyethylene tubing is preferred when sampling for organic compounds.
    - Polyethylene tubing may be used when sampling for inorganics.
4. Pump at a rate specified by project's sampling plan. If a flow rate is not specified, use a purge rate between 100 to 500 mL per minute. Maintain a steady flow rate while intermittently measuring the water level within the well to maintain a drawdown of less than 0.33 feet.
5. Measure and record the discharge rate using a bucket and stopwatch.
    a. Collect the discharge into a bucket with volumetric markings for a timed interval (15 seconds, 30 seconds, 1 minute).
    b. Determine the discharge rate in gallons per minute, or equivalent units, by multiplying the set length of time and the volume collected in the bucket.
6. Remove a minimum of one well volume prior to recording water quality indicator parameters. Monitor and record WQIP every 3 to 5 minutes unless otherwise specified. Refer to Section 4.3.3 for stabilization information.
7. Once stabilization has been reached, maintain the same discharge rate used for purging or reduce slightly for sample collection (100 to 500 mL per minute).
8. Collect required samples directly from the pump tubing. Refer to Section 4.5 for sample collection. Refer to Section 4.7 for filtered metals if necessary.
9. Log all samples on chain-of-custody. Refer to Section 9.0.
10. Record appropriate water level readings, if necessary. Refer to Section 4.3.1.

4.7 Sample Filtration for Metals Analysis

Samples requiring filtration are collected in two ways: in-line or non in-line. For inline filtration, a filter is connected to the pump sample tubing. Sample is then collected directly from the well, through the filter, into the required sample containers. For non in-line filtration, sample is first collected in a container and then filtered from the container into the required sample containers. In-line filtration is preferred, when possible. Do not filter sample if total metal count is to be analyzed.

Procedure

In-line filtering (Directly from well):

1. Connect filter directly to pump sample tubing; use tubing specified in sampling plan for pump and filter discharge lines.
2. Force sample through desired filter with positive pressure using a flow rate less than or equal to 500 mL per minute.

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3. Flush new filter with fresh sample water for approximately 5-10 seconds before sample collection.
4. Collect sample directly into appropriate sample containers. Record all samples on chain-of-custody.
5. Shut off pump and discard filter.

Non in-line filtering (From collection container):

1. Install new, properly cleaned silicone tubing in peristaltic pump for each sampling location. Connect discharge side of pump tubing to disposable 0.45 micron filter assembly.
2. Put suction side of peristaltic pump in aliquot of sample to be filtered. This sample is drawn from bailer or a new, unpreserved laboratory sample bottle.
3. Collect sample directly into appropriate sample containers. Record all samples on chain-of-custody.
4. Shut off pump and discard filter and pump tubing.

5.0 PARAMETER SPECIFIC SAMPLING

5.1 Volatile Organic Compounds (VOCs)

Sample collection for volatile organic analysis must limit the volatilization of the target compounds within the sample. Agitation and aeration of sample must be limited to the least amount of disturbance as possible. A sample for volatile organic compounds (VOCs) typically consists of three or four replicate 40-mL glass vials preserved with hydrochloric acid (HCL) collected from the same source at the same time. Multiple vials allow for possible breakage and to ensure adequate sample volume for QA/QC procedures. A set of travel/trip blanks is collected when sampling for VOCs.

Volatile samples are separated from other samples to prevent contamination when known volatiles or strong smells are present, or when other factors indicate such precautions are necessary. This separation includes enclosing vials in bags or storing in separate coolers.

5.1.1 Collection from Wastewater, Surface Water, and Drinking Water Sources

Grab samples are collected at mid-depth in a well-mixed region of the waste stream. If a transfer device is required for collection, use a non-plastic device cleaned following procedure in Section 2.1.3 when possible. Follow the procedure below. Fill out a sample label for each individual vial; do not attach sample labels until Step 7.

1. Test effluent for total residual chlorine using a chlorine test strip or the procedure in Section 6.4 for Low Range Testing.
2. If no detectable chlorine, proceed to Step 5.
3. If chlorine is detected (> 0.00 mg/L), sodium thiosulfate ($Na_2S_2O_3$) must be added to the sample. Add approximately 80 mg (one large pellet), or equivalent liquid measure, of $Na_2S_2O_3$ into a 1000-mL beaker cleaned as specified in Section 2.1.3, or into a new 1-L unpreserved glass amber sample bottle.
4. Fill container with 1000 mL of sample. Gently swirl to mix.
5. Slowly fill each vial so a convex (positive) meniscus is formed at the rim. Avoid excessive overfilling which dilutes the HCL preservative. Immediately cap vial.
6. Check for trapped air by inverting vial and gently tapping the vial to the palm of hand. Zero headspace is a requirement for VOC sample collection. If an air bubble is present, discard vial and repeat collection procedure with a replacement vial. If unable to collect zero headspace after several attempts, note on the chain-of-custody the inability to collect air free vials and the perceived cause (e.g., oily, soapy).

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7. Complete sample labels and attach to vials.
8. Place each set of vials (one sample) into zip-seal bag or bubble wrap and place into cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
9. Complete chain-of-custody and submit to analytical laboratory (Section 9.0).

5.1.2 Collection from Groundwater Wells

VOC collection from groundwater monitoring wells is performed using a slow, steady sample rate. If not otherwise specified by sampling plan, use a rate of approximately 100 mL per minute for VOC collection. If a collection/transfer device is required, use a new, bottom emptying, disposable bailer. Fill out a sample label for each individual vial BEFORE attaching to vial. Follow procedure in 5.1.1 starting with Step 5.

5.2 Semi-Volatile Organic Compounds (SVOCs)

Semi-Volatile Organic Compounds (SVOCs) are a subset of VOCs that do not volatilize as readily under standard temperature and pressure. A sample for SVOCs typically consists of two replicate 1-L unpreserved glass amber bottles collected from the same source at the same time.

5.2.1 Collection from Wastewater, Surface Water, and Drinking Water Sources

Grab samples are collected at mid-depth in a well-mixed region of the waste stream. If unable to collect sample directly from waste stream, use a non-plastic transfer device cleaned as specified in Section 2.1.2 when possible.

1. Test effluent for total residual chlorine using a chlorine test strip or the procedure in Section 6.4 for Low Range Testing.
2. If no detectable chlorine, proceed to Step 4.
3. If chlorine is detected (>0.00 mg/L), sodium thiosulfate ($Na_2S_2O_3$) must be added to the sample. Add approximately 80 mg (one large pellet), or equivalent liquid measure, of $Na_2S_2O_3$ into each new 1-L unpreserved glass amber sample bottle.
4. Slowly fill each bottle in a way that limits bubbles and overfilling. Fill bottle completely full, eliminating as much air space as possible.
5. Immediately cap sample. If $Na_2S_2O_3$ was added, gentle swirl bottle to mix.
6. Confirm all required information has been completed on the labels and place samples into cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
7. Complete chain-of-custody and submit to receiving laboratory (Section 9.0).

5.2.2 Collection from Groundwater Wells

1. Slowly fill each bottle directly from the discharge tubing or from a new, bottom emptying, disposable bailer. Fill at a rate that limits aeration; approximately 100 mL per minute unless otherwise specified.
2. Fill bottle completely full, eliminating as much air space as possible, and immediately cap sample bottle.
3. Confirm all required information has been completed on the labels and place samples into cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
4. Complete chain-of-custody and submit to analytical laboratory (Section 9.0).

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5.3 Bacteria

Exercise extreme care to avoid contamination by external bacteria not associated with the water source while collecting samples for bacteria analysis. Store sterile sample containers in a clean zip-seal bag until needed. A new pair of latex or nitrile gloves is worn at each sampling location and changed after contact with possible contaminants. Do not touch the inside of the container or lid; discard container if contact occurs.

5.3.1 Collection from Wastewater, Surface Water, and Drinking Water Sources

1. Remove screens, washers and aerators from sample outlet when possible.
2. Sterilize sample outlet by heating with a torch or by flushing with strong chlorine bleach solution (10% bleach solution). Use caution when using a torch to avoid scorching the spigot.
3. Purge the sample outlet for approximately two minutes, or for a sufficient length of time to ensure fresh water will be collected. Purge sufficiently to remove all traces of chlorine if chlorine bleach was used in sterilization. Chlorine can bias samples low if concentration is sufficient to kill bacteria present in the sample. Record exact sterilization and purge procedures applied to each sample outlet in field notes.
4. Reduce the flow of water to a moderate stream, about the size of pencil, when possible. Put on a new pair of latex gloves.
5. Open the sterile sample container for bacteria analysis. The container may contain a preservative – do not rinse container.
6. Fill to the mark indicated on the container.
7. Cap container immediately, affix sample label, and place in cooler with ice to obtain appropriate cooling temperature specified by parameter preservation requirements.
8. Re-assemble any parts removed from sample outlet.
9. Log samples on chain-of-custody and submit to receiving laboratory within the appropriate holding time.
10. The client is informed of bacteria test results as soon as results are available; results are typically available the next day.

5.3.2 Collection from Groundwater Wells

Collect sample using equipment and sampling rate specified in sampling plan.

5.4 Drinking Water Metals

A typical drinking water sampling project includes sources of drinking water within a facility (e.g., drinking fountains, cafeterias, and sinks). A sample from the facility's incoming water near its point of entry is also typically taken. It is recommended the source is purged a minimum of 3-5 minutes the night prior to sampling. Then the water line should remain undisturbed for 6-8 hours but no more than 18 hours preceding sample collection. The primary metals of concern are lead and copper.

Note: Sample containers used in the field collection of drinking water are not pre-preserved with nitric acid (HNO_3) due to safety and health concerns associated with possible splashing and contamination at a drinking water source. The receiving laboratory will follow established protocols for sample preservation upon receipt of samples. Provide relevant comments on chain-of-custody submitted with samples.

1. Identify and confirm all sampling points with the site contact.
2. Label sample bottle using a description that accurately identifies the location for future reference (e.g., nearest room number, first floor lobby).

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3. Record observations including, but not limited to, appearance of source, leaks, filtration systems.
4. Do not purge. Collect the "first-draw" of water into a sample bottle:
   a. For drinking fountains, collect 250 mL at first flush. Fill to the neck of the bottle. The low collection volume is to ensure the sample represents the drinking fountain and not the incoming water source.
   b. For faucets and taps, collect 1000 mL at first flush. Fill to the neck of the bottle.
8. Log sample on chain-of-custody.
9. When all scheduled drinking water sources have been sampled, locate a sample point closest to where water enters the facility.
10. Purge line for 2 –5 minutes. Label bottle, collect sample, and log on chain-of-custody.

5.5 Perfluorochemical (PFC) Grabs

Perfluorochemical analysis is performed at very low detection levels. Extreme care must be given to avoid contamination. Do not allow sampling equipment or sample containers to come in contact with dirty surfaces. Nitrile gloves must be worn when handling sample equipment and bottles. A new pair of gloves is worn at each sampling location and gloves are changed after contact with possible contaminants.

The chemicals of interest are frequently used in the preparation of prepackaged food and fast food. Avoid the handling of these items prior to and during equipment preparation, deployment, and retrieval.

Typically, the receiving laboratory provides prepared sample bottles, pre-spiked sample bottles, pre-filled trip blanks, and trip blank spikes. Trip blanks accompany sample bottles at all times. When applicable, sample bottles are filled in this order:

1. Sample
2. Sample duplicate
3. Sample low spike
4. Sample high spike

Fill bottles to appropriate fill line on bottle if one is provided. If sample bottles are not provided, bottles made from high-density polyethylene, or equivalent, are prepared according to container cleaning procedures in Section 3.4.2.

IMPORTANT – Teflon® must not be used as a component in any equipment coming in contact with the sample or equipment blank water. Check fittings on DI water source to make certain Teflon® is not used. Grundfos submersible pump should not be used for PFC sampling due to Teflon® ports.

If sample tubing is required for sample collection, use new Tygon® sample tubing or equivalent for each sampling event. Prepare according to tubing cleaning procedures in Section 3.4.2.

5.6 Low Level Mercury: EPA Method 1669

For additional information, refer to *EPA Method 1669 Sampling Ambient Water for Determination of Trace Metals at EPA Water Quality Criteria Levels.*

A common low-level mercury sampling method is Clean Hands/Dirty Hands. This requires a two-person team. One member is designated as Dirty Hands (DH) and is responsible for all activities that do not involve direct contact with sample. The second member is designated as Clean Hands (CH) and will handle all activities involving contact with the sample bottle and sample. CH must be very

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cautious to avoid contact with any other material.

Both team members wear non-particle shedding clothing. Avoid dust and dirt as much as possible. A new pair of powder free gloves made from latex, nitrile, or vinyl are worn for each sampling location. Team members wear multiple layers of gloves so outer layer can be easily removed when contamination is suspected.

The need to avoid contamination during transport and collection is extremely important. Potential sources of mercury contamination include metal equipment, reagents, DI water, improperly cleaned equipment, exhaust, dirt, and cigarette smoke. To prevent contamination:

- All equipment contacting sample must be free of metal and cleaned according to EPA Method 1631.
- Sample bottles must be made of fluoropolymer or glass.
- Stored and transported samples in double zip-seal plastic bags.
- Avoid breathing on sample.
- Shoulder length gloves are worn when direct immersion of sample containers occur.
- Do not perform sampling on windy or rainy days.

Discuss equipment needs, preservation, collection, and shipping with analytical laboratory to ensure current sampling requirements will be met. Pace FSD usually obtains necessary equipment and sample bottles cleaned and prepared according to EPA Method 1631 from receiving laboratory.

At a minimum, one field blank is required for each set of samples collected at a given site or one field blank per each set of 10 samples collected. When sampling multiple locations, always sample from the area of lowest expected concentration to the area of highest expected concentration. CH sampler should be down wind and downstream of sample point and avoid stirring up particulate matter in the water during the collection process.

Site Preparation

1. Once on-site, equipment is placed on clean plastic sheeting in close proximity to sampling point.
2. Designate team members as DH or CH.
3. DH opens bag containing gloves and CH removes gloves and puts multiple layers on.
4. CH removes gloves for DH to put on multiple layers.

Field Blank Collection:

1. DH opens cooler containing pre-cleaned, double bagged sample bottles, trace-metal grade DI water for field blank, and any other pre-cleaned components of the sampling apparatus.
2. DH opens outer bag of empty pre-labeled container for field blank.
3. CH opens inner bag, removes bottle, and folds inner bag over to limit atmospheric exposure.
4. DH closes outer bag and returns to cooler.
5. DH opens outer bag containing bottle of trace-metal grade DI water.
6. CH opens inner bag and removes bottle.
7. CH transfers trace-metal grade DI water to empty field blank sample bottle and caps both bottles.
8. DH opens outer bags and CH returns bottles to respective bags.
9. DH returns field blank to cooler.

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Grab Sample Collection:

1. DH selects new sample container and opens outer bag.
2. CH opens inner bag, removes bottle, and folds down inner bag to limit atmospheric exposure. DH closes outer bag.
3. CH removes cap and holds cap so that inner part is against hand. With bottle hand, CH fills sample bottle by dipping into water keeping hand downstream of bottle opening. CH immediately caps bottle when filled.
4. DH opens outer bag and CH places bottle in inner bag. CH closes inner bag and DH closes outer bag and places in cooler.
5. Repeat steps for additional samples.
6. DH will complete required paperwork. Chain-of-custody is placed in clean zip-seal plastic bag before placing in cooler.

NOTE: Samples do not need preservation or cooling during transport to receiving laboratory providing preservation or analysis is performed within 48 hours of collection.

6.0 FIELD ANALYSES TEST PROCEDURES

Pace FSD uses a variety of testing meters capable of analyzing a given parameter. Calibration, maintenance, and operation of specific field instruments are in accordance with the respective manufacturer specifications. Allow probes to equilibrate to sample temperature prior to taking measurements. When possible, temperature dependent parameters are temperature compensated. Minimize exposure to atmosphere, direct sunlight, and time between sample collection and sample analysis. A test sample must be of sufficient volume to insure a thermal mass large enough to prevent significant changes in temperature before measurement. The use of a flow cell is recommended when applicable to project objectives and procedures.

Field parameter instruments undergo calibration procedures before use. Guide 34 Certified Reference Materials are used for calibration when available. For most parameters, a multi-step calibration procedure is performed; refer to parameter-specific sections for detailed instructions.

1) First, an instrument undergoes **Initial Calibration (IC)**. Initial Calibration is the process of analyzing standards, prepared at specified concentrations to define the quantitative response relationship of the instrument to the analytes of interest.
2) Then in most cases, Initial Calibrations are then verified with an **Initial Calibration Verification standard (ICV)** obtained from a second manufacturer (i.e., second source buffer) or from a second lot from the same manufacturer (if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples). Sample results are quantitated from the Initial Calibration unless otherwise required by regulation or method.
3) During the course of analysis, instrument calibration is periodically verified by the analysis of **Continuing Calibration Verification (CCV)** standards, unless otherwise specified in project documentation or instrument procedures. It is recommended to perform a CCV after the measurement of every 10 samples, at a minimum; if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times. Unless otherwise stated, CCV is performed using standards(s) closest to and bracketing the range of measured values (either calibration standard(s) or second source standard(s) when available).

   Record field parameter calibrations and verifications on appropriate data sheets; refer to Attachment 14.

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6.1 Temperature

Pace FSD conducts temperature measurements based on Standard Methods 2550-B. Project situational deviations are fully documented at the time of testing. Certified thermometers are traceable to national standards maintained by NIST for initial calibration and are re-certified annually by Pace FSD. All other field thermometers, and applicable instrument temperature sensors, are compared to a certified thermometer annually. Thermometers are labeled with correction factor as needed. Specific projects may require additional temperature accuracy checks before field measurements. Temperature measurements are taken in-situ when possible. If measuring temperature from a container, the following conditions are met:

- The container must be large enough to allow full immersion of thermometer probe and be of sufficient volume to ensure a thermal mass large enough to prevent significant temperature changes before measurement.
- The thermometer must be placed in the container immediately to avoid biased temperature changes.
- The container must be protected from direct sunlight and strong winds during the measurement procedure.

Measurement Procedure

1. Physically remove any visible contamination from the temperature probe and immerse into sample.
2. Suspend thermometer away from the sides and bottom of the channel, well casing, or container, and agitate temperature probe in the sample.
3. Allow reading to stabilize for at least one minute.
4. Record temperature to the nearest 0.5°C, unless otherwise specified. *Note*: pH temperature measurements are recorded to the nearest 1°C.

6.2 pH

Pace FSD conducts electrometric pH measurements following procedures outlined below; these procedures based on Standard Methods 4500-H+ B. Project situational deviations are fully documented at the time of testing.

An accurate pH reading is dependent on all components of a pH measurement system working properly. Proper storage, cleaning, and calibration of probes are important to system correctness. Store probes in proper storage solution for time periods exceeding 24 hours. Do not store pH probes in DI water or leave exposed to the air. Probes can be stored in pH 7 buffer for time periods less than 24 hours. If a probe is accidentally stored dry, immerse probe in the long-term storage solution for a minimum of 8 hours.

To clean contaminated glass bulb probes, soak in a light detergent solution, or for severe contamination, soak for 30 seconds in a 0.1 M HCl solution, followed by a thorough light-detergent rinse. Take special care to protect the pH probe.

Since temperature differences can influence pH readings, pH instruments used by Pace FSD utilize automatic or manual temperature compensators. For meters with Automatic Temperature Compensation (ATC), a compatible ATC probe must be used. ATC meters receive signals from a temperature element automatically correcting the pH value based on the temperature of the solution. The meter displays temperature corrected pH results. Using an ATC system prevents measurement fluctuations due to temperature and eliminates need for successive measurements of one sample.

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pH meters are calibrated with buffer solutions that bracket the expected range of measurement values. Typically, a two-point calibration is performed using a 7 buffer solution and a second buffer solution of either 4 or 10 depending on the expected pH range of the sample. A three-point calibration is performed in certain situations. If the instrument is capable, perform a three-point calibration when the expected pH range is unknown. pH calibration values are recorded to the nearest hundredth (0.01) and temperature values to the nearest degree.

Repeat adjustments on successive portions of the two buffer solutions as outlined in Section 8.2 until readings are within 0.05 pH units of the buffer solution value.

Pace FSD uses commercially manufactured buffers that are certified, color-coded and traceable to national standards maintained by NIST. Buffer tolerances per manufacturer are: ± 0.01 for pH 4 and 7 buffers, ± 0.02 for pH 10 buffers @25°C. Calibration is confirmed by a pH meter's acceptable slope display. Acceptable slope range is between 92-102%. Generally, slopes below 92% indicate the electrode requires cleaning or replacement, and slopes above 102% indicate the pH buffers are contaminated. If the mV values are outside this range, manually calibrate or perform maintenance/replacement of electrode. The pH electrode is working properly if the slope is between 92-102%.

After Initial Calibration (IC), an Initial Calibration Verification (ICV) using a buffer from a second source is measured and recorded to verify the meter is working properly. Unless otherwise specified, a Continuing Calibration Verification (CCV) is performed, at a minimum, after measurement of every 10 samples. If fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure instrument's accuracy within sample hold time. Unless otherwise stated, CCV is performed using buffer(s) (either calibration buffer(s) or secondary source buffer(s)) closest to and bracketing the range of measured values. For use in single point measurements, calibration is acceptable and meter operation is verified if the ICV and CCV are within one tenth (± 0.1) of the respective buffer's value. For continuous pH, the calibration range (i.e., two or three points) is checked to verify meter operation with calibration acceptable if the ICV and CCV are within one tenth (± 0.1 s.u.) and (± 0.5) s.u., respectively.

Samples should be analyzed as soon as possible preferably in the field at the time of sampling. Portable pH meters may be immersed directly in the sample stream or a sample portion may be collected in a separate glass or plastic container. For all measurements, the probe shall be sufficiently agitated to insure sample movement across probe, indicated by a steady reading on the instrument display.

During sample measurement, record pH values to the nearest tenth (0.1) and temperatures to the nearest 1°C. Continuing calibration measurements confirm instrument measurement stability.

6.2.1 Hand-Held and Bench Meters

Thermo Orion meters, both Hand-Held and Bench, used by Pace FSD, store the slope of the calibration curve in the meter which is used in the calculation of pH. Calibration is retained in meter memory as long as power is sustained. Manual or auto shut off does not affect calibration. Calibration information is lost when battery or line cord is removed.

6.2.1.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on meter and go to calibration mode; refer to specific meter instruction manual for details.
2. Rinse probe with DI water, shake off excess water or wipe electrode if necessary, and place probe into first buffer solution when indicated by meter.
3. Gently agitate probe and allow sufficient time for meter stabilization. When a stable

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value is displayed, press [Yes] to accept the value if within acceptable range. Acceptable pH/temperature values are found on Attachment 12, Quick Reference Table. Press [No] to decline value and use arrow keys to adjust to appropriate value.

4. Record pH calibration values to the nearest hundredths place (0.01) and temperature values to the nearest degree. Record date and time of measurements.
5. Repeat Steps 2-4 for second buffer solution when indicated by meter.
6. Repeat Steps 2-4 for third buffer solution, if applicable.
7. After the meter is calibrated, perform an Initial Calibration Verification (ICV) using a second source buffer:
    a. Return meter to measurement mode and place probe into second source buffer.
    b. Measure the value of the buffer to verify the calibration. If the value displayed is within one tenth (± 0.1) of the buffer value, the calibration is accepted and the meter is ready for use. If outside this criterion, re-calibrate the meter.
    c. Record secondary source buffer information, measured value, temperature, and date and time on the appropriate data sheet.

6.2.1.2 Measurement Procedure and Continuing Calibration Verification (CCV)

A. In-situ Procedure:

1. Immerse pH probe into a well-mixed portion of the waste stream. Gently agitate probe and wait until a stable reading is observed.
2. Record pH value, temperature, date and time of measurement, and technician initials on appropriate data sheet. Record pH value to the nearest tenth (± 0.1) and temperature to the nearest 1°C.
3. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
    a. Select appropriate CCV buffer:
        i. If the pH result of the sample(s) measured is outside the calibration range, use a buffer that brackets the measured result(s).
        ii. If the pH result of the sample(s) measured is within the calibration range, select calibration or secondary source standard buffers that are closest to the pH value(s) measured.
    b. Place probe into the buffer and gently agitate probe and wait until a stable reading is observed. Record the value, temperature, and buffer information on the appropriate data sheet.
    c. Correct meter operation is verified if the CCV is within one tenth (± 0.1) of the buffer value. If the CCV is outside this criterion, re-calibrate the system and re-analyze the sample. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified in the final report.

B. Container Procedure:

1. Collect sample in a container large enough to allow full immersion of probe and be of sufficient volume to ensure a thermal mass great enough to prevent significant temperature changes before measurement. Protect the container from direct sunlight and winds during measurement procedure.
2. Immediately immerse pH probe into the container to avoid bias due to temperature change. Gently agitate probe and wait until a stable reading is

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observed.

3. Record pH value, temperature, date and time of measurement, and technician initials on appropriate data sheet. Record pH value to the nearest tenth (± 0.1) and temperature to the nearest 1°C.
4. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. Select an appropriate CCV buffer:
      i. If the pH result of the sample(s) measured is outside the calibration range, select a buffer that brackets the measured result(s).
      ii. If the pH result of the sample(s) measured is within the calibration range, select calibration or second source buffers that are closest to the pH value(s) measured.
   b. Place probe into the buffer and gently agitate probe and wait until a stable reading is observed. Record the value, temperature, and buffer information on the appropriate data sheet.
   c. Correct meter operation is verified if the CCV is within one tenth (± 0.1) of the buffer's true value. If the CCV is out of this acceptance criteria, re-calibrate the meter and re-analyze the sample. If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified in the final report.

6.2.2 Analytical Measurements Continuous pH Recorder

This unit is capable of recording a continuous log of pH measurements by means of an inkless strip chart recorder. Refer to instrument manual for care and maintenance of Analytical Measurements Recorder pH; probe cleaning is the same as procedure stated in Section 6.2. An internal rechargeable lead acid battery powers the unit. Do not operate unit when voltage is below 5.5V. Do not leave battery in a discharged state for more than 8 hours. A discharged battery can be fully recharged by plugging the 110V power supply cord into the unit for 48 hours. A fully charged battery powers the unit for 10 to 15 days.

1. Prior to departure:
   a. Determine the probe lead length needed for the monitoring site by either referring to the Client Data Sheet/Project Specific Site Information Sheet, or consulting with project administrator.
   b. Check battery voltage.
   c. Obtain thermometer to set temperature of unit.
   d. Check calibration and ICV to make sure unit is working properly; this does not replace the required on-site calibration.
2. Onsite:
   a. Remove probe cap and thoroughly rinse probe with DI water.
   b. Using the thermometer, set 'Temperature' knob to the temperature of the buffer solution.
   c. Turn selector switch to 'Record'. Place probe into pH 7.0 buffer solution.
   d. Adjust calibration knob so recorder pointer strikes the 7 mark on the strip chart. Allow unit sufficient time to advance paper so a visible calibration mark is observed.
   e. Rinse probe with DI water, shake off excess water, and place in the 4 or 10 buffer solution depending on the expected pH range of the effluent. Allow unit sufficient time to advance paper so a visible calibration mark is observed.
   f. If the recorder pointer does not move exactly to the second buffer point, adjust the slope control. To adjust the slope control, swing the control unit out of the

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housing. A small screw adjustment is located on the top left when facing the front of the module. (Ignore the small screw to the right of the slope control; it applies to ATC probes which are not used with these meters.) Turn screw as necessary to match the pointer to the desired location. Allow unit sufficient time to produce a visible calibration mark.

g. After the meter is calibrated, perform an Initial Calibration Verification (ICV):
   i. Return meter to measurement mode.
   ii. Place probe into a second source buffer. Allow unit sufficient time to advance paper so a visible calibration mark is observed. If the value displayed is within one tenth (± 0.1) of the buffer value, the calibration is accepted and the meter is ready for use.
   iii. Record second source buffer information, measured value, temperature, and date and time on an appropriate data sheet.

h. Circle and label the three points. Record the following information directly on the strip chart paper:
   i. Client Name
   ii. Site Location
   iii. Technician's Initials
   iv. Date
   v. Start Time

i. Using the thermometer, measure the temperature of the effluent and set the 'Temperature' dial to the corresponding temperature.

j. Advance strip chart to the correct time and secure probe in a well-mixed region of the waste stream.

3. Take-down
   a. Perform a Continuing Calibration Verification (CCV) using pH buffers estimated to bracket the expected range of results, i.e., a high and low value.
      i. Rinse probe with DI water, shake off excess water, and place into first CCV buffer. Allow unit sufficient time to advance paper so a visible calibration mark is observed.
      ii. Repeat above with second buffer value.
   b. Correct meter operation is verified if CCV is within one tenth (± 0.1) of the buffer value. Circle and label the CCV measurements; initial and record the date and time.
      i. If the CCV is outside this criteria:
         1) If continuous pH measurement is conducted in conjunction with automatic hourly sample collection, perform hourly pH measurements on collected samples.
         2) If unable to perform hourly pH measurements on collected samples, the measured value(s) must be appropriately qualified in the final report.
   c. For events longer than 24 hours in duration, the unit is to be periodically checked with buffer solutions to ensure measurement accuracy.
4. Upon return to Pace, decontaminate probe and return meter to proper storage area.

6.2.3 YSI Sonde Water Monitoring Instruments

Pace FSD commonly uses YSI Sonde Water Monitoring Instruments for continuous pH monitoring of effluent streams. The effluent waste stream must have sufficient depth (approximately 2 inches) to immerse the sensor.

NOTE: All sondes must be calibrated in the 7.00 buffer FIRST, because the sonde creates a calibration curve based on the 7.00 buffer.

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6.2.3.1 YSI 600XL Sonde / 650 MDS

The YSI 650 Multiparameter Display System (650 MDS) is a hand-held instrument used in conjunction with a 6-series sonde to set measurement parameters, display readings, store and recall data, and transfer data to a computer for analysis. Use the arrow keys to select options from the 650 MDS display and press [ENTER] to select options from the menu. Press [ESCAPE] to return to the previous screen. To run the sonde in real-time, select 'Sonde Run' from the menu or active the 'Run/Discrete Samples' option. In the 'Sonde Run' mode, data automatically displays in one-second intervals. Choose 'Run/Discrete Samples' option to set the display interval higher.

1. Turn on meter and select ISE1 pH from the calibration menu.
2. Select 2-Point or 3-Point, depending on expected pH range, and press [Enter].
3. Rinse probe with DI water, dry with lint-free tissue, and immerse probe into 7.00 buffer. (Must calibrate in 7.00 buffer FIRST).
4. Allow approximately one minute for temperature equilibration. Press [Enter].
5. This probe is not temperature compensated. Use the pH/temperature relationship chart to enter the value of the 7.00 buffer solution at the corresponding temperature value and press [Enter]. Refer to Quick Reference Table (Attachment 12).
6. The current value will display. Gently agitate probe and observe pH reading. Press [Enter] to accept value when no significant change occurs for approximately 30 seconds.
7. Press [Enter] again.
8. Rinse sonde probe with DI water and dry with lint-free tissue.
9. Immerse probe into second buffer solution (either 4.00 or 10.00) and allow approximately one minute for temperature equilibration. Press [Enter].
10. Enter the temperature corrected value of the second pH buffer using the pH/temperature relationship chart. (Refer to Attachment 12, Quick Reference Table.)
11. Press [Enter] to display current value.
12. Press [Enter] to accept value when no significant change occurs for approximately 30 seconds.
13. Press [Enter] again and repeat Steps 8-12 for third buffer, if applicable.
14. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a second source buffer:
    a. Return sonde to measurement mode and immerse probe into second source buffer. The current value will display.
    b. Gently agitate probe and allow approximately one minute for temperature equilibration.
    c. Record second source buffer information, measured value, temperature, and date and time on an appropriate data sheet. If the reading is within one tenth (± 0.1) of the buffer value, the calibration is accepted and the meter is ready for use. If outside this criterion, re-calibrate the instrument.
15. Use calibrated meter to measure sample; refer to Section 6.2.1.2 for measurement procedures.

6.2.3.2 YSI 600XL Sonde

The YSI 600XL Sonde is a multi-parameter water quality measurement and data collection device that requires an external power supply. It has multiple interchangeable probes to measure and log a variety of water quality data including, but not limited to, time, temperature, conductivity, pH, DO, turbidity, and a depth-sensor for use in lakes, rivers and ground water wells. The YSI Sonde is programmable for a range of parameters using EcoWatch software or a hand-held control instrument.

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The basic steps in conducting a routine continuous pH study follow. ***Note:*** For additional information refer to *YSI Environmental Operation Manual.*

I. Programming/Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Connect external power source to YSI sonde. Pace FSD uses a custom made connection cable to connect unit to battery.
2. Connect sonde to computer using the amphenol to USB converter cable.
3. Double click the EcoWatch icon to open software on computer.
4. Click on the sonde icon or select "Sonde" from the "Comm" menu button.
5. Select the proper comm port.

a. Select "Other" and "Com5"
b. [OK]

6. The computer screen will display the "#" prompt. Type "menu" and press [Enter].
7. Select "1-Run" by pressing [1]. *Note*: The run or logging step is initiated at this time to obtain an electronic record of the initial calibration points.
8. Select "2-unattended sample" by pressing [2].
9. Define the following parameters:

    a. Interval *(1 minute for most applications)*
    b. Start Date *(current date)*
    c. Start time *(current time)*
    d. Duration *(event duration)*
    e. File Name *(unique file name)*
    f. Site *(can be left blank)*
    g. Free Memory #days *(confirm adequate memory for project duration)*
    h. View Parameters to Log *(confirm pH is selected)*

10. Under the same menu in Step 9, select "A-start logging," followed by selecting "1-start logging." *Note*: menu option 9 will now display "stop logging."
11. Continue selecting menu option "0" by pressing [0] until the main starting menu from Step 7 above is displayed.
12. Select "2-Calibrate" by pressing [2].
13. Select "pH" by pressing corresponding number. *Note*: The option number will vary depending on the number of parameters listed under this menu heading.
14. Select "2-point" by pressing [2] or "3-point by pressing [3].
15. Remove the sensor storage cap, rinse sensor with DI water, shake excess water from sensor or dry with lint-free tissue, and place sensor into 7.00 buffer solution. (Must calibrate in the 7.00 buffer FIRST.).
16. Use the pH/temperature relationship chart to enter the value of the 7.00 buffer solution at the corresponding temperature value and press [Enter]. Refer to the Quick Reference Table (Attachment 12. When reading is stable, press [Enter].
17. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with a lint-free tissue.
18. Place sensor in second buffer solution that will bracket the expected pH range of sample to be measured. Enter the exact value of the second pH buffer at the corresponding temperature value and press [Enter]. When reading is stable, press [Enter].
19. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with a lint-free tissue.
20. If calibrating with a third buffer, place sensor in third buffer solution. Enter the exact value of the third pH buffer at the corresponding temperature value (Refer to Attachment 12, Quick Reference Table.) and press [Enter]. When reading is stable, press [Enter].

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21. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with a lint-free tissue.
22. Press [0] until back at the main menu.
23. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a second source buffer:
    a. Enter measurement mode and immerse probe into second source buffer.
    b. Gently agitate probe and observe pH reading. Record the value, temperature, and buffer information on the appropriate data sheet when no significant change is observed.
    c. If the reading is within one tenth (± 0.1) of the buffer value, the calibration is accepted and the meter is ready for use. If outside this criterion, re-calibrate the instrument.
24. Exit the software program.
25. Screw the storage cap, filled with approximately 1/2 inch of tap water or a moist sponge in the transport/calibration cup, to the sonde for short term storage.
26. Cover amphenol computer connection of sonde with plastic bag or latex glove and seal around cable with tape or twist-tie.

II. Onsite Deployment/Retrieval

1. Before deployment in the waste stream, conduct a performance verification:
    a. Place sonde in the 7 buffer for a length of time sufficient to log 2-4 points. This length of time depends on the sample interval programmed during setup.
    b. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue.
    c. Place sonde in second buffer (4 or 10) for a length of time sufficient to log 2-4 points. This time depends on the sample interval programmed during setup.
    d. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue.
    e. If a 3-point calibration was performed, place sonde in third buffer for a length of time sufficient to log 2-4 points. This time depends on the sample interval programmed during setup.
    f. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue.
2. Tightly secure protective strainer cap. Immerse probe and secure sonde into well-mixed region of waste stream.
3. Secure external battery and excess cable out of the way of other equipment.
4. Allow sonde to collect data for the duration of the project. For events longer than 24 hours in duration, the unit is to be periodically checked with buffer solutions to ensure accurate measurements are obtained.
   NOTE: For projects of relatively long duration in waste streams with high solids or oily discharges, it is recommended to perform calibration onsite using field laptop until it is verified the waste stream does not diminish sonde response.
5. At the end of the project duration, remove sonde from waste stream and rinse probe with DI water. Shake excess water from sensor or dry with lint-free tissue.
6. Perform a Continuing Calibration Verification (CCV) using pH buffers estimated to bracket the expected range of results, i.e., a high and low value.
    a. With the meter in measurement mode, place sensor into the first buffer for a length of time sufficient to log 2-4 points. This length of time depends on the sample interval programmed during setup.
    b. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue.

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c. Place sonde in second buffer for a length of time sufficient to log 2-4 points. This time depends on the sample interval programmed during setup.
d. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue.
e. If a 3-point calibration was performed, place sonde in third buffer for a length of time sufficient to log 2-4 points. This time depends on the sample interval programmed during setup.

7. Thoroughly rinse sensor with DI water and shake excess water from sensor or dry with lint-free tissue. Screw the storage cap, filled with approximately 1/2 inch of tap water or a moist sponge in the transport/calibration cup, to the sonde for short term storage.

III. Retrieving Data

1. Connect sonde to field computer using the amphenol to USB converter cable.
2. Double click the EcoWatch icon to open software on computer.
3. Click on the sonde icon or select "sonde" from the "Comm" menu button.
4. Select the proper Comm (Communications) Port:
a. Select *Other* and *Com5*
b. [OK]
5. The computer screen will display the "#" prompt. Type "menu" and press [Enter].
6. Select: *1-Run*
7. Select: *2-Unattended Sample*
8. Select: *9-Stop Logging*
9. Press [0] until you have returned to the main menu.
10. Select: *3-File*
11. Select: *2-Upload*
12. Select the file name of the desired data to retrieve.
13. Enter the report "Start" and "Stop" date and time; include initial calibration times.
14. Select: *1-Proceed*
15. Select: *2-Comma & "" "" Delimited*
16. The data file is then transferred from the sonde memory to the computer. "File Transfer" window appears. The data is automatically saved to "C:\Ecowin\data" on the field computer.
17. Continue pressing [0] to exit Ecowatch software.
18. Open the C:\Ecowin\data folder.
19. Open the text document with the data of interest, i.e., the file just transferred.
20. Insert USB memory stick into USB port on computer.
21. Select "File – Save As" from File menu and in the "Save In" prompt at the top of the screen, select removable Disk "E" (the memory stick).
22. Type the file name as "unique name.xls" and click "Save".
23. Close Notepad file.
24. Open "Excel" and select "File Open" from the File menu.
25. Open the "Disk E" (the memory stick) and double click the file saved in Step 22 above.
26. The "Text Import Wizard" opens:
    a. Select: "Next"
    b. Click "Comma" and "Tab" then "Next"
    c. Click "Finish"
27. Save the data as formatted or copy and paste the data into the "Continuous pH Reporting Workbook".
28. Review the data paying attention to the CCV results. If the CCV is out of the stated acceptance criteria,

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a. If continuous pH measurement is conducted in conjunction with automatic hourly sample collection, perform hourly pH measurements on collected samples.
b. If unable to perform hourly pH measurements on collected samples, the measured value(s) must be appropriately qualified in the final report.

6.2.3.3 YSI 556 MPS

For additional operational instructions, refer to Section 6.10.

I. Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Highlight the **pH** selection and press **[Enter]** to display the pH calibration screen.
2. Highlight a 1-point, 2-point, or 3-point calibration.
   a. Select the **1-point** option if adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, adjust the calibration by carrying out a one point calibration.
   b. Select the **2-point** option to calibrate the pH sensor using two calibration standards. Use this option if the media being monitored is known to be either basic or acidic.
   c. Select the **3-point** option to calibrate the pH sensor using three calibration standards. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated.
3. Press **[Enter]** to display the pH Entry Screen.
4. Ensure the sensor is as dry as possible. Rinse the pH sensor with a small amount of buffer that can be discarded.
5. Place the correct amount of 7.00 pH buffer into a clean, dry or pre-rinsed transport/calibration cup and screw the transport/calibration cup on the probe and securely tighten. Do not over-tighten as this could cause damage to the threaded portions. Gently agitate to remove any bubbles from the pH sensor.
6. Use the pH/temperature relationship chart (Refer to Attachment 12, Quick Reference Table) to determine the value of the 7.00 buffer solution at the corresponding temperature value. Enter this value using the keypad, then press [Enter] to display the pH calibration screen. Current values of all enabled sensors appear on the screen and change as they stabilize.
7. Watch the reading under pH and when the reading shows no significant change for approximately 30 seconds, press **[Enter]** to accept the calibration.
8. Press **[Enter]** to return to the Specified pH Calibration Screen.
9. Rinse the probe module, transport/calibration cup, and sensors in tap or purified water and dry.
10. If necessary, repeat steps using a second and third pH buffer.
11. Press **[Enter]** to return to the pH Calibration Screen.
12. Press **[Escape]** to return to the calibrate menu.
13. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a second source buffer:
    a. Enter measurement mode by selecting 'Run' from the main menu.
    b. Immerse sensor into second source buffer and observe pH reading. When no significant change is observed, record pH value, temperature, and buffer information on appropriate data sheet.
    c. If ICV result is within one tenth (± 0.1) of the true value of the standard, the calibration is accepted and the meter is ready for use. If outside this criterion, recalibrate the instrument.

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II. Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using On/Off key.
3. Display run screen by selecting Run from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
5. Watch the readings on the display until they are stable.
6. Record pH, temperature, and date and time of reading.
7. Rinse probe and perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. Select appropriate CCV buffer:
      i. If the pH result of the sample(s) measured is outside the calibration range, select a buffer value that brackets the measured result(s).
      ii. If the pH result of the sample(s) measured is within the calibration range, select a calibration or second source buffer that is closest to the pH value(s) measured.
   b. Place the probe and gently agitate probe and wait until a stable reading is observed. Record the value, temperature, and buffer information on the appropriate data sheet.
   c. Correct meter operation is verified if the CCV is within one tenth (± 0.1) of the buffer value. If the CCV is outside this criterion, recalibrate the system and re-analyze the sample. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified in the final report.
8. Rinse probe, and turn off instrument.
9. Refer to Section 6.10 for instructions on logging sample data.

6.2.4 YSI Sonde Troubleshooting

If trouble calibrating a sonde, go through the reset procedure to fix the issue. This procedure works for pH, conductivity, and DO.

1. When in the calibration menu, select the parameter having issue, i.e., pH, Conductivity, and Dissolved Oxygen.
2. When prompted to input a value (i.e., buffer, conductivity solution, barometric pressure), enter "uncal" instead to reset the unit to factory values. The sonde automatically returns to the calibration menu.
3. Proceed to calibrate as normal.

6.2.5 Hourly pH

For hourly pH analysis, a pH measurement is taken on the sample collected in each bottle of a sampler base representing an hour of facility discharge. Hourly pH measurements are performed on take-down day.

Procedure

1. Calibrate pH meter according to procedures in Section 6.2.1.
2. Sufficiently mix sample in bottle to be measured.
3. Gently agitate probe in sample and allow sufficient time for meter stabilization.

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4. Record pH measurement and temperature in space corresponding to the time of sample collection on appropriate data sheet. Record the time the first and last bottle measurements are taken on the data sheet.
5. Repeat Steps 2-4 for remaining sample bottles.
6. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. Select appropriate CCV buffer:
      i. If the pH result of the sample(s) measured is outside the calibration range, select a buffer value that brackets the measured result(s).
      ii. If the pH result of the sample(s) measured is within the calibration range, select a calibration or second source buffer that is closest to the pH value(s) measured.
   b. Place probe sensor into the buffer and gently agitate probe and wait until a stable reading is observed. Record the value, temperature, and buffer information on the appropriate data sheet.
   c. Correct meter operation is verified if the CCV is within one tenth (± 0.1) of the buffer's value. If the CCV is outside this criterion, recalibrate the system and re-analyze the hourly samples. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified in the final report.
7. Notify project administrator of pH values out of compliance.

6.2.6 Sper Datalogging pH Meter

The Datalogging pH Meter is a handheld instrument for measuring either pH or mV together with temperature in °C or °F. The meter features automatic temperature compensation (ATC) or manual temperature compensation, five points of calibration, and automatic buffer solution recognition. The pH electrode probe is stored in a protective cap containing pH probe storage solution. The meter's pH electrode is always rinsed in de-ionized or tap water before and after storage. The meter software allows for up to 99 manual measurements and automatic logging of up to 4000 measurements. The meter uses 4 AAA batteries.

6.2.6.1 Set-up Meter Procedure

1. Turn on meter using the ON/OFF button to display the Welcome menu.
2. Press **F4** (SET) to enter setup mode.
3. Use the ▲ or ▼ buttons to move the on-screen cursor, **[F2]** (EDIT), and the **Keypad** to input changes.
   a. To change **LCD Contrast:** Display contrast from 1-5 (5 = least contrast).
   b. To change **Auto Off:** Set from 1~20 minutes, Enable/ Disable.
   c. To set **Clock:** Select the date mode MM-DD-YY, DD-MM-YY or YY-MM-DD and set local time.
   d. To set **ID:** Alpha/numeric user name, Enable/Disable datalogging.
   e. To **Select Item:** Press **[F2]** (EDIT) to select: X = not selected, √ = selected.
   f. To select from **3 units of measurement**: Press **[F2]** (EDIT), ▲ or ▼ buttons to cycle through the 3 units of measurement: pH, millivolt, and temperature.
   g. To select **temperature unit**: With the temperature item selected, press **[F2]** (EDIT) to switch between °C and °F.
4. Press **[F3]** (LOG) to view data-logs.
   a. "Expect" is the total number of memory points (4000).
   b. "Remain" shows the number of available data points (i.e., 4000 minus the number of already recorded data points).

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c. If the maximum number of data points is reached, logging stops with the records are held in the datalogger.

5. If changes are made, press **[F4]** (ENTER) to save the new settings or **[F1]** (ABORT) to cancel the change.
6. Press **[F4]** (NEXT) to access the next page.
7. Press **[F1]** (EXIT) to return to the Welcome menu.

6.2.6.2 Set-up Software Configuration

Use the RS-232 cable and software to download saved data to a PC for further analysis or for a faster datalogger parameter set-up process.

1. PC requirements
   a. Operating System: Windows98 or above.
   b. Hardware: Serial Port or compatible USB—RS232 Adapter.

2. Communications Port Settings
   a. Select the Com port and ensure the meter is communicating with the PC.
   b. The selected Com port is displayed in the bottom-left hand corner of the software screen.
   c. When connected, "PC Mode" and the Com port number (1-8) are displayed on the meter.

6.2.6.3 Calibration with ATC Probe

It is best to perform a calibration (1) when the probe has not been used for a long time, (2) when using the meter with a new probe for the first time, or (3) when the readings seem erratic.

NOTE: During calibration, pressing **[F2]** (RST) will cancel the process and restore the default setting. There are 5 points of calibration using USA or NIST pH buffers 1.68, 4, 7, 10, 12 and 12.45.

1. Start the calibration with pH buffer 7.00. Immerse probe into the buffer solution.
2. With the meter on, press **[F1]** (MEAS) to start the calibration process.
3. Press **[F3]** (CAL) for at least two seconds to enter the calibration mode.
4. "Auto Judging . . . " and the number of days since the last calibration is displayed. The meter is automatically determining the buffer solution value at this time. Press **[F4]** (ENTER) to continue.
5. If "XX days" is displayed, the clock has not been set (Refer to Section 6.2.6.1 for clock set-up).
6. The calibration point that best matches the buffer solution will display. To manually change the calibration point (1.68, 4, 7, 10, 12, or 12.45), use the ▲ or ▼ buttons.
7. Press **[F4]** (ENTER) again. As needed, use the ▲ or ▼ buttons to adjust the pH value to ±0.5, then press **[F4]** (ENTER).
8. If "Failure!!" is displayed, the range is not within 85~105%. If unable to calibrate, replace probe.
9. For multiple-point calibration, press **[F4]** (ENTER) and repeat the above steps to perform up to 5 points of calibration.
10. When finished, press **[F3]** (SAVE).
11. Confirmation is displayed, press **[F2]** (YES) to quit and save or **[F3]** (NO) to return to the previous menu.
12. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV):

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a. Enter measurement mode and immerse sensor into secondary source buffer.
b. Gently agitate probe and observe pH reading. When no significant change is observed, record pH value, temperature, and buffer information on the appropriate data sheet.
c. If the reading is within one tenth (± 0.1) of the buffer value, the calibration is accepted and the meter is ready for use.

13. Refer to Section 6.2.6.4, Step I for measurement procedures.

6.2.6.4 Datalogging

I. Measurement Modes

The meter has three measurement modes: Single, Multiple, Auto

1. **F1** (MEAS) Single Measurement
   i. **Single Measurement with ATC**
      a. Immerse probe tip in the solution to be measured.
      b. Press **[F1]** (MEAS) and a single measurement is displayed.
      c. Press **[F1]** (EXIT) to return to the Welcome menu.
   ii. **Single Measurement without ATC**
      a. Immerse probe tip in the solution to be measured.
      b. Press **[F1]** (MEAS) and a single measurement is displayed.
      c. Press **[F2]** (TEMP) to change the temperature value. Use the Keypad, ▲ or ▼ buttons, and/or **F3** (±) to change the temperature value.
      d. Press **[F4]** (ENTER) to save the setting and return to the pH measurement.
      e. Press **[F1]** (EXIT) to return to the menu without making a change.
2. **F2** (MEM) Multiple Measurement
   This option allows manual recording of up to 99 data points with real time and editable file names.
      a. Press **[F2]** (MEM) to enter this function.
      b. Press **[F2]** (MEAS) and the first measurement is displayed.
      c. To save the data, press **[F4]** (SAVE).
      d. Use the Keypad, ▲ or ▼ buttons to select the next data point number.
      e. To take another measurement, repeat Steps c-e as needed.
      f. To edit the file name of a recorded data point, press **[F3]** (EDIT). Use the **Keypad** to edit the file name.
3. **F3** (LOG) Auto Logging
      a. Press **[F3]** (LOG) to enter this function.
      b. Press **[F3]** (SET) to setup datalogging parameters.
      c. Press **[F2]** (EDIT) and use the **Keypad** to edit the selected parameter. Press **[F4]** (ENTER) to accept each value.
      d. Use the ▲ or ▼ buttons to select "Begin" date, "Start" time, "End" date, "Suspend" time, or sampling "Rate".

NOTE: The date mode, MM-DD-YY, DD-MM-YY or YY-MM-DD, defaults to the initial setting. For 24 hour logging, set the Start time to the desired start time and then set the Suspend (end) time to 1 second less than 24 hours later (Example: If desired start time is 11:00:00, set the suspend time to 10:59:59). The sampling "Rate" is adjustable from 1 to 7200 Sec(s).

      e. Repeat Steps b-d as needed.
      f. Press **[F1]** (EXIT) to return to the Welcome menu.
      g. Press **[F2]** (START) to begin the logging function. Datalogging will automatically begin and end according to the set parameters. To set

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parameters use the auto logging function that automatically records (logs) up to 4000 measurements based on user-input.

4. To display recorded measurements, press **[F4]** (VIEW). To display real-time measurements, press **F2** (MEAS). Press **[F1]** (ESC) to escape view mode.
5. To quit recording, press **[F1]** (STOP).
6. To review the previous or next 100 data points press **[F4]** (NEXT), then press **[F1]** (P-PG) or **[F2]** (N-PG).
7. To continue recording, press **[F2]** (START). The previously recorded data points are not erased.

II. Downloading Data

1. Open the datalogger software on the computer.
2. Ensure the Com Port is set up as "Com5." (This is displayed in the lower left corner of the program window.) This can be changed in the "Port" menu.
3. Connect the transfer cable to the meter and laptop. Enter logger mode by selecting "Logger" from the pull-down menu.
4. Download data by either selecting "Download Logger Data" from the "Command" pull-down menu or by clicking the small folder button with "L" on it.
5. "PC MODE" will display on the meter, and the program will display a box tracking the downloading of data points.
6. Once completed, click "OK".
7. The program will automatically display the first 100 data points.
8. Save the data file by going to "Save As" in the "File" menu. Select the location for the file and create a name including the client, site, date, and day number, as applicable.
9. The data can now be saved to a flash drive and processed for submission to client.

**To begin another 24 hours of logging, first follow the logger data clearing instructions (See Section 6.2.6.4, Step III) and then the logging instructions. *Note:* the logger will not begin at your entered start time if it has already passed. For example, for an 11:00 am set up: if this start time has passed, set the logger to begin approximately one minute after the current time displayed: If the time displayed on the logger is 11:08, set the start time for 11:09:00, and the end time as 11:08:59 in order to avoid missing data or pushing back the start time by 15 minutes every day.

III. Clearing Data

1. While in the log menu, press **[F3]** (SET) and then **[F4]** (NEXT) to access the option to clear data.
2. To delete a single record, use the ▲ or ▼ buttons to move the on-screen cursor. Then, press and release **[F2]** (CLR) to delete the selected record.
3. The confirmation, "Clear ?" is displayed. Press **[F2]** (CLR) to delete the selected record from MEM Data.
4. To delete all records, press **[F2]** (CLR) for at least 2 seconds.
5. The confirmation, "Clear All?" is displayed. Press **[F2]** (CLR) to delete all records from MEM Data.
6. Press **[F4]** (BACK) to return to the previous menu and **[F1]** (EXIT) to return to the Welcome menu.

IV. Processing Data

1. Insert flash drive containing the pH logger data into the computer. The logger data will have been saved as a text file. Right-click the file and choose 'Open with' and then 'Microsoft Excel'.

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2. Highlight the first column by clicking on the 'A' over it.
3. Go to the 'Data' menu and select 'Text to Columns'.
4. Choose 'Delimited', then click 'Next.'
5. Select Tab, Semicolon, and Comma.
6. Click 'Finish'. Most of the data is now separated into columns. The next step is to remove any unnecessary data.
   a. Remove the mV readings and any text columns by right-clicking the letter over the column to delete and then selecting "Delete" from the drop-down menu.
   b. Do not delete the first column (A) containing the data point numbers (1, 2, 3, etc.). Select this column, right click, and choose 'Clear Contents'. This removes the data without removing the column.
   c. Now only four columns should remain: Column 'A' is empty, Column 'B' contains time and date information, Column 'C' contains pH data, and Column 'D' contains temperature in °C.
7. To separate Time and Date information:
   a. Select Column 'B'.
   b. Click the black border that appears around the outside of the selected cells and drag it to the left, placing these data points into Column 'A'.
   c. Select Column 'A'.
   d. In the 'Data' menu, choose 'Text to columns'.
   e. Select 'Fixed Width' and click 'Next'.
   f. Click 'Finished'. The time and date are now separated.
   g. Select Column 'A' again, right-click, and choose 'Format Cells'.
   h. Under the 'Number' tab, select 'Date', and scroll through the options. Select the following format: MM/DD/YYYY. Click 'OK'.
8. Pace FSD templates are formatted to display temperature data in Column 'C' and pH data in Column 'D'. When the data logger program downloads data, it reverses this. To correct the columns:
   a. Select Column 'D'.
   b. Click the black border that appears around the outside of the selected cells and drag it to the right, placing the temperature data points into Column 'E'.
   c. Select Column 'C'.
   d. Click and drag these data points to the right, so pH data goes to Column 'D'.
   e. Select Column 'E'.
   f. Click and drag these data points to the left, so temperature data goes to Column 'C'.
   g. The data is now ready to be placed into the appropriate Pace FSD template and graphed according to project requirements.

6.2.7 Hach Hydrolab Minisonde 5 (MS5)

Pace FSD uses Hach Hydrolab MS5 Water Monitoring Instrument for continuous pH monitoring of effluent streams. The effluent waste stream must have sufficient depth (approximately 2 inches) to immerse the sensor.

NOTE: All sondes must be calibrated in the 7.00 buffer FIRST, because the sonde creates a calibration curve based on the 7.00 buffer.

6.2.7.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

This procedure calibrates pH using a two-point or a three-point calibration. A pH standard between 6.8 and 7.2 is treated as the "zero" and all other values are treated as the "slope". After the sensors have been properly maintained, the sensors can be calibrated. Always

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allow sufficient time for thermal stabilization of the standards. To reduce the time for stabilization, try to keep all calibration standards and equipment stored at the same temperature before parameter calibration.

1. Attach the power and data cable to the Sonde; attach the 9-pin connector to the surveyor.
2. Turn on the Surveyor, and wait approximately 10 seconds for initialization.
3. Press **SETUP/CAL**. Press **CALIBRATION**. Press **SONDE**.
4. Prepare sensors for calibration by rinsing sensors with deionized water:
   a. Remove sensor guard and attach calibration cup.
   b. Unscrew and remove cap of calibration cup.
   c. Half-fill calibration cup with deionized water and place cap on calibration cup.
   d. Shake sonde in DI water to ensure sensors are free from contaminants. Discard DI water. Repeat several times if necessary.
   e. Place small amount of calibration solution into calibration cup and rinse sonde sensors. Discard calibration solution.
5. Use the **ARROW** keys to highlight pH and press **SELECT**. A calibration screen will display.
6. Pour the pH standard to within a centimeter of the top of the Calibration Cup.
7. Enter the units for pH.
8. Rinse the probe module, transport/calibration cup, and sensors in tap or purified water and dry.
9. Repeat, if necessary, for second and third calibration standards.
10. Press **DONE** to finish the calibration.
11. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a second source buffer:
    a. Enter measurement mode.
    b. Immerse sensor into second source buffer and observe pH reading. When no significant change is observed, record pH value, temperature, and buffer information on appropriate data sheet.
    c. If ICV result is within one tenth (± 0.1) of the true value of the standard, the calibration is accepted and the meter is ready for use. If outside this criterion, recalibrate the instrument.

6.2.7.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using On/Off key and display run screen.
3. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
4. Watch the readings on the display until they are stable.
5. Record pH, temperature, and date and time of reading.
6. Rinse probe and perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. Select appropriate CCV buffer:
      i. If the pH result of the sample(s) measured is outside the calibration range, select a buffer value that brackets the measured result(s).
      ii. If the pH result of the sample(s) measured is within the calibration range, select a calibration or second source buffer that is closest to the pH value(s) measured.
   b. Place the probe and gently agitate probe and wait until a stable reading is

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observed. Record the value, temperature, and buffer information on the appropriate data sheet.

c. Correct meter operation is verified if the CCV is within one tenth (± 0.1) of the buffer value. If the CCV is outside this criterion, recalibrate the system and re-analyze the sample. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified in the final report.

7. Rinse probe, and turn off instrument.

6.3 Conductivity

Pace FSD conducts conductivity measurements following procedures outlined below; these procedures follow EPA Method 120.1 or Standard Methods 2510-B. Project situational deviations are fully documented at the time of testing.

Conductivity is a measure of how well water conducts an electrical current. Conductivity is temperature dependent and values vary as much as 3% for every 1°C of temperature change. Conductivity is reported at a particular temperature to compensate for these variations and allow for more accurate comparison of measurements over time. Depending on the instrument model, measurements may or may not be temperature compensated. The term conductivity is used when values are not temperature compensated. **Specific conductance** is used when values are temperature compensated to 25°C. Inspect probe daily for chips in coating. Follow specific meter model instruction manual for care and maintenance. Conduct meter-specific calibration procedures before daily use.

Conductivity can be measured in the field or laboratory. If sample not analyzed within 24 hours of collection, sample should be filtered and stored at 4°C.

Conductivity is measured in micro siemens per centimeter (µS/cm) which can be converted to other units as needed: 1 µS/cm = 0.001 mS/cm = 0.000001 S/cm = 1 µmho/cm
Record conductivity measurements to the nearest 10 µS/cm.

6.3.1 YSI 600XL Sonde / 650 MDS

Note: Enter calibration standard value at µS/cm at 25 °C.

6.3.1.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on meter and allow several minutes for meter to warm-up.
2. Fill a clean, dry cup with conductivity standard.
3. Select conductivity from the calibration menu, then select specific conductance.
4. Calibration and verification is performed using two standards: a high standard is used to calibrate the instrument and a low standard is used to verify linearity.
   a. Enter the calibration value for the high standard at µS/cm at 25 °C.
   b. Immerse conductivity cell into the high standard, agitate probe, and press enter. Observe the reading. Compare the meter measurement with the chart value for the standard reference solution at the given temperature. When stable for approximately 30 seconds, accept value.
   c. Press [Enter] to return to calibrate menu.
   d. Record specific conductance calibration result.
5. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a low standard:
   a. Enter measurement mode and immerse conductivity cell into low standard.

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b. Gently agitate and observe reading. When no significant change is observed, record measured result and standard value on appropriate data sheet. The ICV result must be within ±5% of the standard or 10 µS/cm of the standard value, whichever is greater, in order to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, recalibrate the instrument.

6.3.1.2 Measurement Procedure and Continuing Calibration Verification (CCV)

2. To measure sample, immerse conductivity cell into the sample, agitate probe to ensure homogeneity of temperature and conductivity in the sample.
3. Observe reading; when stable for approximately 30 seconds, record the value on the appropriate data sheet.
4. It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times, using standard values closest to measured result(s):
   a. In measurement mode, immerse conductivity cell into the calibration standard that is closest to measured results: either the high or low standard.
   b. Agitate probe and wait until a stable reading is observed. Record value, temperature, and buffer information on appropriate data sheet.
   c. The measured result must be within ±5% or 10 µS/cm of the standard value, whichever is greater. If data does not meet this acceptance criteria, re-calibrate the meter and re-analyze sample(s). If unable to recalibrate and reanalyze, the measured result(s) must be appropriately qualified when reported.
5. Rinse probe with DI water and shake off excess water.

6.3.2 YSI 556 MPS Specific Conductance

For additional operational instructions, refer to Section 6.10.

6.3.2.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

Calibrating specific conductance automatically calibrates conductivity, and salinity.

1. Turn on meter using **On/Off** key.
2. Press **Escape** to display the main menu screen.
3. Select **Calibrate** and press **Enter**.
4. Highlight the **Conductivity** selection and press **Enter.** The Conductivity Calibration Selection Screen is displayed.
5. Highlight **Specific Conductance** and press **Enter**. The Specific Conductance calibration screen displays.
6. Calibration and verification is performed using two standards: a high standard is used to calibrate the instrument and a low standard is used to verify linearity.
7. Calibrate using high value standard:
   a. Ensure the sensor is as dry as possible. Rinse the conductivity sensor with a small amount of standard that can be discarded. Make certain there are no salt deposits around the oxygen and pH/ORP sensors which can interfere with readings.
   b. Place the conductivity standard into a clean, dry or pre-rinsed transport/calibration cup.
   c. Completely immerse sensor into the solution (i.e., liquid covers vent hole) and gently agitate probe to remove any bubbles from the conductivity cell.

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d. Screw transport/calibration cup onto the probe module and tighten. Do not over tighten as this could cause damage to the threaded portions.
e. Enter the calibration value for the high standard at µS/cm at 25 °C. Press **Enter** to display the Conductivity Calibration Screen where current values of all enabled sensors appear and change with time as they stabilize.
f. Allow at least one minute for temperature equilibration before proceeding.
g. Watch the reading under Specific Conductance. Compare the meter measurement with the chart value for the standard reference solution at the given temperature. When the reading shows no significant change for approximately 30 seconds, press **Enter** to accept the calibration.

8. Record calibration value and standard information on appropriate data sheet..
9. Press **Enter** again to return to the Conductivity Calibration Selection Screen.
10. Press **Escape** to return to the calibration menu.
11. Rinse probe and sensors in tap or purified water and dry.
12. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a low standard:
    a. Enter measurement mode and immerse sensor into low standard.
    b. Gently agitate and observe reading. When no significant change is observed, record measured result and standard value on appropriate data sheet. The ICV result must be within ±5% or 10 µS/cm of the standard value, whichever is greater, in order to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, recalibrate the instrument.

6.3.2.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using **On/Off** key.
3. Display run screen by selecting Run from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
5. Observe reading; when stable for approximately 30 seconds, record the value on the appropriate data sheet.
6. It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times, using standard values closest to measured result(s):
    a. In measurement mode, immerse conductivity cell into the calibration standard that is closest to the result(s) measured: high or low standard.
    b. Agitate probe and wait until a stable reading is observed. Record result, temperature, and buffer information on appropriate data sheet.
    c. The measured result must be within ±5% or 10 µS/cm of the standard value, whichever is greater. If data does not meet this acceptance criteria, re-calibrate the meter and re-analyze sample(s). If unable to recalibrate and reanalyze, the measured value(s) must be appropriately qualified when reported.
7. Rinse probe with DI water and shake off excess water.
8. Refer to Section 6.10 for instructions on logging sample data.

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6.3.2.3 Clearing Calibration

If calibration needs to be removed from conductivity probe:

1. Press Calibrate
2. Press Conductivity
3. Press Specific Conductance
4. Hold down enter key and press Esc key at the same time.
5. Display will show "Uncal?"
6. Select Yes to remove the current calibration.
7. Then recalibration probe.

6.3.3 Hach Hydrolab Minisonde 5 (MS5)

6.3.3.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

This procedure calibrates TDS, raw Conductivity, and Salinity. Specific conductance requires a two-point calibration. Calibrate the sensor to zero and then to the slope buffer.

1. Attach the power and data cable to the sonde. Attach the 9-pin connector to the Surveyor.
2. Turn on the Surveyor, and wait approximately 10 seconds for initialization.
3. Press SETUP/CAL. Press CALIBRATION. Press SONDE.
4. Prepare sensors for calibration by rinsing sensors with deionized water:
   a. Remove sensor guard and attach calibration cup.
   b. Unscrew and remove cap of calibration cup.
   c. Half-fill calibration cup with deionized water and place cap on calibration cup.
   d. Shake sonde in DI water to ensure sensors are free from contaminants. Discard DI water. Repeat several times if necessary.
   e. Place small amount of calibration solution into calibration cup and rinse sonde sensors. Discard calibration solution.
5. Use the **ARROW** keys to highlight specific conductance and press **SELECT**. A calibration screen will display.
6. Pour the specific conductance standard to within a centimeter of the top of the Calibration Cup.
7. Make sure there are no bubbles in the measurement cell of the specific conductance sensor.
8. Enter the SpCond standard for mS/cm or µS/cm.
9. Press **DONE** to finish the calibration.
10. Rinse probe and sensors in tap or purified water and dry.
11. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a low standard:
    a. Enter measurement mode and immerse sensor into low standard.
    b. Gently agitate and observe reading. When no significant change is observed, record measured result and standard value on appropriate data sheet. The ICV result must be within ±5% or 10 µS/cm of the standard value, whichever is greater, in order to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, recalibrate the instrument.

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6.3.3.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using **On/Off** key.
3. Display run screen by selecting Run from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
5. Observe reading; when stable for approximately 30 seconds, record the value on the appropriate data sheet.
6. It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times, using standard values closest to measured result(s):
   a. In measurement mode, immerse conductivity cell into the calibration standard that is closest to the result(s) measured: high or low standard.
   b. Agitate probe and wait until a stable reading is observed. Record result, temperature, and buffer information on appropriate data sheet.
   c. The measured result must be within ±5% or 10 µS/cm of the standard value, whichever is greater. If data does not meet this acceptance criteria, re-calibrate the meter and re-analyze sample(s). If unable to recalibrate and reanalyze, the measured value(s) must be appropriately qualified when reported.
7. Rinse probe with DI water and shake off excess water.
8. Refer to Section 6.10 for instructions on logging sample data.

6.4 Chlorine and Chloramines

Pace FSD conducts chlorine analysis following procedures outlined below; these procedures are based on Standard Methods 4500-CL G. Pace FSD Hach meters calculate and display results in mg/l rather than the Calibration Curve Method stated in 330.5. Project situational deviations are fully documented at the time of testing.

The volatile nature of chlorine in water means it may be found in a variety of forms.

Free chlorine is the presence of hypochlorus acid or hypochlorite ions in water.

Chloramines form when chlorine reacts with ammonia or nitrogen creating a more stable compound. Various chloramine compounds form depending on the pH of the water including monochloramine (NH_2Cl), dichloramine ($NHCl_2$), and trichloramine (NCl_3). Chloramines are also referred to as combined chlorine.

Total chlorine is the sum of both free chlorine and chloramines (combined chlorine) levels.

Pace FSD uses commercially prepared standards and reagents. The holding time for chorine testing is considered immediate. On-site testing of chlorine is necessary to assure the time between sample collection and analysis is minimal. Use round glass 10-mL cells for low range testing and plastic 1-cm/10-mL cells for high range testing. Use glass containers if collection or transfer device is needed. It is preferred that separate sample cells are used for free and total chlorine analysis as iodide from the total chlorine reagent can carry over and bias the free chlorine result.

To collect a representative sample, allow water to purge for at least one minute. Rinse sample cells several times with sample water before collecting the volume to be analyzed. To compensate for color and turbidity interference, a sample blank is used to zero the instrument. Thoroughly rinse

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sample cells after use with deionized water to remove any residue.

The following procedures are used with the Hach Pocket Colorimeter II Chlorine Meter with wavelength of 528 nm. Procedures 6.4.1 - 6.4.5 are adapted from Hach Pocket Colorimeter II Instruction Manual. Refer to manual for additional information. Record Calibration Verification and measurement data on appropriate data sheets; refer to Attachments within this manual.

NOTE: Temperature differences between the chlorine meter and the sample cell may cause water to condense on the meter lense resulting in biased readings. To reduce the possibility of this occurring, maintain the instrument in a heated portion of the vehicle in cool weather, and ensure the sample cell is wiped thoroughly to remove moisture before placing cell in meter in warm weather.

6.4.1 Switching Ranges

1. Press the MENU button to display "SEL" in the measurement screen. An arrow will indicate the current range.
2. Press the READ/ENTER button to switch between ranges.
3. Press MENU button to accept indicated range.

6.4.2 Initial Calibration Verification (ICV)

Hach Pocket Colorimeter II Chlorine Meters are calibrated during manufacturing. To ensure the instrument is working properly, use the Spec√ Secondary Standards to perform an Initial Calibration Verification (ICV) prior to field analysis and a Continuing Calibration Verification (CCV) after field analysis. It is suggested to perform one CCV per ten samples measured or one CCV per project, at a minimum. Use appropriate Standards, LR or HR, for the corresponding instrument modes. Results should fall within the given tolerance limits listed on the Certificate of Analysis included in each standard kit.

1. Record required information on field data sheet for meter and standard identification.
2. Wipe Spec√ blank with lint-free tissue to remove dirt and fingerprints.
3. Insert blank into instrument by aligning the marking on the cell to the marking on the meter. Tightly cover cell with instrument cap.
4. Press the ZERO button and record result on field data sheet.
5. Insert STD 1 into instrument by aligning the marking on the cell to the marking on the meter. Tightly cover cell with instrument cap.
6. Press READ/ENTER button and record result on field data sheet.
7. Repeat Steps 5 and 6 with STD 2 and 3.

6.4.3 High Range Test Procedure for Total or Free Chlorine Using DPD Powder Pillows

1. Make sure instrument is in the high range. The display reads to the tenths place (0.0) when in high range. Refer to Section 6.4.1 to switch ranges.
2. Collect a representative sample of water from a well-mixed region of the waste stream.
3. To prepare sample blank, fill a 1-cm/10-mL high range cell to the 5-mL line with sample. Wipe cell with lint-free tissue to remove moisture and fingerprints.
4. Insert sample blank into instrument by aligning the marking on the cell to the marking on the meter.
5. Cover cell with instrument cap, making sure cap fits tightly against the instrument and press the ZERO button.
6. Fill a second 1-cm/10-mL high range cell to the 5-mL line with sample.
    a. For total chlorine, add the contents of two DPD Total Chlorine Powder Pillows to the sample cell. This initiates the start of the 3-minute waiting period required for the DPD reagent to react with chlorine. This will be the prepared sample. Cap

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cell and gently shake to mix. Keep prepared sample away from light to avoid light sensitivity reactions and to maintain constant temperature during the waiting period. Proceed to Step 7.

b. For free chlorine, add the contents of two DPD Free Chlorine Powder Pillows to the sample cell. Cap the cell, gently shake for about 20 seconds to mix, and wipe cell with lint-free tissue to remove moisture and fingerprints. Proceed to Step 8.

7. As close as possible to the end of the 3-minute waiting period, wipe cell with lint-free tissue to remove moisture and fingerprints.
8. Place prepared sample cell into the instrument cell holder and align markings. Tightly fit instrument cap over cell.
9. Press the READ/ENTER button to obtain results in mg/L.
10. A flashing "5.0" in the display indicates the sample is over range. Dilute a fresh sample and repeat test. Refer to Section 6.4.7 for dilution information.
11. After sampling is complete, perform and record a Continuing Calibration Verification (ICV) using the standards closest to the measurement results obtained. Refer to procedures in Section 6.4.2

6.4.4 Low Range Test Procedure for Total Chlorine Using AccuVac Ampuls

1. Make sure instrument is in the low range. The display reads to the hundredths place (0.00) when in low range. Refer to Section 6.4.1 to switch ranges.
2. Collect a representative sample of water from a well-mixed region of the waste stream.
3. To prepare the sample blank, fill a round 10-mL cell to the 10-mL line with sample and cap.
4. Wipe cell with lint-free tissue to remove moisture and fingerprints. Insert sample blank into instrument by aligning the marking on the cell to the marking on the meter.
5. Cover cell with instrument cap, making sure cap fits tightly against instrument and press the ZERO button.
6. Submerse the tip of a DPD Total Chlorine Reagent AccuVac Ampul into additional sample and snap off tip to fill ampul. This initiates the start of the three-minute waiting period required for the DPD reagent to react with chlorine.
7. Quickly invert the ampul several times to mix. Keep prepared sample away from light to avoid light sensitivity reactions and to maintain constant temperature during the waiting period.
8. As close as possible to the end of the 3-minute waiting period, wipe ampul with a lint-free tissue to remove moisture and fingerprints.
9. Place ampul into instrument cell holder and tightly fit cap over ampul.
10. Press the READ/ENTER button to obtain results in mg/L.
11. For results less than the established Practical Reporting Limit (PRL), report as < 'PRL value'.
12. A flashing "2.20" in the display indicates result is over range. Proceed with High Range Mode Procedure 6.4.3.
13. After sampling is complete, perform and record a Continuing Calibration Verification (ICV) using the standards closest to the measurement results obtained. Refer to procedures in Section 6.4.2

6.4.5 Low Range Test Procedure for Total or Free Chlorine Using DPD Powder Pillows

1. Make sure instrument is in the low range. The display reads to the hundredths place (0.00) when in low range. Refer to Section 6.4.1 to switch ranges.
2. Collect a representative sample of water from a well-mixed region of the waste stream.
3. To prepare the sample blank, fill a round 10-mL cell to the 10-mL line with sample and cap.

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4. Wipe cell with a lint-free tissue to remove moisture and fingerprints. Insert sample blank into instrument by aligning the marking on the cell to the marking on the meter.
5. Cover cell with instrument cap, making sure cap fits tightly against instrument and press the ZERO button.
6. Fill a second round 10-mL cell to the 10-mL line with sample.
   a. For total chlorine, add the contents of one DPD Total Chlorine Powder Pillows to the sample cell. This initiates the start of the 3-minute waiting period required for the DPD reagent to react with chlorine. This will be the prepared sample. Cap the cell and gently shake to mix. Keep prepared sample away from light to avoid light sensitivity reactions and to maintain constant temperature during the waiting period. Proceed to Step 7.
   b. For free chlorine, add the contents of one DPD Free Chlorine Powder Pillows to the sample cell. Cap the cell, gently shake for about 20 seconds to mix, and wipe cell with a lint-free tissue to remove moisture and fingerprints. Proceed to Step 8.
7. As close as possible to the end of the 3-minute waiting period, wipe cell with a lint-free tissue to remove moisture and fingerprints.
8. Place prepared sample cell into the instrument cell holder, align markings, and tightly fit cap over cell
9. Press the READ/ENTER button to obtain results in mg/L.
10. For results less than the established Practical Reporting Limit (PRL), report as < 'PRL value'.
11. A flashing "2.20" in the display indicates result is over range. Proceed with High Range Mode Procedure 6.4.3.
12. After sampling is complete, perform and record a Continuing Calibration Verification (ICV) using the standards closest to the measurement results obtained. Refer to procedures in Section 6.4.2

6.4.6 Chloramines: Total and Speciated

The following procedures are adapted from *Standard Methods, 18th Edition, 4500-CL G. DPD Colorimeter Method.*

A. Total Chloramines

1. Analyze Total Chlorine using procedure in appropriate sections. Refer to Section 6.4.3 through 6.4.5.
2. Analyze Free Chlorine using procedure specified in relevant section. Refer to Section 6.4.3 or 6.4.5.
3. Subtract Free Chlorine result from Total Chlorine Result.

   Total Chlorine – Free Chlorine = Total Chloramines

B. Speciated Chloramines

1. Make sure instrument is in the low range. The display reads to the hundredths place (0.00) when in low range. Refer to Section 6.4.1 for steps to switch ranges. Dilute sample with chlorine-demand-free water when total chlorine exceeds 4 mg/L.
2. Collect a representative sample of water from a well-mixed region of the waste stream.
3. To prepare the **sample blank**, fill a round 10-mL cell to the 10-mL line with sample and cap.
4. Wipe cell with a lint-free tissue to remove moisture and fingerprints. Insert sample blank into the instrument by aligning the marking on the cell to the marking on the

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meter.

5. Cover cell with instrument cap, making sure cap fits tightly against instrument and press the ZERO button.
6. Fill a second round 10-mL cell to the 10-mL line with sample.
7. For **free chlorine**, add the contents of one DPD Free Chlorine Powder Pillow to the sample cell. Cap the cell, gently shake for about 20 seconds to mix, and wipe cell with a lint-free tissue to remove moisture and fingerprints. Immediately place prepared sample cell into the instrument cell holder and align markings.
8. Tightly fit instrument cap over cell and press the READ/ENTER button to obtain results in mg/L.
9. For **monochloramine (NH_2Cl)**, continue by adding one very small crystal (approximately 0.1 mg) of Potassium Iodide (KI) to this same sample cell.
10. Cap cell, gently shake to mix, and wipe cell with a lint-free tissue to remove moisture and fingerprints. Immediately place prepared sample cell into instrument cell holder and align markings.
11. Tightly fit instrument cap over cell and press the READ/ENTER button to obtain the results in mg/L.
12. For **dichloramine ($NHCl_2$)**, continue by adding several more crystals of KI (approximately 0.1g) to this same sample cell.
13. Cap cell, gently shake to mix, and wipe cell with a lint-free tissue to remove moisture and fingerprints and place into instrument.
14. Cover cell and wait 2 minutes.
15. After the waiting period, press the READ/ENTER button to obtain the results in mg/L.
16. For **trichloramine (nitrogen trichloride) (NCl_3),** use a clean sample cell. Place a very small crystal (0.1 mg) of KI into cell. Add 10 mL of sample and mix. Using a second, clean sample cell, add 0.5 mL each of buffer and indicator reagents and mix. Add the contents of the second cell to the first cell. Cap cell, gently shake to mix, and wipe cell with a lint-free tissue to remove moisture and fingerprints. Immediately place prepared sample cell into instrument cell holder and align markings. Tightly fit instrument cap over cell and press the READ/ENTER button to obtain results in mg/L.
17. Use the readings and the table below to calculate the chloramines present within the sample.
18. After sampling is complete, perform and record a Continuing Calibration Verification (ICV) using the standards closest to the measurement results obtained. Refer to procedures in Section 6.4.2

6.4.7 Dilutions for Chlorine

Dilution is often required when a high level of chemical is found in a sample. When the Hach Pocket Colorimeter II instruments high range chlorine limit is reached, "5.0" will flash in the viewing window. In order to bring the level of chlorine into a measurable range and obtain an accurate chlorine reading, a dilution is performed.

To dilute a chlorine sample, DI water is added to the sample in a specific ratio. A dilution factor is found by dividing the final volume by the initial volume of the solution. The measurement obtained from the diluted sample is multiplied by the dilution factor in order to obtain the actual result.

A dilution factor of 1 indicates a sample was not diluted. A dilution factor of 2 indicates equal parts sample and DI water were mixed together. A dilution factor of 5 indicates 1 part sample was mixed with 4 parts DI water. A dilution factor of 10 means 1 part sample was mixed with

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9 parts DI water or 10 parts sample to 90 parts DI water.

To make a dilution:

1. Choose a dilution factor.
2. Determine the appropriate ratio of sample to DI water needed in order to obtain the dilution factor.

   Dilution Factor = Final Volume / Sample Volume

3. Accurately measure determined volumes using pipets. Combine and mix in appropriate container.
4. Proceed with chlorine procedure using the diluted solution for sample and sample blank.
5. Multiply result by dilution factor to obtain actual chlorine level in mg/L.

6.5 Oxidation Reduction Potential (ORP) / Redox

Oxidation Reduction Potential (ORP), or redox, measures the tendency of a medium to transfer electrons as sensed by a metal electrode. A voltage is produced relative to a reference electrode. A positive value indicates oxidation: the ability to accept electrons. A negative value indicates reduction: the giving up of electrons. Agents influencing ORP values include salts, chlorine, and sulfite ions.

Note about ORP vs. Eh: the two parameters are similar in that both quantify the potential of a medium to transfer electrons. The two parameters differ in the reference electrode used in the measurement. Eh is the voltage reading using the Standard Hydrogen Electrode (SHE). ORP is the voltage reading using any theoretical reference electrode. It is difficult to use SHE in the field, so Eh is usually not determined directly. Voltage obtained as ORP readings can be converted into Eh values using an offset of the fixed difference between the SHE and the other reference method. The difference may be accounted for prior to field measurements during the calibration of the instrument or after readings are performed.

The temperature of the sample being measured affects the voltage output of the sensor. ORP readings measured by YSI sondes are not temperature compensated. When calibrating probe, use a temperature and reference solution table to obtain the proper value for the relevant calibration standard and temperature. Maintain a constant temperature when measuring sample and report both ORP value and temperature. ORP is also sensitive to pH. Values tend to increase as pH decreases and decrease as pH increases. Record ORP values to the nearest 1 mV and temperature values to the nearest 0.1°C

In the field, variations in measurements may be due to temperature changes. Large changes in ORP readings, i.e., >100 mV, are generally associated with analyte fluctuations rather than temperature variations. Technicians must be aware of electrode contamination. Special care is required in treating and storing the platinum electrodes. Periodic maintenance of probe will increase consistency and accuracy of measurements.

6.5.1 YSI 600XL Sonde / 650 MDS

Since the ORP and pH sensors are combined on YSI sondes, the pH sensor must be calibrated first, before calibrating ORP, to ensure proper functioning. If the pH probe does not properly calibrate for any reason, the ORP function is disabled.

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6.5.1.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Place Zobell solution or equivalent into a clean, dry calibration cup.
2. Record temperature of solution; solution temperature should be within ± 5°C of sample temperature. *Note*: an ice bath can be used to adjust the ZoBell solution temperature.
3. Immerse probe into the Zobell solution, or equivalent, and allow probe to equilibrate for a minimum of 1 minute.
4. Select 'ORP' from calibration menu and enter the value of Zobell solution listed on Attachment 12 - Quick Reference Table at the appropriate temperature.
5. Press **Enter** and observe displayed values. Once stable, press **Enter** to accept value if within ± 10 mV of the theoretical value. If the reading is not within ± 10 mV, one of the following steps will usually solve the problem.
   a. Check reference electrode filling solution: fill or replace.
   b. Polish the platinum end of the electrode (see manufacturer instructions).
   c. Replace electrode.
6. Record calibration value to the nearest 1 mV and temperature to the nearest 0.1°C.
7. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using a second source standard if available:
   a. Enter measurement mode and immerse probe into second source standard.
   b. Gently agitate probe and observe reading. When no significant change is observed, record measured result and standard information on appropriate data sheet. The ICV result must be within ± 10 mV of the standard value to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, re-calibrate the meter.
8. Rinse electrode with DI water and blot dry with a lint-free tissue.

6.5.1.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Rinse probe with sample water and immerse into sample.
2. Let value equilibrate; equilibration times vary depending on the composition of the sample. Record ORP value to nearest 1 mV and temperature to nearest 0.1°C.
3. Repeat Steps 1-2 with a second sample portion. Successive readings varying less than ± 10 mV over 10 minutes are adequate for most purposes.
4. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. In measurement mode, immerse probe into clean, dry calibration cup containing Zobell solution.
   b. Agitate probe and wait until a stable reading is observed. Record value to nearest 1 mV and temperature to nearest 0.1°C
   c. Verify temperature reading matches solution value used. The measured result must be within ± 10 mV of the standard value to verify correct meter operation. If the data does not meet this acceptance criterion, re-calibrate the system and re-analyze the sample(s). If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.

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6.5.2 YSI 556 MPS

For additional operational instructions, refer to Section 6.10.

6.5.2.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on meter using **On/Off** key.
2. Press **Escape** to display the main menu screen.
3. Highlight the **ORP** selection and press **Enter.** The ORP Calibration Selection Screen is displayed.
4. Highlight the **ORP** selection and press **Enter** to display the ORP calibration screen.
5. Ensure the sensor is as dry as possible. Rinse the ORP sensor with a small amount of ORP solution that can be discarded.
6. Put a small amount of a known ORP solution (i.e., Zobell solution) into a clean, dry or pre-rinsed transport/calibration cup.
7. Completely immerse sensor into solution and gently agitate to remove any bubbles from the ORP sensor.
8. Screw the transport/calibration cup on the probe and securely tighten. Do not over tighten as this could cause damage to the threaded portions.
9. Enter the correct value of the Zobell solution as listed on Attachment 12 - Quick Reference Table at the current temperature using the keypad and press **Enter**.
10. Current values of all enabled sensors appear on the screen and change as they stabilize. Allow at least one minute for temperature equilibration before proceeding. Verify the temperature reading matches the solution value used.
11. Watch the reading under ORP and when the reading shows no significant change for approximately 30 seconds, press **Enter** to accept the calibration.
12. Record calibration value to the nearest 1 mV and temperature to the nearest 0.1°C.
13. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) if available:
    a. Enter measurement mode and immerse probe into second source standard.
    b. Gently agitate probe and observe reading. When no significant change is observed, record measured value and standard information on appropriate data sheet. The measured result must be within ±10 mV of the standard value to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, re-calibrate the meter.
14. Rinse the probe and sensors in tap or purified water and dry.

6.5.2.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using **On/Off** key.
3. Display run screen by selecting Run from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate probe to provide fresh sample to the sensor.
5. Watch the readings on the display until they are stable. Record value to nearest 1 mV and temperature to nearest 0.1°C,
6. Rinse probe and shake off excess water.

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7. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. In measurement mode, immerse probe into clean, dry calibration cup containing Zobell solution.
   b. Agitate probe and wait until a stable reading is observed. Record result to nearest 1 mV and temperature to nearest 0.1°C
   c. Verify the temperature reading matches the solution value used. The measured result must be within ±10 mV of the standard value to verify correct meter operation. If the data is outside this criterion, re-calibrate the system and re-analyze the sample(s). If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.
8. Refer to Section 6.10 for instructions on logging sample data.

6.5.3 Hach Hydrolab Minisonde 5 (MS5)

6.5.3.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Attach the power and data cable to the Sonde. Attach the 9-pin connector to the Surveyor.
2. Turn on the Surveyor, and wait approximately 10 seconds for initialization.
3. Press **SETUP/CAL**. Press **CALIBRATION**. Press **SONDE**.
4. Prepare sensors for calibration by rinsing sensors with deionized water.
   a. Remove sensor guard and attach calibration cup.
   b. Unscrew and remove cap of calibration cup.
   c. Half-fill calibration cup with deionized water and place cap on calibration cup.
   d. Shake sonde in DI water to ensure sensors are free from contaminants. Discard DI water. Repeat several times if necessary.
   e. Place small amount of calibration solution into calibration cup and rinse sonde sensors. Discard calibration solution.
5. Use the **ARROW** keys to highlight ORP and press **SELECT**. A calibration screen will display.
6. Pour a known ORP standard (i.e., Zobell solution) to within a centimeter of the top of the Calibration Cup.
7. Enter the units for ORP; use the correct value of the Zobell solution as listed on Attachment 12 - Quick Reference Table at the current temperature.
8. Completely immerse sensor into solution and gently agitate to remove any bubbles from the ORP sensor.
9. Watch the reading and when the reading shows no significant change for approximately 30 seconds, press **DONE** to finish the calibration.
10. Record calibration value to the nearest 1 mV and temperature to the nearest 0.1°C.
11. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) if available:
    a. Enter measurement mode and immerse probe into second source standard.
    b. Gently agitate probe and observe reading. When no significant change is observed, record measured value and standard information on appropriate data sheet. The measured result must be within ±10 mV of the standard value to verify correct meter operation; the meter should not be used

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unless a passing ICV is achieved. If this criterion is not met, re-calibrate the meter.

12. Rinse the probe and sensors in tap or purified water and dry.

6.5.3.2 Measurement Procedure and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on instrument using **On/Off** key.
3. Display run screen by selecting Run from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate probe to provide fresh sample to the sensor.
5. Watch the readings on the display until they are stable. Record value to nearest 1 mV and temperature to nearest 0.1°C,
6. Rinse probe and shake off excess water.
7. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. In measurement mode, immerse probe into clean, dry calibration cup containing Zobell solution.
   b. Agitate probe and wait until a stable reading is observed. Record result to nearest 1 mV and temperature to nearest 0.1°C
   c. Verify the temperature reading matches the solution value used. The measured result must be within ±10 mV of the standard value to verify correct meter operation. If the data is outside this criterion, re-calibrate the system and re-analyze the sample(s). If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.

6.6 Dissolved Oxygen (DO)

Dissolved oxygen analysis measures the amount of oxygen dissolved in water. This oxygen can come from surrounding air, rapidly moving water, or as a product of photosynthesis. Temperature influences the amount of oxygen a body of water can hold; warm water holds less oxygen than cold water.

Pace FSD conducts dissolved oxygen analysis following procedures outlined below; these procedures are based on Standard Methods 4500-O and manufacturer calibration and verification procedures. Project situational deviations are fully documented at the time of testing.

The dissolved oxygen probe is calibrated each day prior to use. An Initial Calibration (IC) is performed with water-saturated air. Calibrations performed using water-saturated air calibrate percent saturation. Since mg/L is calculated within the meter from percent saturation, calibrating percent saturation automatically calibrates mg/L. An Initial Calibration Verification (ICV) is performed using water-saturated air, or when specified by project requirements a zero-oxygen solution, and a Continuing Calibration Verification (CCV) is performed after measurement results are obtained. An initial inspection and calibration should be performed the day before use to inspect for damage and to assure the membrane is in good shape the instrument is working properly. Follow meter's instruction manual for maintenance and replacement procedures.

Barometric pressure measurements required for dissolved oxygen meter calibrations are obtained from barometers traceable to national standards maintained by NIST. These barometers are

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calibrated every 6 months. Barometric pressure readings can be recorded in either mm Hg or inches Hg.

Dissolved Oxygen results are reported in mg/L. To measure, immerse probe into sample and rapidly stir sample with probe to avoid oxygen depletion at the water-probe interface. It is suggested to move the probe approximately 6 inches per second. Record DO measurements to the nearest 0.1 mg/L unless otherwise specified.

General Dissolved Oxygen Calibration Procedures:

1. Create a water-saturated air environment in a calibration cup by following either:
   a. Dampen sponge/tissue in bottom of calibration cup.
   b. Place about 1/8" of water in bottom of calibration cup.
2. Place probe into calibration cup without the membrane contacting the damp sponge/tissue or water.
3. While probe is stabilizing, calculate the % Saturation/Calibration Value of the air using this equation:

   (Measured Barometric Pressure / Sea Level Barometric Pressure) * 100

   Note: *Sea Level Barometric Pressure is a constant of 760 mm Hg or 29.92 Inches Hg.*

4. Initial Calibration (IC): Once a stable reading is observed, record the meter reading on the data sheet.
5. Remove the probe from the calibration cup.
6. Initial Calibration Verification (ICV): Once again, place the probe into the calibration cup and allow the reading to stabilize. Record the meter reading on the data sheet. If the reading is within 2% of the % Saturation/Calibration Value, the ICV is acceptable.
7. Continuing Calibration Verification (CCV): At the appropriate interval during dissolved oxygen sampling, perform a CCV by placing the sensor back into the same calibration environment as that used during initial calibration. The CCV is acceptable if the meter reading is within 2% of the % Saturation/Calibration Value once stable.

6.6.1 YSI 51B DO Meter

6.6.1.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Set indicator knob to off position and adjust meter pointer to zero with the screw in the center of the meter panel.
2. Set indicator knob to the zero position and adjust the meter pointer to zero using the zero knob.
3. Set indicator knob to the Full Scale position and adjust the meter pointer to the 15 mark on the mg/L scale.
4. Allow approximately 15 minutes for probe to stabilize before calibration. (Allow 15 minutes for repolarization whenever the instrument has been turned off or the probe disconnected.)
5. After stabilization, switch the indicator dial to CALIB O_2 to calibrate the meter using water-saturated air.
6. Create a water-saturated air environment in the calibration cup by either:
   a. Dampen sponge/tissue in bottom of calibration cup.
   b. Place about 1/8" of water in bottom of calibration cup.
7. Place probe into cover without the membrane contacting the damp sponge/tissue or water.

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8. While probe is stabilizing, set meter pointer to the local altitude by using the CALIB knob.
9. Once stabilized (approximately 10 min.), the reading must be within 2% of the % Saturation/Calibration Value.
10. Record the barometric pressure, % Saturation/Calibration Value, and DO measurement on the data sheet.
11. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using either water-saturated air or a zero-oxygen solution depending on project objectives. Be sure to obtain the correct data sheet if using zero-oxygen solution.
    a. If using water-saturated air:
        i. Create a water-saturated air environment in calibration cup following the steps above.
        ii. In measurement mode, read and record the DO measurement once stable. The reading must be within 2% of the % Saturation/Calibration Value for the ICV to be acceptable.
        iii. If the acceptance criterion is not met, re-calibrate the meter.
    b. If using a zero-oxygen solution:
        i. Enter measurement mode and immerse probe into a zero-oxygen solution.
        ii. Agitate probe and observe reading. When no significant change is observed, record the measured value on appropriate data sheet. The measured result of the zero-oxygen solution must read less than 0.5 mg/L to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If this criterion is not met, re-calibrate the meter.
12. Once acceptable Initial Calibration and Initial Calibration Verification results are obtained, the meter is ready for use.

6.6.1.2 Measurement and Continuing Calibration Verification (CCV)

1. Immerse probe into sample and rapidly stir. Allow a few minutes for temperature equilibration. Switch knob to Temp. and read the temperature from the lower meter scale.
2. Set O_2 Solubility Factor dial to that temperature.
3. Switch to Read O_2 and read value from meter in mg/L.
4. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
    a. Create a water-saturated air environment in the calibration cup by either:
        i. Dampen a sponge/tissue in calibration cup.
        ii. Place about 1/8" of water in the bottom of the calibration cup.
    b. In measurement mode, loosely place probe into calibration cup; do not let the membrane contacting the damp sponge/tissue or water.
    c. Allow dissolved oxygen and temperature readings to stabilize.
    d. Record dissolved oxygen and temperature on appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable. If the data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified when reported.

6.6.2 YSI 600XL Sonde / 650 MDS

Barometric pressure is measured by the 650 MDS data logger and used for depth and dissolved oxygen calculations. The barometric pressure is checked against a barometer traceable to national standards maintained by NIST every six months.

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6.6.2.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on sonde and allow it to idle (not in "Run" mode) for 5 minutes prior to starting DO calibration procedures.
2. Create a water-saturated air environment in the calibration cup by either:
   a. Dampen a sponge in calibration cup.
   b. Place about 1/8" of water in the bottom of the calibration cup.
3. Place the probe end of the sonde into the cap. Engage only 1 or 2 threads of the calibration cap to insure the DO probe is vented to the atmosphere. Do not allow probe to contact sponge/water and ensure the Sonde is not in direct sunlight.
4. Allow approximately 10 minutes for the air in the calibration cup to become water saturated and for the temperature to equilibrate.
5. From calibrate menu select Dissolved Oxy and press Enter. Select DO% to calibrate as a percentage. Calibrating using DO% also calibrates DO mg/L results.
7. Enter the current barometric pressure in mm of Hg. (Inches of Hg x 25.4 = mm Hg)
8. Press Enter to display the current values on screen.
9. Allow DO value to stabilize (i.e., no significant change for approximately 30 seconds).
10. Press Enter to accept value. Record dissolved oxygen in mg/L and %, temperature, and barometric pressure on the appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable.
11. If the data does not meet this criterion, the meter must be recalibrated.
12. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using either water-saturated air or a zero-oxygen solution depending on project objectives. Be sure to obtain the correct data sheet if using zero-oxygen solution.
13. If using water-saturated air:
    a. Create a water-saturated air environment in the calibration cup and measure DO following procedures above.
    b. Record the reading. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable.
    c. If the acceptance criterion is not met, re-calibrate the meter. The meter should not be used unless a passing ICV is achieved.
14. If using a zero-oxygen solution:
    a. Enter measurement mode and immerse probe into a zero-oxygen solution.
    b. Agitate probe and observe reading. When no significant change is observed, record measured value on appropriate data sheet. The measured result of the zero-oxygen solution must read less than 0.5 mg/L to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If outside this criterion, re-calibrate the meter.
    c. Once acceptable Initial Calibration and Initial Calibration Verification results are obtained, rinse the probe module and sensors in tap or purified water. The meter is ready for use.

6.6.2.2 Measurement and Continuing Calibration Verification (CCV)

1. Turn on instrument using On/Off key.
2. Display run screen by selecting 'Run' from the main menu.
3. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
4. Watch the readings on the display until they are stable.
5. Record reading and rinse probe.
6. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times,

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a. Create a water-saturated air environment in the calibration cup by either:
   i. Dampen a sponge in the bottom of the calibration cup.
   ii. Place about 1/8" of water in the bottom of the calibration cup.
b. In measurement mode, loosely place probe into calibration bottle; do not allow membrane to contact sponge/water and ensure the Sonde is not in direct sunlight.
c. Allow dissolved oxygen and temperature readings to stabilize (i.e., no significant change for approximately 30 seconds).
d. Record dissolved oxygen and temperature on appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable. If the data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified when reported.

7. Refer to Section 6.10 for instructions on logging sample data.

6.6.3 YSI 556 MPS

For additional operational instructions, refer to Section 6.10.

6.6.3.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

Note: Calibrating any one option (% or mg/L) automatically calibrates the other.

1. Turn on meter using **On/Off** key and allow it to idle (not in "Run" mode) for 5 minutes prior to starting DO calibration procedures.
2. Press **Escape** to display the main menu screen.
3. The instrument must be on for at least 20 minutes to polarize the DO sensor before calibrating.
4. Highlight the **Calibrate** selection and press **Enter**. The Calibrate screen is displayed.
5. Highlight the **Dissolved Oxygen** selection and press **Enter** to display the dissolved oxygen calibration screen.
6. To select membrane material of DO sensor,
   b. Highlight "DO NO" (DO None) entry and press **Enter** to display the membrane choice screen.
   c. Highlight the desired membrane choice and press **Enter** to activate the selection with a dot to the left of the screen. **NOTE:** Blue membrane caps using 2 mil polyethylene (PE) are likely to be the best choice for most 556 field applications.
   d. Press **Escape** to return to the Sensor menu. The DO membrane selected is displayed.
7. For DO Calibration in % Saturation, use the arrow keys to highlight the DO% selection.
   a. Press **Enter** to display the DO Barometric Pressure Entry Screen. Enter the current barometric pressure from a calibrated barometer.
   b. Place approximately 3 mm (1/8 inch) of water in the bottom of the transport/calibration cup.
   c. Place the probe module into the transport/calibration cup but **DO NOT** immerse the DO and temperature sensors in the water.
   d. Engage only 1 or 2 threads of the transport/calibration cup to ensure the DO sensor is vented to the atmosphere to allow pressure equilibration.
   e. Enter the current local barometric pressure using the keypad. If the unit has an optional barometer, no entry is required. Barometer readings that appear in meteorological reports are generally corrected to sea level and must be uncorrected before use.
   f. Press **Enter** to display the DO% saturation calibration screen where current values of all enabled sensors appear on the screen and change as they stabilize.

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Allow approximately ten minutes for the air in the transport/calibration cup to become water saturated and for the temperature to equilibrate before proceeding.

g. Watch the reading under DO %. When the reading shows no significant change for approximately 30 seconds, press **Enter** to accept the value.
h. Record dissolved oxygen in mg/L and %, temperature, and barometric pressure on the appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable. If the data does not meet this requirement, the meter must be recalibrated.
i. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using either water-saturated air or a zero-oxygen solution depending on project objectives. Be sure to obtain the correct data sheet if using zero-oxygen solution.
    a. If using water-saturated air,
        i. Create a water-saturated air environment in the calibration cup by following either Step 1) or 2):
            1) Dampen a sponge in bottom of calibration cup.
            2) Place about 1/8" of water in bottom of calibration cup.
        ii. In measurement mode, loosely place probe into calibration cup; do not allow membrane to contact sponge/water and ensure the meter is not in direct sunlight.
        iii. Allow dissolved oxygen and temperature readings to stabilize (i.e., no significant change for approximately 30 seconds).
        iv. Record measured value and temperature on appropriate data sheet.
        v. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable.
    b. If using a zero-oxygen solution,
        i. Enter measurement mode and immerse probe into a zero-oxygen solution.
        ii. Agitate probe and observe reading. When no significant change is observed, record measured value on appropriate data sheet. The measured result of the zero-oxygen solution must read less than 0.5 mg/L to verify correct meter operation.
j. The meter should not be used unless a passing ICV is achieved. If criterion is not met, re-calibrate meter.
k. Rinse the probe module and sensors in tap or purified water. The meter is now ready for use.

8. For DO Calibration in mg/L, calibration is carried out in a water sample which has a known concentration of dissolved oxygen (usually determined by a Winkler titration).
    a. Turn on meter using **On/Off** key.
    b. Press **Escape** to display the main menu screen.
    c. The instrument must be on for at least 20 minutes to polarize the DO sensor before calibrating.
    d. Highlight the **DO mg/L** selection and press **Enter** to display the DO mg/L Entry Screen.
    e. Completely immerse all sensors in water with a known DO concentration and use the keypad to enter the known DO concentration of the water.
    f. Press **Enter** and rapidly agitate the probe to provide fresh sample to the DO sensor.
    g. The current value of all enabled sensors appear on the screen and change as they stabilize. Allow at least one minute for temperature equilibration before proceeding.
    h. Watch the DO mg/L reading and when the reading is stable (shows no significant change for approximately 30 seconds), press **Enter** to accept the calibration.

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i. Record dissolved oxygen in mg/L and %, temperature, and barometric pressure on the appropriate data sheet. The dissolved oxygen value should be within ±0.5 mg/L of the known value. If the data does not meet this criterion, the meter must be recalibrated.

j. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using either water-saturated air or a zero-oxygen solution depending on project objectives. Be sure to obtain the correct data sheet if using zero-oxygen solution.
   a. If using water-saturated air,
      i. Create a water-saturated air environment in the calibration cup by following either Step 1) or 2):
         1) Dampen a sponge in bottom of calibration cup.
         2) Place about 1/8" of water in bottom of calibration cup.
      ii. In measurement mode, loosely place probe into calibration cup; do not allow membrane to contact sponge/water and ensure the meter is not in direct sunlight.
      iii. Allow dissolved oxygen and temperature readings to stabilize (i.e., no significant change for approximately 30 seconds).
      iv. Record measured value and temperature on appropriate data sheet.
      v. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable.
   b. If using a zero-oxygen solution,
      i. Enter measurement mode and immerse probe into a zero-oxygen solution.
      ii. Agitate probe and observe reading. When no significant change is observed, record measured value on appropriate data sheet. The measured result of the zero-oxygen solution must read less than 0.5 mg/L to verify correct meter operation.

k. The meter should not be used unless a passing ICV is achieved. If criterion is not met, re-calibrate meter.

l. Rinse the probe module and sensors in tap or purified water. The meter is now ready for use.

6.6.3.2 Measurement and Continuing Calibration Verification (CCV)

1. Make sure correct probe is attached to the instrument and is enabled to measure sample.
2. Turn on the instrument using On/Off key.
3. Display run screen by selecting 'Run' from the main menu.
4. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
5. Watch the readings on the display until they are stable.
6. Record reading and rinse probe.
7. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
   a. Create a water-saturated air environment in the calibration cup by either:
      i. Dampen a sponge in bottom of calibration cup.
      ii. Place about 1/8" of water in bottom of calibration cup.
   b. In measurement mode, loosely place probe into calibration cup; do not allow membrane to contact sponge/water and ensure meter is not in direct sunlight.
   c. Allow sufficient time for dissolved oxygen and temperature readings to stabilize.

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d. Record dissolved oxygen and temperature measurements on appropriate data sheet.
e. Record dissolved oxygen and temperature on appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable. If the data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified when reported.

8. Refer to Section 6.10 for instructions on logging sample data.

6.6.4 Hach Hydrolab Minisonde 5 (MS5)

Dissolved oxygen calibrations can be performed using water-saturated air or using a water sample with a known dissolved oxygen concentration. Note: If there is a change in barometric pressure after calibration, the readings for D.O. % Saturation will not be correct. You must enter a new barometric pressure. However, the readings for D.O. mg/L will be correct regardless of changes in barometric pressure

6.6.4.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

I. Saturated-Air Method

Calibration of D.O. % Saturation also calibrates D.O. mg/L. ***NOTE:*** The saturated-air method is valid only for the Clark Cell dissolved oxygen sensor.

1. Attach the power and data cable to the Sonde; attach the 9-pin connector to the Surveyor.
2. Turn on the Surveyor, and wait approximately 10 seconds for initialization.
3. Press SETUP/CAL. Press CALIBRATION. Press SONDE.
4. Use the ARROW keys to highlight DISSOLVED OXYGEN and press SELECT. A calibration screen will display.
5. Fill the Calibration Cup with deionized or tap water (Water must have specific conductance less than 0.5 mS/cm.) until the water is just below the membrane O-ring. Do not allow water to contact the membrane or the O-ring. Carefully remove any water droplets from the membrane with the corner of a tissue.
7. Turn the black cap upside down (concave upward) and lay it over the top of the Calibration Cup. This stops the exchange of air and allows the local environment to equilibrate. Wait for the reading to stabilize.
8. Determine the true barometric pressure for entry as the calibration standard. Barometric pressure information can be obtained from a local weather station or airport or the Surveyor (if equipped with BP).

9. Press DONE to finish calibration. Record dissolved oxygen in mg/L and %, temperature, and barometric pressure on the appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable.
11. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV) using either water-saturated air or a zero-oxygen solution depending on project objectives:
    a. If using water-saturated air:
        i. Create a water-saturated air environment in the calibration cup and measure DO following procedures above.
        ii. If the acceptance criterion is not met, re-calibrate the meter. The meter should not be used unless a passing ICV is achieved.

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b. If using a zero-oxygen solution:
   i. Enter measurement mode and immerse probe into a zero-oxygen solution.
   ii. Agitate probe and observe reading. When no significant change is observed, record measured value on appropriate data sheet. The measured result of the zero-oxygen solution must read less than 0.5 mg/L to verify correct meter operation; the meter should not be used unless a passing ICV is achieved. If outside this criterion, re-calibrate the meter.

12. Once acceptable Initial Calibration and Initial Calibration Verification results are obtained, rinse the probe module and sensors in tap or purified water. The meter is ready for use.

II. Known-Concentration Method

Calibration of D.O. mg/L also calibrates D.O. % Saturation.

This calibration method is more difficult to perform than the saturated-air method but can yield a higher accuracy if the "known" D.O. concentration is highly accurate.

1. Immerse the sensor in a water bath for which the D.O. concentration in mg/L is known.
2. Enter the barometric units (mmHg).
3. Enter the D.O. units in mg/L.

6.6.4.2 Measurement and Continuing Calibration Verification (CCV)

1. Turn on instrument using On/Off key, and display run screen.
2. Install probe sensor guard, completely immerse all sensors in sample and rapidly agitate the probe to provide fresh sample to the sensor.
3. Watch the readings on the display until they are stable.
4. Record reading and rinse probe.
5. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times,
   a. Create a water-saturated air environment in the calibration cup by following either Step i or ii:
      i. Dampen a sponge in the bottom of the calibration cup.
      ii. Place about 1/8" of water in the bottom of the calibration cup.
   b. In measurement mode, loosely place probe into calibration bottle; do not allow membrane to contact sponge/water and ensure the Sonde is not in direct sunlight.
   c. Allow dissolved oxygen and temperature readings to stabilize (i.e., no significant change for approximately 30 seconds).
   d. Record dissolved oxygen in mg/L and %, temperature on appropriate data sheet.
   e. Record dissolved oxygen and temperature on appropriate data sheet. The reading must be within 2% of the % Saturation/Calibration Value for the value to be acceptable. If the data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured values must be appropriately qualified when reported.

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6.7 Turbidity

Pace FSD conducts turbidity analysis following procedures outlined below; these procedures are based on Standard Method 2130B. Project situational deviations are fully documented at the time of testing.

Turbidity is a measure of the amount of light scattered by suspended particles within a water sample. Suspended materials found in water may include sand, clay, organic matter, microbes, or other substances. A turbidity meter contains a light source that illuminates the water sample and a photoelectric cell which measures the intensity of light scattered by particulate. More suspended material increases the scattering of light resulting in a higher turbidity value.

Each meter is calibrated before use according to manufacturer instructions using commercially prepared standards. Measure turbidity immediately after collection to minimize bias from particulate settling. Remove air or other entrained gasses using approved techniques, as needed. If result is over meters range, dilute sample. Bailers are not recommended for collecting turbidity samples as its use can increase turbidity. Turbidity is measured prior to a flow cell if in use.

Record turbidity measurements according to the table below:

Measurement Range (NTU)	Record to Nearest
0.0-1.0	0.05
1-10	0.1
10-40	1
40-100	5
100-400	10
400-1000	50
> 1000	100

6.7.1 Turb 350 IR: Portable Turbidimeter

The Turb 350 IR enables rapid and reliable turbidity measurements of individual samples using cuvettes. Initial Calibration (IC) is performed using four calibration standards provided with the meter. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) are conducted using these same standards unless a second source standard is available. If a second source standard is available, a single point Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) is performed.

6.7.1.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on meter and allow to warm up for approximately 75 seconds.
2. Press Cal key. Cal 1 will appear on display.
3. Wipe Cal 1 standard (1000 NTU) with a lint-free tissue to remove dirt and fingerprints.
4. Align in meter and press the arrow key.
5. Record turbidity value on appropriate data sheet.
6. Repeat Steps 2 - 5 for Cal 2 standard (100 NTU), Cal 3 standard (10.0 NTU), and Cal 4 standard (0.02 NTU).
7. Meter returns to measurement mode after Cal 4 standard is complete.

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8. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV):
    a. If using the meter's four calibration standards:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. Refer to procedures above. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
    b. If using a second source standard:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
9. The meter should not be used unless a passing ICV is achieved. If criterion is not met, re-calibrate the meter.
10. Rinse the probe module and sensors in tap or purified water. The meter is now ready for use.

6.7.1.2 Measurement and Continuing Calibration Verification (CCV)

1. Turn meter on and allow to warm up.
2. Rinse clean cuvette with sample water.
3. Fill cuvette with sample ensuring no air bubbles are present.
4. Cap and wipe cuvette with a lint-free tissue to remove moisture and fingerprints.
5. Insert cuvette into turbidimeter and press the arrow key.
6. Record turbidity value on display.
7. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
    a. If using the meter's four calibration standards:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. Refer to procedures in Section 6.7.1.1. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
    b. If using a second source standard:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
8. If data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.

6.7.2 Turbidity DRT 15CE

The Turbidity DRT 15CE enables rapid and reliable turbidity measurements of individual samples using cuvettes. Initial Calibration (IC) is performed using four calibration standards provided with the meter. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) are conducted using these same standards unless a second source standard is available. If a second source standard is available, a single point Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) is performed.

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6.7.2.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn range dial to the 10-NTU range.
2. Set the reference adjust to the maximum up position.
3. Wipe the 0.18-NTU calibration standard with a lint-free tissue and insert into instrument holder.
4. Adjust the reference offset R2 until the display reads 0.18-NTU.
5. Set the reference adjust to 0.02-NTU.
6. Wipe the 10-NTU standard with a lint-free tissue and insert into instrument holder.
7. Adjust reference offset R11 to obtain a reading of 10.00-NTU.
8. Rotate the range dial to the 100-NTU range.
9. Wipe the 100-NTU standard with a lint-free tissue and insert into instrument holder.
10. Adjust the reference offset R12 to obtain a reading of 100-NTU.
11. Rotate the range dial to the 1000-NTU range.
12. Wipe the 1000-NTU standard with a lint-free tissue and insert into instrument holder.
13. Adjust the reference offset R13 to obtain a reading of 1000-NTU.
14. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV):
    a. If using the meter's four calibration standards:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. Refer to procedures above. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
    b. If using a second source standard:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
15. The meter should not be used unless a passing ICV is achieved. If criterion is not met, re-calibrate the meter.
16. Rinse the probe module and sensors in tap or purified water. The meter is now ready for use.

6.7.2.2 Measurement and Continuing Calibration Verification (CCV)

1. Fill clean cuvette to within approximately one half inch of the top with sample water.
2. Cap cuvette and wipe with lint-free tissue to remove fingerprints and moisture.
3. Place cuvette into instrument cell and record displayed reading. Select a range for best resolution.
4. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
    a. If using the meter's four calibration standards:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. Refer to procedures in Section 6.7.2.1. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
    b. If using a second source standard:
        i. Set meter to measurement mode.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.

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5. If data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.

6.7.3 Hach Portable Turbidimeter Model 2100P

NOTE: *Do not hold the instrument during measurements; place the instrument on a flat, stable surface.*

The Hach Model 2100P Portable Turbidimeter operates on the nephelometric principle of turbidity measurement. The instrument operates on batteries. The instrument automatically shuts off after 5.5 minutes if no keystrokes occur. If this occurs, simply turn the instrument on – the 2100P will resume operation as if the power had not been interrupted.

The optical system includes a tungsten-filament lamp, a 90° detector to monitor scattered light, and a transmitted light detector. The optical design minimizes stray light, increasing measurement accuracy, and corrects for interferences from color and/or light absorbing materials and compensates for fluctuations in lamp intensity. It is suggested to remove sample cell and batteries from instrument if the instrument is stored for more than a month.

NOTE: *Avoid prolonged exposure to ultraviolet light and sunlight.*

6.7.3.1 Initial Calibration (IC) and Initial Calibration Verification (ICV)

1. Turn on meter and allow to warm up for approximately 75 seconds.
2. Press Cal key. Cal 1 will appear on display.
3. Wipe Cal 1 standard (800 NTU) with a lint-free tissue to remove dirt and fingerprints.
4. Align in meter and press the arrow key.
5. Record turbidity value on appropriate data sheet.
6. Repeat Steps 2 - 5 for Cal 2 standard (100 NTU), Cal 3 standard (20 NTU), and Cal 4 standard (0.1 NTU).
7. Meter returns to measurement mode after Cal 4 standard is complete.
8. Once calibration steps have been completed, perform an Initial Calibration Verification (ICV):
   a. If using the meter's four calibration standards:
      i. Set meter to measurement mode.
      ii. Read and record standard information and measurement data on appropriate data sheet. Refer to procedures above. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
   b. If using a second source standard:
      i. Set meter to measurement mode.
      ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
9. The meter should not be used unless a passing ICV is achieved. If criterion is not met, re-calibrate the meter.
10. Rinse the probe module and sensors in tap or purified water. The meter is now ready for use.

6.7.3.2 Measurement and Continuing Calibration Verification (CCV)

1. Collect a representative sample in a clean container.

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2. Rinse clean cuvette with sample water. Fill a sample cell to the 15 mL line, taking care to handle the sample cell by the top.
3. Cap the cell and wipe the cell with a soft, lint-free cloth to remove moisture and fingerprints.
4. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface. When applied in a thin, uniform coat, the oil fills in and masks minor scratches and other imperfections in the glass. Avoid application of excess oil as it may retain dirt and contaminate the instrument's cell compartment.
5. Turn on the instrument using the power [I/O] key. Place the instrument on a flat, sturdy surface. Do not hold the instrument while making measurements.
6. Insert the sample cell in to the cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the lid.
7. Select meter range and mode:
    a. Select manual or automatic range selection by pressing the RANGE key. The display will show AUTO RNG when the instrument is in automatic range selection.
    b. Select signal averaging mode by pressing the SIGNAL AVERAGE key. The display will show SIG AVG when the instrument is using signal averaging. Use signal average mode if the sample causes a noisy signal (i.e., display changes constantly).

    NOTE: Signal averaging uses more power and should be used only when the sample causes an unstable reading. Signal averaging measures and averages ten measurements while displaying intermediate results.

8. Press the [READ] key. The display will show "- - - - NTU", then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

9. Perform a Continuing Calibration Verification (CCV). It is recommended to perform one CCV per ten samples measured, or if fewer than ten samples are measured, a CCV is performed at the conclusion of analysis to ensure accuracy within sample hold times.
    a. If using the meter's four calibration standards:
        i. Refer to procedures in Section 6.7.1.1.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
    b. If using a second source standard:
        i. Refer to procedures in Section 6.7.1.1.
        ii. Read and record standard information and measurement data on appropriate data sheet. The measured result of each standard must be within ±5% of the standard value to verify correct meter operation.
10. If data does not meet this criterion, the instrument must be recalibrated and the sample(s) reanalyzed. If unable to re-calibrate and re-analyze, the measured value(s) must be appropriately qualified when reported.

    NOTE: Hach recommends recalibration with formazin once every three months, or more often as experience dictates.

6.8 Hach Colorimeter DR890

The following procedures are to be used with the Hach DR/890 Colorimeter II. Procedures 6.8.1-6.8.3 are adapted from Hach DR/890 Colorimeter Procedures Manual. Refer to instrument manual for additional information.

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6.8.1 Ferrous Iron (0-3.00 mg/L)

Ferrous Iron (Fe^{2+}) must be analyzed immediately after collection to prevent oxidation to ferric iron.

1. Enter set program number for Fe^{2+} by pressing [PRGM] key and then entering '33' and [Enter].
2. Collect a representative sample and rinse sample cells several times with sample water.
3. To prepare sample blank, fill sample cell with 25 mL of sample. Wipe cell with lint-free tissue to remove moisture and fingerprints.
4. Insert sample blank into instrument by aligning the marking on the cell to the marking on the meter.
5. Cover cell with instrument cap, making sure cap fits tightly against the instrument and press the [ZERO] key.
6. Fill a second sample cell with 25 mL of sample. Add the contents of one Ferrous Iron Reagent Powder Pillow to the cell. Cap and invert to mix powder. This is the prepared sample.
7. Press [TIMER] and then [ENTER] to initiate the 3 minute waiting period required for the reagent to react.
8. Wipe cell with a lint-free tissue to remove moisture and fingerprints. Place prepared sample cell into instrument cell holder and align markings. Cover cell with instrument cap.
9. Once the 3 minute waiting period has expired, press [READ]. The result in mg/L is displayed.

6.8.2 Manganese (Mn) Low Range (0-0.700 mg/L)

1. Enter set program number for Mn LR by pressing [PRGM] key and then entering '43' and [ENTER].
2. The display will show mg/L, Mn and the ZERO icon.
3. Rinse all glassware with a 1:1 nitric acid solution and rinse with DI water.
4. Collect a representative sample and fill sample cell with 10 mL of DI water. This is the sample blank.
5. Fill a second cell with 10 m of sample. This is the prepared sample.
6. Add the contents of 1-Ascorbic Acid Powder Pillow to each cell and swirl to dissolve.
7. Add 15 drops of Alkaline-Cyanide Reagent to each cell and swirl to mix.
8. Add 21 drops of Pan Indicator Solution, 0.1%, to each sample cell and swirl to mix.
9. Press [TIMER] and then [ENTER] to initiate the 2 minute waiting period required for the reagent to react.
10. Wipe the sample blank cell with a lint-free tissue to remove moisture and fingerprints and place into instrument cell holder. Align markings and cover cell tightly with instrument cap.
11. Once the 2 minute waiting period has expired, press [ZERO].
12. Wipe prepared sample cell with a lint-free tissue to remove moisture and fingerprints and place into instrument cell holder. Align markings and cover cell tightly with instrument cap.
13. Press [READ]. Results are displayed in mg/L. Readings greater than 0.700 mg/L are reported as > 0.700 mg/L.

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6.8.3 Sulfide (0-0.70 mg/L); Methylene Blue Method

Sulfide (S_2^-) must be analyzed immediately after collection.

1. Enter set program number for S_2^- by pressing [PRGM] key and then entering '93' and [ENTER]. The display will show mg/L and then ZERO.
2. Collect a representative sample and pipet 25 mL of sample into sample cell. This is the prepared sample.
3. Fill a second cell with 25 mL of DI water. This is the sample blank.
4. Add 1.0 mL of Sulfide 1 Reagent to each cell and swirl to mix.
5. Add 1.0 mL of Sulfide 2 Reagent to each cell and swirl to mix.
6. Press [TIMER] and then [ENTER] to initiate the five minute waiting period required for the reagents to react.
7. Wipe sample blank with a lint-free tissue to remove moisture and fingerprints. Insert sample blank into instrument by aligning the marking on the cell to the marking on the meter.
8. Cover cell with instrument cap, making sure cap fits tightly against the instrument and press the [ZERO] key.
9. Once the 5 minute waiting period has expired, wipe prepared sample cell with a lint-free tissue to remove moisture and fingerprints. Place cell into instrument cell holder and align markings.
10. Cover cell with instrument cap and press [READ].
11. The result is displayed in mg/L.

6.9 Hach Digital Titrator: Method 8203 - Alkalinity (10-4000 mg/L as $CaCO_3$)

Analyze sample alkalinity as soon as possible after collection. The following procedure is adapted from Hach Digital Titrator Manual. Refer to manual for additional information.

1. Select the sample volume and sulfuric acid (H_2SO_4) titrator cartridge that corresponds to the expected alkalinity concentration as mg/L $CaCO_3$ in Table 6.9.

Table 6.9

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge	Digit Multiplier
10-4	100	0.1600	0.1
40-160	25	0.1600	0.4
100-400	100	1.600	1.0
200-800	50	1.600	2.0
500-2000	20	1.600	5.0
1000-4000	10	1.600	10.0

2. Assemble titration cartridge by inserting clean delivery tube into cartridge, and the cartridge into the titrator body.
3. Eject a few drops of titrant, wipe tip, and reset the counter to zero.
4. Collect a representative sample. Avoid agitating the sample.
5. Measure volume from Table 6.9 into 250 mL flask.
6. Dilute to 100 mL with DI water if necessary.
7. Add one Phenolphthalein Indicator Powder Pillow to the flask and swirl to mix.
8. If the solution turns pink:
   a. Titrate to a colorless end point by slowly turning dial to eject drops of sulfuric acid into the flask while gently swirling the flask.
   b. Record the number of digits required to turn the solution clear.

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c. Calculate: digits required (x) digit multiplier (=) mg/L as $CaCO_3$ Phenolphthalein alkalinity.
d. Proceed to Step 10.

9. If the solution remains colorless, the Phenolphthalein alkalinity is zero. Proceed to Step 10.
10. Add contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the flask. Swirl to mix.
11. Continue titrating solution with sulfuric acid (H_2SO_4) to a light pink (pH 4.5). Record the number of digits to achieve appropriate color.
12. Calculate: digits required (x) digit multiplier (=) mg/L as $CaCO_3$ Total Alkalinity.
13. If required by project, carbonate, bicarbonate, and hydroxide concentrations can be individually determined using the procedure in the Hach Digital Titrator instruction manual.

6.10 Hach Ferrous Iron Test Kit

The 1,10 phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron (Fe^{2+}) in the sample to form an orange color in proportion to the ferrous iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

To prevent contamination and obtain accurate measurements:

• Wash all tubes between tests with a non-abrasive detergent or a solvent such as isopropyl alcohol. Rinse with deionized water. Wipe dry with soft cloth; do not use paper towels or tissue on plastic tubes as this may scratch them.
• Rinse all viewing tubes with the sample water before testing.
• Use clippers to open plastic powder pillows.
• Reagent accuracy should be checked with each new lot of reagents.

To prepare ferrous iron stock solution:
Prepare solution immediately before use.

1. Prepare a ferrous iron stock solution (100 mg/L Fe)
2. Dissolve 0.702 grams of ferrous ammonium sulfate hexahydrate in one liter of deionized water.
3. Dilute 5.00 mL of this solution to 100 mL with deionized water to make a 5.0 mg/L standard solution.
4. Follow the ferrous iron test instructions using this solution instead of a water sample.

To conduct test:

1. Fill a viewing tube to the first (5-mL) line with sample water. This is the blank.
2. Place this tube in the top left opening of the color comparator.
3. Fill the measuring vial to the 25-mL mark with sample water and add the contents of one Ferrous Iron Reagent Powder Pillow to the vial. Swirl to mix. An orange color will develop if ferrous iron is present. Allow three minutes for full color development.
6. Fill the second viewing tube to the first (5-mL) with the prepared sample in the measurement vial.
7. Place the second tube in the top right opening of the color comparator.
8. Hold comparator up to a light source such as the sky, a window or a lamp. Look through the openings in front.
9. Rotate the color disc until the color matches in the two openings.
10. Read the mg/L ferrous iron in the scale window.

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6.11 YSI 556 MPS

The YSI 556 MPS is a handheld, waterproof instrument used for measuring dissolved oxygen, temperature, conductivity, and optional pH and ORP. Users are able to enable or disable each of the sensors. The sensors are enclosed in a heavy duty probe guard with attached sinking weight. Port plugs are installed in all ports where sensors are not installed to keep electrical connectors dry.

All sensors require periodic calibration to assure high performance. It is important to keep sensors moist without immersing them in liquid. Immersing in liquid can cause sensors to drift or result in a shorter lifetime. YSI recommends short term storage of all multi-parameter instruments be done by placing approximately 1/2 inch of tap water in the transport/calibration cup, and by placing the probe module with all of the sensors installed into the cup. The use of a moist sponge instead of a 1/2 inch of tap water is also acceptable, as long as its presence does not compromise the attachment of the cup to the probe module. Seal the transport/calibration cup to prevent evaporation. Refer to manufacturer instructions for sensor installation, removal, maintenance, and cleaning.

The YSI 556 MPS is compatible with YSI EcoWatch™ for Windows™ software. The instrument can store more than 49,000 data sets. If a circumstance arises in which the instrument does not respond to keypad entry, instrument function is restored by removing and then reapplying battery power.

6.11.1 Meter Operation

Note: Refer to specific field parameter section for additional procedures.

1. Turn meter on and off using the **On/Off** button in the upper left corner of the instrument keypad.
2. To adjust display contrast,
    a. increase contrast: press and hold down the backlight key in the upper right corner of keypad and press the up arrow.
    b. decrease contrast: press and hold down the backlight key in the upper right corner of keypad and press the down arrow.
3. To turn backlight on or off, press and release backlight key in the upper right corner of keypad. *NOTE:* Backlight turns off automatically after two minutes of non-use.
4. To enter letters and numbers, press the appropriate key repeatedly until the desired letter or number appears. Press **Enter** when entry is complete.
5. To turn sensors on or off,
    a. Press **On/Off** to display the run screen.
    b. Press **Escape** to display the main menu screen.
    c. Highlight the **Sensor** selection and press **Enter** to display the sensors enabled screen. *NOTE:* Temperature sensor cannot be disabled; most other sensors require temperature compensation for accurate readings. In addition, the conductivity sensor must be activated in order to obtain accurate dissolved oxygen mg/L readings.
        i. A black dot to the left of a sensor indicates that sensor is enabled.
        ii. An empty circle to the left of a sensor indicates that sensor is disabled.
    d. Highlight the sensor to change, then press **Enter** to enable or disable it.
    e. Repeat steps to enable/disable each sensor.
    f. Press **Escape** to return to the main menu screen.
6. Report Setup – to select sample parameters and units to display on screen.
    a. Press **On/Off** to display the run screen.
    b. Press **Escape** to display the main menu screen.
    c. Highlight the **Report** selection and press **Enter** to display the report setup screen.

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i. A black dot to the left of a parameter indicates that parameter is selected for display.
ii. An empty circle to the left of a parameter indicates that parameter will not be displayed.

d. Highlight the parameter to change, then press **Enter**. If the parameter is not displayed in the list, even after scrolling down past the bottom of the screen, the sensor used for that parameter is disabled. Refer to Step 5 for enabling/disabling sensors.
   i. If Temperature, Specific Conductivity, Conductivity, Resistance or Total Dissolved Solids was selected, the Units screen will appear.
      1) Use the arrow keys to select the units desired
      2) Press **Enter** to return to the report setup screen.
   ii. If Salinity, Dissolved Oxygen %, Dissolved Oxygen mg/L, pH, pH mv or ORP mv was selected, the selection dot will simply toggle on or off.
e. Repeat steps for each parameter to change.
f. Press **Escape** to return to the Main menu screen.

6.11.2 Managing Data

1. The File menu allows the user to view, upload, or delete sample data and calibration record files stored in the YSI 556 MPS.
2. With the instrument on, press **Escape** to display the main menu screen.
3. Highlight **File** and press **Enter** to display file screen.
4. To access the Directory,
   a. Highlight the **Directory** selection and press **Enter** to display file list screen. *NOTE:* Files are listed in the order in which they are logged to memory. Sample data files have the file extension **.dat**.
   b. Press **Enter** to display file details screen and press **Enter** to view the file data.
   c. Press **Escape** repeatedly to return to the main menu screen.
5. For View File,
   a. From the file screen, highlight the **View File** selection and press **Enter** to display list of files.
   b. Highlight an individual file and press **Enter**. The file data is displayed with the file name at the top of the display. *NOTE:* If no file name was specified, the data is stored under the default name NONAME1.dat.
   c. Use the arrow keys to scroll horizontally and/or vertically to view all the data.
   d. Press **Escape** repeatedly to return to the main menu screen.
6. To upload data using EcoWatch software,
   a. Disconnect the YSI Probe Module from the YSI 556 MPS instrument and connect the YSI 556 MPS to a serial (Comm) port of a computer via a PC Interface cable.
   b. Open EcoWatch on the computer and click the sonde/probe icon in the upper toolbar.
   c. Set the Comm port number to match the port the YSI 556 MPS is connected to.
   d. Go to the YSI 556 MPS file screen and highlight the **Upload to PC** selection.
   e. Press **Enter** to display the file list screen.
   f. Highlight the **.DAT** file to transfer and press **Enter.** Both the YSI 556 MPS and PC displays show the progress of the file transfer. *NOTE:* The file is transferred to folder C:\ECOWWIN\DATA, designated with a .DAT extension.
   g. After the file transfer is complete, close the terminal window and press **Escape** on the YSI 556 MPS repeatedly to return to the main menu screen.
7. To upload a Calibration Record (.glp) File,
   a. Connect the YSI to a computer using a Comm port and cable as described in Step 6 above.

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b. Open EcoWatch, click the sonde/probe icon in the upper toolbar, and set the Comm port number to match the port the YSI 556 MPS is connected to.
c. Go to the YSI 556 MPS file screen and highlight **Upload to PC**.
d. Press **Enter** to display the file list screen.
e. Highlight the calibration record file to transfer and press **Enter.** Upload file using one of three file format options:
    1) Binary
    2) Comma & "" Delimited
    3) ASCII Text
f. Choose an option and press **Enter**, both the YSI 556 and PC displays show the progress of the file transfer. The file is transferred to folder C:\ECOWWIN\DATA, designated with the appropriate file extension. After the file transfer is complete, close the terminal window.
g. Press **Escape** repeatedly to return to the main menu screen.
h. To view the Calibration Record data after upload, open the .txt file in a general text editor such as Wordpad or Notepad.

8. To view memory
    a. With the instrument on, press **Escape** to display the main menu screen.
    b. Highlight **File** and press **Enter** to display file screen.
    c. Highlight the **File memory** selection and press **Enter** to display the file bytes in use. The amount of free memory is listed in line 4 of the file bytes used screen. *NOTE:* If the amount of free memory is low, delete all files (after first uploading all data to a PC).
    d. Press **Escape** repeatedly to return to the main menu screen.
9. To Delete All Files

    NOTE: It is not possible to delete individual files in order to free up memory. The only way to free up memory is to delete ALL files present. Take care to transfer all files to a computer *before* deleting them.

    a. With the instrument on, press **Escape** to display the main menu screen.
    b. Highlight **File** and press **Enter** to display file screen.
    c. Highlight the **Delete all files** selection and press **Enter** to display the Delete all Files screen.
    d. Highlight the **Delete** selection and press the **Enter** key. The progress of file deletion is displayed in bar graph format.
    e. Press the **Escape** key repeatedly to return to the main menu screen.
10. Logging
    a. Press **On/Off** to display run screen.
    b. Press **Escape** to display main menu screen.
    c. Highlight **Logging setup** and press **Enter**.
    d. To set Logging Interval,
        i. Go to logging setup screen. Enter an interval between 1 second and 15 minutes using the keypad and press **Enter** to set the interval.
        ii. If no interval is specified, the instrument will use a default interval setting of one second.
    e. Press **Escape** repeatedly to return to the main menu screen.

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6.12 Hach Hydrolab Surveyor 4A

The Surveyor 4a can connect with Sonde Series 3 or Series 4. When connected to a Sonde, the Surveyor 4a will display the parameters included on the Sonde and allow for parameter setup options.

6.12.1 Battery Charging

The Surveyor 4a must be powered on with charger cable and a power adapter properly connected in order to charge. It is suggested to charge the instrument when the internal battery voltage (IBV) reaches 6.5 volts. A full charge is complete in three and a half hours.

NOTE: Charging is not complete when ETTC=0 or IB%=100. Charging is complete when Δt equals or exceeds three and a half hours, $-\Delta V$ equals or exceeds 60 mV, or ΔTCO equals or exceeds 15 °C.

1. Connect the Surveyor 4a charger cable to the Surveyor 4a and then attach the power adapter to the other end of the charger cable.
2. Turn on the Surveyor 4a, then insert the loose end of the power adapter to an outlet.
3. Press any key on the Surveyor 4a to continue, and the battery will automatically start to recharge.
   a. A low external power supply screen will display if external power source is providing insufficient voltage or poor connections where made.
   b. The Estimated Time to Charge (ETTC) and estimated current charge level of the internal battery back in percent will display (IB%) after the Welcome screen. IB% is an estimate. Disconnecting from charger before being prompted may severely reduce instrument operating time.
   c. Press the Suspend key to pause the recharge process, and press Resume to restart the recharge process. Press the All Stats key to provide more detailed information on the recharge process.
4. When the charge event is complete, the Surveyor 4a will display "Charging Complete! Disconnect Charger for Normal Operation. Press any key..."
5. Disconnect the Surveyor 4a charge cable. The screen will display the NoConn message.

6.12.2 Calibration

Calibration of the Surveyor 4a is not necessary, unless there is an option that requires calibration such as the internal barometer. The Surveyor 4a can be used to calibrate a Sonde multiprobe. Proper maintenance of all sensors should precede calibration. Refer to Sonde user guide for detailed sensor maintenance procedures.

1. Set the I/F mode to Series 3.
2. Press the Setup/Cal key.
3. Press the Calibrate key.
4. Press the Sonde key. A pop-up screen will displayed.
5. Highlight the parameter to be calibrated and press Select.
6. Follow the Surveyor 4a prompts using the appropriate calibration sections from the Sonde manual.

6.12.3 File Creation

The Surveyor 4a memory logging system comes in several options. The instrument logging capacity depends on the size of the memory installed. Memory is available in the following sizes: "Clipboard" memory which allows 12 manually stored scans (up to 300 manually

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entered readings), memory which allows approximately 120,000 readings, or memory which allows approximately 375,000 reading.

6.13 YSI Professional Plus (Pro Plus)

The ProPlus has a waterproof case and backlit display. It has a menu-based interface with user-selectable cable options. Press the keys to access the system, sensor, calibration, and file menus.

- Use arrows to navigate menus and highlight desired selection.
- Use Enter key to activate selection.
- Use left arrow to go back one screen.
- Press the Esc key to return to the run screen or to exit an alpha/numeric entry screen.

Enable Sensors:

Ensure appropriate cable sensor is in cable assembly by inserting the sensors into the ports and hand tightening. A sensor must be enabled in the Sensor menu for it to operate. Once a sensor is enabled, the desired units for the sensor must be selected in the Display menu to determine what will be displayed.

1. Press the Sensor (!) key.
2. Highlight Setup and press enter.
3. Highlight the parameter of interest and press enter.
4. Highlight Enabled and press enter to ensure a checkmark in the box. When enabling the ISE1 and ISE2 ports, select the correct sensor after enabling the port.
5. When Dissolved Oxygen is enabled, a submenu will open to select the sensor type and membrane type being used. Highlight Sensor Type or Membrane and press Enter to modify these settings.
6. Press the left arrow key to return to the previous screen or press Esc to return to the Run screen.

Select units:

1. Select the Sensor hot key on the keypad, highlight Display, and press enter.
2. Highlight the parameter to access and press the Enter. A submenu will open showing reporting units.
3. Make selections from the submenu, and then press the left arrow key to return to the Display menu or press Esc to return to the Run screen.

Calibration of Conductivity, pH, and ORP:

1. Press the Cal key.
2. Highlight the parameter to calibrate and press enter.
   a. For Conductivity, a second menu will offer the option of calibrating Specific Conductance, Conductivity, or Salinity. Calibrating one automatically calibrates the other two. An additional sub-menu requires selection of calibration units.
   b. For pH, auto-buffer recognition will determine which buffer the sensor is reading; allows calibration up to 6 points.
3. Place the correct amount of calibration standard into a clean, dry or pre-rinsed container.
4. Immerse the probe into the solution, making sure the sensor and thermistor are adequately immersed. Allow at least one minute for temperature to stabilize.
5. For any parameter, enter the calibration solution value by highlighting Calibration Value, pressing enter, and then using the alpha/numeric keypad to enter the known value.

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6. Once the calibration standard value is entered, highlight <<<ENTER>>> and press enter.
7. Wait for the readings to stabilize, highlight Accept Calibration and press enter to calibrate.
   a. For pH, continue with the next point by placing the probe in a second buffer and following the on-screen instructions or press Cal to complete the calibration.

Calibration of Dissolved Oxygen:

The Pro Plus offers four options for calibrating dissolved oxygen:

1. Air calibration method in % saturation.
2. Known solution concentration in mg/L
3. Known solution concentration in ppm
4. Zero calibration. If performing a zero calibration, a % or mg/L calibration must be performed following the zero calibration.

For both ease of use and accuracy, YSI recommends performing the following 1-point DO % calibration:

1. Moisten the sponge in the cal/transport sleeve with a small amount of water and install it on the probe. The cal/transport sleeve ensures venting to the atmosphere.
2. For dual port and Quatro cables, place a small amount of water (1/8 inch) in the calibration/transport cup and screw it on the probe. Disengage a thread or two to ensure atmospheric venting.
3. Make sure the DO and temperature sensors are not immersed in the water.
4. Turn the instrument on. If using a polarographic sensor, wait 10 minutes for the DO sensor to stabilize. Galvanic sensors do not require a warm up time.
5. Press the Cal key, highlight DO and press enter.
6. Highlight DO%, then press Enter.
7. Verify the barometric pressure and salinity displayed are accurate.
8. Once DO and temperature are stable, highlight Accept Calibration and press enter.

To measure:

1. To Log One Sample:
   a. Power on instrument; it will turn on in Run mode.
   b. Insert probe into sample; agitate probe until readings stabilize.
   c. Log One Sample is already highlighted in Run mode. Press enter to open a submenu of Sites and Folders.
   d. Highlight Sites or Folders and press enter to select the site or folder to log the sample. If necessary, use the keypad to create a new Site or Folder. *Note:* Option will not appear if Site List and Folder List are disabled in the system menu.
   e. Once the Site and/or Folder name is selected, highlight Log Now and press enter. The instrument will confirm the data point was logged successfully.
2. To log at a specific interval,
   a. Power on instrument; it will turn on in Run mode.
   b. Press System key. Use the arrow keys to highlight Logging and press enter.
   c. Enable Continuous Mode and adjust the time Interval if necessary.
   d. On the Run screen, the option to log will change from Log One Sample to Start Logging based on the time interval entered. During a continuous log, the Start Logging dialog box on the Run screen will change to Stop Logging.
   e. Insert probe into sample; agitate probe until readings stabilize.

To Download Data:

1. Connect the Communications Saddle to the back of the Pro Plus instrument.

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2. Connect ProPlus to PC with Data Manager and USB drivers by connecting the saddle to the USB port on the PC.
3. Open Data Manager on the PC and turn on the Pro Plus.
4. Click on the correct instrument in Data Manager under the Select Instrument heading. Once the correct instrument is highlighted, click the Retrieve Instrument Data tab and check Data, GLP, Site List, Configuration or Select All options to retrieve data.
5. Click Start.
6. After the file transfer is complete, the data is available for viewing, printing, and exporting from Data Manger and the data can be deleted from the Pro Plus if desired.
7. To delete data, press the File key and choose Delete Data.

7.0 FLOW DETERMINATION

7.1 Primary Measuring Devices

Open channel flow is characterized by a free flowing liquid surface. A hydraulic structure inserted into an open channel is used to measure a channel's discharge rate. A hydraulic structure's shape and dimension produces a flow with a known relationship between a channel's liquid level and discharge rate. The relationship derives a flow rate from a liquid level measurement taken at a known location. Two broad categories of primary measuring devices are weirs and flumes.

Refer to *Isco Open Channel Flow Measurement Handbook* for detailed information on primary device selection and design requirements.

7.1.1 Open Channel Weir

A weir is a dam built across an open channel restricting liquid flow through a specifically shaped notch. A weir is classified by the shape and size of its notch. Each weir has an associated equation to determine flow rate. Four common classifications of weirs are:

1. V-Notch (Triangular): accurate device to measure low flows.
2. Trapezoidal: less accurate than the v-notch or rectangular weir
3. Rectangular (contracted): able to measure high flows.
4. Rectangular (Suppressed): able to measure high flows, easier to construct than contracted. Width of weir crest must correspond to width of channel so use is restricted.

7.1.1.1 Weir Construction

1. Place adequate barriers around work area to protect equipment, vehicles, and people while working.
2. Open manhole cover to confirm weir installation is appropriate and possible.
3. Unload vehicle for easy access of equipment during weir build and installation.
4. Measure diameter of manhole opening.
5. Cut weir board length wise to fit in manhole opening.
6. Enter manhole only after safety procedures have been followed completely.
7. After entering manhole, measure length needed for weir board to fit across waste stream approximately 12 inches from effluent exit side of manhole.
8. Cut weir board to appropriate measurement of Step 7.
9. Clear debris and build-up from bottom and sides of channel. Place weir board flush between manhole walls and temporarily level weir board.
10. Scribe channel contour onto weir board with use of joystick; move joystick center to left and center to right to produce a pattern.

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11. Remove weir board from manhole and cut out bottom contour.
12. Place desired weir plate approximately 1 inch below the top edge on the upstream side of weir board. This will provide clearance between the weir plate and plywood notches allowing discharge to pass through v-notch without resistance from the plywood edge.
13. Trace weir plate and cut out notch.
14. Extend weir plate above plywood notch approximately 1 inch and secure with nails.
15. Lower weir down into manhole to measure T-braces.
16. Measure from one end of weir board across stream to other manhole wall.
17. Cut 2 x 4 from above measurement and install.
18. Measure from other end of weir to center of first T-brace; add about a half-inch if measured directly from manhole wall to give added strength when installing.
19. Refer to Section 7.1.1.2 for weir installation.

7.1.1.2 Weir Installation

1. Place adequate barriers around work area to protect equipment, vehicles, and people while working.
2. Open manhole cover to confirm weir installation is appropriate and possible.
3. Unload vehicle for easy access of equipment during weir build and installation.
4. Enter manhole only after safety procedures have been followed completely.
5. After entering manhole, clear debris and build-up from bottom and sides of channel.
6. Place weir board flush between manhole walls, adjusting as necessary to obtain the best fit between board and channel bottom.
7. Secure weir in place using T-braces. If necessary, use hammer to secure.
8. Seal seams on the upstream side of weir where channel and weir touch using a sealing compound.
9. Attach stainless steel bubbler tubing to the upstream side of weir using a clamp. Place at a sufficient distance from weir notch to prevent solid accumulation.
10. Once weir has filled and discharge is flowing through notch, set head measurement. Refer to Section 7.1.1.3.
11. Install required monitoring equipment.

7.1.1.3 Weir Head Measurement Procedure

All weir head measurements are taken using a level and a ruler. A single measurement can be taken when a level and ruler are incorporated onto a 90° carpenters square. The short leg of the device is placed on the crest of the weir plate with the ruler upstream. A head measurement is read directly off the fixed ruler when level.

Use the following steps to take measurements with an engineer's rule and standard level. Refer to diagrams in *Isco Open Channel Flow Measurement Handbook* for measurement locations.

1. Anchor the level from a fixed object, such as the weir or T-brace, so it is level and does not move during measurements.
2. Take measurements upstream of weir at a distance approximately 3 to 4 times the maximum head expected over the weir
   a. Measure distance from weir plate crest to level.
   b. Measure upstream water surface to level.
3. Subtract the second measurement from the first to determine the height of water

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passing over notch.

7.1.2 Weir Box

A weir box is a prefabricated structure providing specific upstream channel dimensions. A weir box is generally made from plastic or wood. Water flows into a weir box and over weir configuration to measure flow. A baffle is often used at the inflow end to slow the velocity of incoming water.

7.1.2.1 Weir Box Installation

1. Special plumbing may be required to accommodate weir box prior to installation.
2. Accommodate discharge pipe by erecting a platform for weir box to rest. In some instances, the discharge pipe elbow may be shortened or extended to accommodate weir box.
3. Position weir box so incoming water is deposited directly into back of weir box behind baffle, if applicable.
4. Level weir box using shims when necessary.
5. Install secondary measuring device. Refer to Section 7.2.

7.1.2.2 Weir Box Head Measurement Procedure

Refer to Section 7.1.1.3.

7.1.3 Flumes

A flume is a specially shaped open channel flow section restricting channel area and/or slope. This defined restriction results in an increased velocity and/or change in liquid level flowing through flume. The constricted flume throat produces a liquid level relating to discharge. One or two measurements (head readings) determine a channel's flow rate.

Flumes can be built of wood, concrete, galvanized sheet metal, or fiberglass. Large flumes are usually installed permanently at a site, while smaller prefabricated flumes may be temporarily installed in a channel.

A flume can measure a higher flow rate compared to a similar sized weir. A flume is better suited to measuring flows containing solids, as deposits are limited due to higher velocity flow. Parshall and Palmer-Bowlus flumes are the two most common types encountered in the field.

The **Parshall Flume** is most widely used for permanent installations. The advantages include a self-cleaning effect and relatively low head loss. The constricted throat of the flume produces a differential head related to discharge. A level converging section followed by a downward sloping throat gives the Parshall flume its ability to withstand relatively high degrees of submergence without affecting flow rate.

The **Palmer-Bowlus flume** is useful for temporary installation to provide flow data for determining equipment requirements. It can also be permanently installed by embedding flume in concrete. Most Palmer-Bowlus flumes are trapezoidal in configuration; others are rectangular, with various slopes, base, widths, and heights.

Refer to *Isco Open Channel Flow Measurement Handbook* for detailed information on

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primary device selection and design requirements.

7.1.3.1 Portable Flume Installation

1. Apply sealing compound to outside ends of flume.
2. Press flume into a channel.
3. Use level to verify position both longitudinally and perpendicular. Make adjustments as needed.
4. Hold and seal flume in place with sand bags and/or additional sealing compound.
5. Install secondary measuring device. Refer to Section 7.2.

7.1.3.2 Flume Head Measurement

Refer to diagrams in *Isco Open Channel Flow Measurement Handbook* for measurement placement.

Parshall flume head measurements can be obtained by a single head measurement taken directly downstream of the point on the flume where the upward converging slope intersects the level floor, except for conditions of submerged flow. Reference the *Isco Open Channel Flow Measurement Handbook* for submerged condition flows.

Palmer-Bowlus flume head measurements are best taken using a tool that incorporates a level and ruler onto a 90° carpenters square. Place the short leg of the tool so it rests on the flat throat section of the flume with the fixed ruler upstream. Measure water level at a distance of one half the conduit diameter upstream of flume.

An engineer's ruler and level can also be used. Anchor the level at a distance one half the conduit diameter upstream in the throat section. Make certain it is level and does not move during measurements.

1. Measure distance from throat floor to level (a).
2. Measure distance from bottom of channel to level (b).
3. Measure distance from bottom of channel to water surface at the same point measurement (b) was taken (c).
4. Head height = (c - (b – a))

7.1.4 Isco Flow Metering Inserts

Isco Flow Metering Inserts measure and record flow in round pipes with diameters from 6 to 12 inches when used with Isco 730 or 4230 bubbler flow meters. The assembly consists of a round, metal insert, inflatable rubber collar, foot-powered air pump, and a multi-sectional pole (six-2½ foot sections). The Insert is installed from ground level with an attachable, interlocking pole that can reach a depth of 16 feet. A 60° V-notch weir plate can be attached to the insert to obtain a higher accuracy at a lower flow rate. Refer to Isco Flow Metering Insert Installation and Operation Guide and the applicable flow meter's manual for typical flow measurement accuracy of a specific flow metering assembly.

7.1.4.1 Flow Metering Assembly Preparation (before taking into the field)

1. Inspect the flow metering assembly.
   a. Ensure all parts of system are present and cleaned.
   b. Examine the bubbler and air pressure tubing for cuts or contamination.

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c. Examine rubber collar for cuts or contamination.

Note: Discharge that contains chemical agents capable of attacking aluminum (strong alkalies) or the inflated rubber collar (certain organic solvents) should be avoided.

2. Set the flow meter's bubble rate.
   a. With the insert upright in water deep enough to cover the bubble line outlet by a few inches, adjust the bubble rate to 1-2 bubbles per second.
3. Set the flow meter's zero level
   a. Remove the insert from the water, and purge the flow meter. Remove all traces of water from line as any remaining will affect the accuracy of the zero setting.
   b. With the line dry and the insert sitting in open air, adjust the level reading on the flow meter's display to zero. Refer to Section 7.2 Secondary Flow Device: Isco 4230 for details on flow meter programming procedures.

Note: Do not set the zero point before setting the bubble rate; the bubble rate affects the zero level and must be set first.

4. Select the insert to use for monitoring site.
   a. Select insert size. Inserts are designed to be used in pipes with inside diameters of 6", 8", 10", or 12". The actual outside diameter of each insert is approximately 90% of the pipe diameter. Typically, the insert size matches the inside pipe diameter of the monitoring location, although due to undersized pipes or pipes that are out-of-round, some monitoring sites can require a smaller size.
   b. Select round or V-notch flow control section. The round orifice flow control section is used with higher flow rates and the V-notch section with lower flow rates. In general, if the pipe is flowing more than 1/4 to 1/3 full, use the round orifice flow control section. If flowing less than this, use the V-notch flow control section. For details on flow rate ranges for various sized inserts, refer to the table in the Isco Flow Metering Insert Installation and Operation Guide.
      i. To attach the V-notch weir plate to the metering insert,
         1) Loosen the two wing nuts and line up the hole in the bottom of the V-notch weir plate above the bubbler tube on the front of the metering insert and below the bottom of the orifice.
         2) Reach through the insert, and grasp the retaining plate on the back of the weir plate, while slipping the retaining plate over the back of the round orifice plate in the metering insert.
         3) Slide the weir plate down along the face of the orifice towards the bubbler tube.
         4) Slip the hole in the weir plate over the insert's bubbler tube; make sure the studs on the back of the weir plate fall inside the orifice opening.
         5) Tighten the two wing nuts while holding the weir plate in place, tighten the two wing nuts.
      ii. To remove the V-notch weir plate from the metering insert,
         1) Loosen the two captive wing nuts on the back side of the weir plate (inside the metering insert).
         2) Pull the weir plate up and out of the metering insert.

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7.1.4.2 Installation of Flow Metering Insert

CAUTION: Be aware of surroundings. Make sure pole does not contact power lines or other property when extended.

1. Snap together enough interlocking pole sections to reach the pipe. The total length should be somewhat greater than the distance from the top of the manhole to the bottom of the invert.
2. Assemble the three separate pieces of the right-angle tube with the end closest to the bend being snapped to the flow insert.
3. Attach the air pump to the pole assembly by securing the valve stem on the inflation hose to the chuck on the air pump. Coil any excess hose near the top of the pole to keep it from interfering with the flow metering assembly.
4. Using the pole from ground level, lower the flow metering assembly into the manhole. It is recommended that the top of the pole assembly be waist-to chest-high, when the metering insert is installed.
5. Position the insert into the upstream pipe by sliding it into the pipe so that the back end of the insert is totally surrounded by a full section of the pipe. It may be necessary to rock the insert from side-to-side or top-to-bottom, while pushing the insert into the pipe.
6. Make sure the pole is centered over the pipe and is as close to straight up-and-down as possible. The center line of the V-notch weir or round opening should be straight up-and-down and the upstream face of the metering insert should be perpendicular to the oncoming flow.
7. Seal and secure the insert into place by using the foot-powered pump to inflate the rubber collar. Monitor the pressure gauge attached to the pump to obtain the proper pressure. Inflate 6" and 8" inserts to a pressure of 15 to 20 psi. Inflate 10" and 12" inserts to a pressure of 10 to 15 psi. All flow from the pipe should be channeled through the insert.
8. Attach the bubbler line from the assembly to the flow meter and program flow meter to record flow. Refer to Section 7.2 Secondary Flow Device: Isco 4230 for details on flow meter programming procedures.
9. For multiple monitoring days, the pressure of the rubber tube is checked to ensure proper inflation and the insert is observed for large obstructions.

Note: When the air pump is disconnected from the hoses, there is a slight loss of pressure each time the pump is re-connected to the air hose; this is normal as long as it does not exceed a pound.

7.1.4.3 Removal of Flow Metering Insert

1. Disconnect bubbler line from flow meter.
2. Deflate the rubber collar by pressing the valve stem inside the metal fitting on the end of the insert's air inflation hose.
3. Use the pole to release and lift the metering insert to ground level. Disconnect the pole sections one at a time as the pole is drawn out of the manhole by pressing the snaps and pulling the sections apart.
4. Check the insert for significant debris. If there are any, the accuracy of the flow readings would be doubtful and should be rechecked.
5. Remove and disassemble the insert, right-angle tube, and pole assembly.
6. Either onsite or upon return to Pace, wash the flow metering assembly with water and a brush.
7. If used the V-notch weir plate should be removed from the insert, washed, dried, and then reassembled.

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7.1.4.4 Repairs

Replacement inflatable collars and clamps are available from Isco. Refer to the Isco Flow Metering Insert Installation and Operation Guide for details.

7.2 Secondary Flow Device: Isco 4230

7.2.1 Preparation

1. Refer to Section 3.1.2 for flow meter operation and partitioning instruction.
2. Check desiccant and change if necessary.
3. Obtain accessories for flow meter installation including tubing with stainless steel end section, clamp, printer paper, and a cable handle for hanging flow meter.

7.2.2 Installation

1. Place meter in secure location where it will not be submerged.
2. For weirs, attach stainless steel end section of bubble line to upstream side of weir using a clamp. Place at sufficient distance from weir notch to prevent solid accumulation on line.
3. For flumes, place bubble line tubing near the upstream end of flume in middle of waste stream. The end should be flush to flume to prevent solid buildup.
4. Adjust bubble rate to about one bubble per second. To adjust bubble rate, use knob on side near bottom of flow meter.
5. To set head reading, use a weir level or an engineer's rule to measure water level. Refer to Section 7.1.1.3, Section 7.1.3.2, or the *Isco Open Channel Flow Measurement Handbook* for specific primary device procedures.
6. Set level in Step 3 of program under PARAMTERS TO ADJUST. Select LEVEL and ENTER.
7. Read the actual level and quickly enter level into flow meter.
8. Compare actual level with level displayed on meter. If necessary, repeat process until actual level and displayed level concur.
9. Continue with flow meter setup by pressing the ENTER key. Step through and set options as needed. Typical settings are listed below. Refer to flow meter instruction manual for additional information.

    a. Reset totalizer: *YES*
    b. Enable Totalizer: *ENTER*
    c. Reset Sampler Enable Totalizer: *NO*
    d. Sampler Pacing: *DISABLE*
    e. Sampler Enable Mode: *DISABLE*
    f. Plotter Speed: *OFF*
    g. Report Generator: *ON*
    h. Report Duration to be in: *HOURS*
    i. Report A Duration: *typically 1 hour*
    j. Print the first report at: *sampling start time*
    k. Report Generator B: *OFF*
    l. Print Flow History: *NO*
    m. Clear History: *NO*

10. Record head measurements on the appropriate data sheet.

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7.2.3 Removal

1. Measure head reading for primary device following appropriate procedure. Refer to Sections 7.1.1.3 and 7.1.3.2 for measurement instruction or refer to the *Isco Open Channel Flow Measurement Handbook.*
2. Compare measured level and meter display level. Do not change the level on the flow meter. Make several checks to verify measured versus actual levels. Discuss course of action with project administrator if large discrepancies are observed.
3. Unclamp bubble tubing and remove flow meter from manhole.
4. Remove primary device when necessary.
5. Record head measurements on the appropriate data sheet.
6. Remove printout and/or download data for hourly proportional compositing when applicable.
7. Clean flow meter recharge battery, and return unit to proper storage area.

7.3 Secondary Flow Device: Isco 4210 Ultrasonic Flow Meter

7.3.1 Preparation

1. Refer to Section 3.1.2 for flow meter operation and partitioning instruction.
2. Make sure an unsaturated desiccant canister is securely installed in the case lid; change if necessary. Do not operate the flow meter with a saturated desiccant canister or with the door left open as dust and moisture will damage the unit.
3. Obtain accessories for flow meter installation including tubing with stainless steel end section, clamp, printer paper, and a cable handle for hanging flow meter.

7.3.2 On-Site Installation

1. Place flow meter in secure, level location where it will not be submerged.
2. Secure the ultrasonic level sensor as close to the maximum expected level as possible leaving a distance of at least 12 inches between the sensor face and the maximum expected level. This minimum distance (12 inches/30.5 cm) is referred to as the "dead band" between which no measurements are taken by the sensor.
3. When securing the ultrasonic level sensor onsite:
    a. Prevent the ultrasonic level sensor from being submersed for prolonged periods of time. Temporary submersion in the flow stream should not cause any harm to the unit but prolonged submersion causes inaccurate readings as:
        i. A submersed sensor does not read a level.
        ii. Extend submersion may cause the surface of the sensor to become coated with solid matter reducing its accuracy or causing it to malfunction.
    b. Secure the sensor so it is stable and cannot move or swing. This is imperative to maintain calibration between the level sensor and the channel.
    c. Correctly place the ultrasonic level sensor over center of the flow stream according to the primary device's intended location for head (level) measurement.
        i. For weir, refer to Section 7.1.1.3 for head measurement location and instruction.
        ii. For Parshall flume, refer to Section 7.1.3.2 for head measurement location and instruction.
        iii. For Palmer-Bowlus flume, refer to Section 7.1.3.2 for head measurement location and instruction.
        iv. For other flow devices or gravity flow equation, determination of the location of the ultrasonic level sensor must be done from the hydraulic characteristics of the site and the method of level-to-flow rate conversion used. Refer to the

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4210 Flow Meter Section 3 Installation Isco Open Channel Flow Measurement Handbook or to information provided by the manufacturer of the primary device for more details about the location of the head measuring point.

d. Use a circular bubble level to align the sensor vertically. The aim is to have the sensor absolutely level. Misalignment may result in erratic or erroneous level readings, resulting from the echo bouncing off walls of channel.
e. Protect the ultrasonic level sensor from wind, vibrations, and sunlight. Both conditions add error to the level calculations.

4. Once the meter and sensor are properly secured, calibrate the sensor level. Be aware that subsequent movement of the level sensor (i.e., bumping it when entering or leaving the manhole) can introduce serious error into the measurements.
5. To set head reading, select Step 3, Port to Adjust, of program. Select LEVEL and ENTER. Measure the level of the flow stream as accurately as possible as this level setting determines the accuracy of all subsequent level and flow rate measurements made by the meter.
6. Read the actual level and quickly enter level into flow meter.
7. Compare actual level with level displayed on meter. If necessary, repeat process until actual level and displayed level concur.
8. Signal Strength
   a. The flow meter checks the measured level for validity. If it cannot obtain a valid reading after one minute the level reading will drop to zero with an asterisk (*) to indicate there is an error.
   b. The reflection cone beneath the ultrasonic level sensor can be fine-tune:
      i. Go to Step 1, select SETUP.
      ii. Select MEASUREMENT, then VARIABLE BLANKING.
      iii. Measure the face of the installed ultrasonic level sensor to the surface of the channel just above the maximum expected liquid level. This value tells the flow meter to ignore any echo reflected from this distance or less. Proper selection of a value here will ensure echoes selected by the flow meter as valid will come from the surface of the flow stream, not the walls or sides of the channel.
   c. The signal strength of the ultrasonic level sensor can be checked to confirm position above flow stream:

      Note: Objects or foam on the surface of the flow stream can absorb or weaken the ultrasonic pulses. If

      i. Go to Step 1, select SETUP.
      ii. Select STATUS menu. The flow meter will display the number, software revision, etc. Press [Enter] again.
      iii. A number will display indicating the strength of the ultrasonic return echo. Typical values range from 10 to 90.
         1. 1 indicates a very weak return echo.
         2. 100 indicates a very strong return echo.
      iv. Adjust the ultrasonic level sensor for the highest number possible.
9. When satisfied that the flow stream has been measured accurately, change the level reading on the flow meter to show this reading.
10. Once the proper value is displayed, press [Enter/Program Step] key.
11. Continue with flow meter setup by pressing the ENTER key. Step through and set options as needed. Typical settings are listed below. Refer to flow meter instruction manual for additional information.

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7.3.3 Removal

1. Measure head reading for primary device following appropriate procedure. Refer to Sections 7.1.1.3 and 7.1.3.2 for measurement instruction or refer to the *Isco Open Channel Flow Measurement Handbook.*
2. Compare measured level and meter display level. Do not change the level on the flow meter. Make several checks to verify measured versus actual levels. Discuss course of action with project administrator if large discrepancies are observed.
3. Unsecure ultrasonic flow sensor and remove flow meter from manhole.
4. Remove primary device when necessary.
5. Record head measurements on the appropriate data sheet.
6. Remove printout and/or download data for hourly proportional compositing when applicable.
7. Clean flow meter, recharge battery, and return unit to proper storage area.

7.4 Secondary Flow Device: Panametrics TransPort® Flow Meter

The Panametrics TransPort® Model PT868 is a portable liquid flow meter used to measure closed pipe flow. It is compatible with a variety of pipe diameters and materials. Data can be displayed in real time or stored in the internal data logger.

Four function keys [F1], [F2], [F3], and [F4] are used to select the corresponding functions or parameters displayed on the bottom of the screen. Four arrow keys are used for scrolling options. A shift key accesses special task keys and the letters indicated above each key.

Additional information can be found in the *TransPort® Model PT868 User's Manual* and support video. Appendix references for Section 7.4 refer to those in *TransPort® Model PT868 User's Manual.*

I. Programming:

1. Turn on TransPort unit by pressing [ON]. The unit will go through a self-check and eventually display a measurement screen. Press [PROGram].
2. Press [F1] for SYSTM program menu.
3. ENERGY OPTION - Press [F1] OFF
4. SITE MESSAGE - Up to 21 characters, using the alphanumeric keys. Press [ENT].
5. SYSTEM UNITS - [F1] ENG for ENGLISH or [F2] METRC for Metric.
6. STOP WATCH TOTALIZER – [F1] for AUTO
7. VOLUMETRIC UNITS – Choose the desired screen option using the [←] [→] keys to preview options. GAL/M used for most applications. Press the corresponding [F] key.
8. TOTALIZER UNITS - Choose the desired screen option using the [←] [→] keys to preview options. GAL used for most applications. Press the corresponding [F] key.
9. DATE – Accept date [F1] OK or [F2] EDIT.
10. TIME - Accept time [F1] OK or [F2] EDIT.

II. Pipe Parameters:

1. [F2] for PIPE.
2. TRANSDUCER NUMBER – Enter number engraved on the transducer selected for your application and press [ENTer]
3. PIPE MATERIAL - Choose the desired screen option using the [←] [→] keys to preview options. Press the corresponding [F] key. ***Note:*** enter sound speed found in Appendix B if selecting OTHER.

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4. PIPE OD – Enter pipe outside diameter and press [ENTer]. Refer to Appendix B or the pipe material reference insert.
5. PIPE WALL – Enter pipe wall thickness and press [ENTer]. Refer to Appendix B or the pipe material reference insert.
6. LINING – Typical piping is not lined. Select [F1] for NO unless confirmed.
7. FLUID TYPE - Choose the desired screen option using the [←] [→] keys to preview options. Press the corresponding [F] key. Note: if you select OTHER, you must enter the sound speed found in Appendix B.
8. WATER TEMPERATURE – Measure fluid temperature if possible, otherwise enter closest estimate for average temperature and press [ENTer].
9. REYNOLDS CORRECTION – For clamp on applications (diametrical path) press [F2] for ON. The TransPort unit will automatically calculate the kinematic viscosity for water (example: 14.07 ft^2/s X 10^{-6}). Press [ENTer].
10. CALIBRATION FACTOR – Set to 1.00 for typical water applications. Press [ENTer].
11. NUMBER OF TRAVERSES – Press [F2] for 2(V). This is the typical application where the transducers are clamped onto the outside of the pipe with a single mounting bracket.
12. TRANSDUCER SPACING – The TransPort unit will automatically calculate the distance between the two transducers. Record this value for future reference or set the transducer space using the scale on the mounting bracket at this time. Press [ENTer].
13. SAVE – To save the site data, press [F4] to SAVE, and enter in a five-character site name and [ENTer].
14. EXIT - Press [EXIT]. Real time data is now displayed for the pipe and location configured.

III. Mounting Clamp-On Transducer:

Optimal transducer location is an area with approximately 10 pipe diameters of straight, undisturbed pipe upstream and approximately 5 pipe diameters of straight undisturbed pipe downstream. Choose a section of pipe having minimal corrosion or pitting. Clean pipe surface and remove corrosion with sandpaper. A vertical section of pipe is best to assure a full pipe. If mounting transducer to a horizontal pipe, orient transducer on the side of the pipe to avoid interferences with accumulated gasses on top or sediment on the bottom.

1. Use small transducers (#24) for pipe diameters 0.5 to 2.0 inches in diameter. Use large transducers (#30) for pipe diameters greater than 2.0 inches.
2. SET THE TRANSDUCER SPACING – Using the transducer spacing calculated by the TransPort, align the transducers.
3. Apply a generous amount of couplant to the face of the transducer.
4. Using the Universal Mounting Brackets, strap and tighten transducers to the pipe surface (after the site-specific spacing is set), making sure to confirm the cables "upstream" and "down stream" are orientated correctly.

IV. Logging Data:

Once the SYSTEM and PIPE settings are programmed and saved SAVE, data logging can begin.

The TransPort logs data on "pages". There are 120 pages available, and each page is capable of storing 120 records, with three parameters per record. To check the memory, press [EXIT], wait for reading display screen), then [LOG], [F2] for MEM. Previous logs can be cleared to free up pages by pressing [EXIT] (wait for reading display screen), [CLeaR], then [F3] for LOG, and then select the log to clear.

1. Press [PROGram] and select RECLL from the PROGram menu by using the [→] and appropriate [F] key. Select the site file using the appropriate [F] key, then [EXIT]. Once you

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have the correct site file shown on the upper left corner of the display screen, proceed to next step. The screen should be displaying readings.

2. NAME - Press [LOG] and select [F1] for PARAM. Enter a name to create a new log, or use the [←] [→] keys to select an existing log. Then [ENTer].
3. LOG MESSAGE – Type a site message up to 21 characters, then [ENTer]
4. LOG UNITS - use the [←] [→] keys to select desired log units. Note: [F2] VOLUM is typical.
5. START TIME – enter start time, taking into consideration process activities and/or coinciding sample collection with automatic samplers. [ENTer] (if you select NOW , you will automatically go to Step 7.)
6. START DATE – Enter start date [ENTer]
7. END TIME – Enter end time [ENTer]
8. END DATE – Enter end date [ENTer]
9. TIME INCREMENT – Select time interval to suite project needs. Set to 60 minutes for hourly composite sampling [ENTER].
10. The log is now setup. Press [EXIT] and the TransPort unit will start to log at the time specified. An * will be blinking in the upper black section of the screen if the unit is logging.

V. Printing Log Data:

The following steps are compatible with the Seiko DPU-411 Thermal Printer.

1. CONNECT – Connect the printer to the RS232 port on the Transport unit using the cable provided in the kit.
2. ACCESS PRINTER MENU – To access the printer menu, press [PRiNT]
3. SELECT – Press [F2] to select LOG
4. FORMAT – Press [F1] to print numeric data NUM or [F2] to plot data on a graph PLOT. (Numeric is typical).
5. SELECT LOG – Using the [←] [→] keys, select the desired log using the corresponding [F] key.
6. PRINTING – The selected log will automatically start printing if you chose NUM in Step 4. If you chose PLOT , you will be prompted to enter additional X and Y axis range information.

7.5 Timed – Gravimetric and Volumetric Flow Measurement

(Also known as "bucket and stopwatch" method)

Timed Gravimetric: the entire contents of a stream are collected in some type of container for a set length of time. The weight of the fluid is determined and the flow rate calculated. For example, if the weight of water collected over one minute is 12.51 pounds:

12.51 pound/minute (divided by) 8.34 pound/gallon = 1.5 gallons/minute
Note: Remember to subtract the weight of the container.

If flow rate is uniform over collection period, the result is a true flow rate; if flow rate is not uniform over collection period, the result is an average flow rate.

Timed Volumetric: the entire contents of a stream are collected into a container with volumetric measurement markings for a set length of time. For example, if the volume of water collected over one minute was 9.5 liters:

9.5 liters/minute (divided by) 3.8 liters/gallon = 2.5 gallons/minute

If flow rate is uniform over collection period, the result is a true flow rate; if flow rate is not uniform over collection period, the result is an average flow rate.

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7.6 Fixed or In-line Plant Meters

In-line, or fixed, plant meters are commonly used to determine daily flows and hourly flows for sample compositing. Meters required for flow determinations are identified on a project's Client Data Sheet or Specific Site Information. All incoming meters as well as deductible flows such as sprinklers, cooling, evaporation, and product usage are recorded. Clarify meter identity and units onsite. Numeric and analog meters are the two most common meter types encountered in the field.

I. Numeric (digital) Meters

1. Record meter ID and units.
2. Record all numbers indicated on register.
    a. Include zeros before first registered number.
    b. Record to the nearest unit of measure (gallon or cubic feet) when possible. If present, record dial/needle hands for fixed zeros unless discharge volume is very high. Identify dial/needle digits with line above numbers.

II. Analog (dial or clock-type) Meters

Analog meters (sometime referred to as dial or clock-type) have multiple dials. Use caution when reading analog meters. The numbers on the dials are backwards on every other dial due to the rotation sequence of gears. A number is written under each dial indicating what unit-place the dial represents. For example, if the written number is "10", then one complete revolution would be ten and the number recorded from that dial would not have a fixed zero. If the number is "100", one revolution would be 100, and the number from that dial would have one fixed zero. (If the dial hand is on the "7", it would represent 70.)

1. Record meter ID and units.
2. Begin reading the smallest increment dial, typically "one" or "10" gallons or cubic feet.
3. If the hand is between two numbers, use the smallest number
4. If the hand is near a number and it is difficult to determine if it is before or after the number, look at the preceding dial. If the preceding number is large, then the dial hand in question has not yet passed the number. If the preceding number is small, the dial hand in question is past the number.

7.7 Meter-Master Model 100EL Flow Recorder

The METER-MASTER MODEL 100EL Flow Recorder is a battery-powered, portable flow recording instrument that converts a water meter's magnetic drive signal to a digital output that is stored for later downloading. It is compatible with almost all water meters. Meter-Master has data storage capacity of 256K with interval settings of 5, 10, 30, and 60 seconds. The meter can hold up to 20 individual records. Recording stops when memory is used up; the meter will not overwrite data. The Meter-Master is stored connected to the charger. If this is not possible, recharge the unit at least once every two months.

1. Confirm battery charge and available memory before taking MM into the field.
    a. Verify the rocker switch is in the OFF position.
    b. Connect the MM100 to the computer.
    c. Enter the Model 100 Program by selecting the Meter-Master program group and then selecting the Model 100 Program.
    d. Check that "Flow Recorder is connected" appears at the upper left of the Main Screen.
    e. Click "Change MM100 Settings"

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i. Select the desired meter make, model, size, and unit of measure from the options presented on the pull-down menus. Click on item to display a pulldown menu; click on option to select.
ii. Select the desired Data Storage Interval from the options listed in the pulldown menu.
iii. A 10 second interval provides adequate resolution in most cases and 15 days of recording time.
iv. Click "OK" to save the new settings. Click on "Cancel" to keep the prior settings.

f. Click "Clear MM100 Data" to clear MM100 memory of prior records. The MM100 Interface Screen displays the remaining battery charge and data storage capacity.

Note: Changing the Model 100EL settings also automatically sets the Meter-Master date/time to the computer date/time.

2. Begin a Test
   a. Attach the sensor cable to the MM100.
      i. The sensor receptacle is in the middle one and two keyways align the cable connector with the receptacle.
      ii. Align the keyways before threading the cable into place.
      iii. Never push or press a connector into a receptacle.
   b. Place sensor flat against the meter casing or register with the Velcro on the outside, away from the meter. It is not necessary for the sensor to touch the meter face.
   c. Place sensor farthest from any nearby equipment that can generate electromagnetic interference (EMI) (e.g., motors, burglar alarms, generators, and other meters).
   d. The best location for the sensor varies depending on the type of meter.
      i. Typically, the sensor is placed on a side of the meter register that is not over the pipe.
      ii. When placed on the side of the register, the sensor cable should extend either straight down or up.
      iii. When placed on the side of the meter body, the sensor cable should extend in a horizontal direction.
   e. Once a good sensor location has been identified, secure the sensor to the meter by tightening the Velcro straps as much as possible to prevent sensor movement. Tape may also be used.
   f. Always wait 10 seconds after moving the sensor to allow the electronics to settle.
   g. Press and release the side of the rocker switch with the LED to turn on MM100 recording. The switch will automatically return to its middle position. The LED will light for 3 seconds indicating the recording session has started.
   h. After the 3-second signal, the light will flash 30 times in synchronization with the meter's dial movement.
      i. If the sensor signal does not flash regularly or in proportion to the flow rate, try the sensor in other location.
      ii. Alternating long-short flashes for 6 seconds indicates that the MM100 has powered down due to low battery.
      iii. If the LED flashes extremely quickly or stays solid for a brief period when no flow is occurring, this can indicate that the meter is too close to other electromagnetic noise sources preventing an accurate record of flow.
   i. Record on the field data sheet,
      i. The meter reading.
      Ensure all meter readings include all digits to the decimal place, including the exact position of the sweep hand on the dial face. A digit must be read for each rotating dial position, black and white, and each painted-on zero. Zeros painted on a dial face represent additional digits before the decimal place.
      ii. The meter reading start time and date.
      iii. The meter type, make, model, units and size.
3. To end recording,
   a. Push the ON side of the switch again to verify that the sensor is still recording accurately.

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b. Record on the field data sheet,
   i. The meter reading.
   ii. The meter reading end time and date.
c. Push the rocker switch to OFF position to end the recording session.
d. Remove the MM100 from the meter.

4. Real Time Display
   a. A notebook computer can be used to test the sensor location while in the field. Connect the MM100 to the notebook computer.
   b. Choose "Realtime Display" option at the Main Screen.
   c. Select a method for calculating the rate from the two options shown.
      i. The 10 second option causes each second's rate calculation to be based on the number of pulses sensed in the preceding 10 seconds.
      ii. The 60 second option uses a sliding 60 second interval to calculate the rate each second.
   d. The screen will display both the current rate and the cumulative volume since the beginning of the current record making it possible to do quick, in-field accuracy checks while the Meter-Master is recording. If not recording, the cumulative volume display starts at zero each time the Realtime Display is used.
5. Processing Data
   a. To download Data
      i. With MM connected, open MM Model 100 Software
      ii. Click on the "Download Data" button.
      iii. Choose appropriate file from list provided (files are time and date stamped with start time) by clicking the number to the left.
      iv. In the window that appears, create a new file name containing client name. Click "OK".
      v. In the window that appears, enter all relevant information. Be sure to click "Change MM 100 Settings" and select the correct meter type, size, etc.

      Note: The "Data Conversion Factor" can be used to force volume recorded to match volume observed from first and last meter readings. The ratios and composite volumes will be unchanged. To use, click the button next to "Automatic" and then click "OK."

      vi. Follow the above steps to download all needed files.
      vii. When finished downloading files, click "Exit" in the Download box.
   b. Create a Report and Export to Excel.
      i. Open MM Model 100 software. (MM does not need to be connected if the file has been downloaded.)
      ii. Click "Create Graph/Report."
      iii. Choose the appropriate file from the list provided. Click "OK."
      iv. In the popup window that appears, choose "Report/Export" under "Output Format."
      v. Make sure "Volume, Max, Avg, Min" is selected. If necessary, click "Customer Information" to change meter type, etc. (last chance to change).
      vi. Click "Data Grid."
      vii. A spreadsheet is displayed where the grid interval can be selected. To display hourly totals, set the grid interval to 3600 seconds and click "Recalculate."
      viii. Click "Select Graph/Report Start Time." Enter date and time for the start of readings in the format MM/DD/YY HH:MM:SS AM/PM. Click "OK."
      ix. Click "OK" to return to the display options window.
      x. Click "OK" to create the report.
      xi. The report is now displayed to export to Excel. Click the envelope button. Under "Format", choose "Excel 5.0 (XLS)." Click "OK."
      xii. Create a file name (8 character max) containing the client name. Save as ".xls" file.
      xiii. Select a location to save the file from the "Folders" list. Be sure to select the appropriate drive from the drop-down menu. Click "OK."

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xiv. Check to see that the file was properly saved in the appropriate folder.
xv. Close MM Model 100 software.

c. Place Data into the Flow Calculation Worksheet
   i. Open report in excel and open client's flow calculation worksheet. (Create a new worksheet using the correct Meter Master composite spreadsheet template found on the network.)
   ii. Copy the "volume" column (totals only) from the report (Make sure data is centered before copying.) and paste into the "GPH" or "FT3" column in the client's spreadsheet. Change dates and times to match start date and time.

8.0 MANUAL WASTEWATER COMPOSITING

Manual compositing is the physical act of combining at least two samples or sample aliquots together in a known proportion to create one representative sample for analysis.

Pace FSD utilizes Isco automatic/programmable samplers for the majority of wastewater projects. The samplers are typically equipped with a set of 24 bottles, each bottle representing an hour of discharge. A site's compositing method (equal volume, flow proportional or diurnal variation) is dependent upon discharge characteristics and project objectives. A regulatory agency dictates the compositing method for permitted work and is listed in a facility's discharge permit. Refer to the Client Data Sheet, or the Project Specific Site Information Form for compositing information for a given site.

A representative composite sample closely reflects the discharge characteristics of a given period of time – typically 24 hours. It is extremely important to consider the significance of missed hours of sample in the collection process. As a general rule, the minimum acceptable discharge capture percentage is 75%. This percentage is based on flow, not the number of hourly bottles containing sample. It is always recommended to discuss abnormalities and discrepancies with the project coordinator while considering project objects and facility process schedules.

In order to maintain the integrity of the waste stream characteristics, the composting should be done as soon after the takedown of the sampler as is feasible. Consult the project administrator if the samples cannot be composited and submitted to the laboratory the same day as the takedown.

8.1 Basic Compositing Procedure

The basic compositing procedure is the same regardless of the aliquot determination process.

1. Always start with a clean graduated cylinder, composite bucket, and labeled sample containers. Refer to the most current quality manual for preservation requirements.
2. Shake each bottle vigorously to mix sample prior to measuring the sample aliquot.
3. Composite according to determined aliquots measuring each sample aliquot to an accuracy of ± 2 mL. Make note of insufficient aliquot volumes, missed samples, and any variations of the effluent over the collection period, such as color and particulates.
4. After all sample aliquots are deposited into the composite bucket, slosh the composite sample to mix by swirling in opposite directions. Avoid one directional "swirling" to mix, as this separates the heavier particulate material to the outer edge of the composite sample and may bias the sample deposited into individual sample bottles.
5. After mixing the composite sample, begin filling bottles required for the project. For the given bottle set, half fill each bottle, mix the remaining composite sample a second time, and then finish filling the bottles and cap.
6. If composite pH measurement is required, calibrate pH meter (Section 6.2.1). Record meter identification and calibration on appropriate documentation. Measure and record pH and

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temperature of the composite sample. Also record time of measurement and technician initials.
7. Begin cooling process for samples requiring thermal preservation.
8. Finalize chain-of-custody and relinquish samples to receiving laboratory.
9. Check for project completeness. Update any project information on the Client Data Sheet.
10. Complete relevant information on the Project Completion Record and place project folder in "Data Package Complete" bin for project administrator.

8.2 Equal Volume Composites

Equal volume composites are suitable for discharges having a relatively constant flow rate over the sample collection period.

1. Determine the volume of composite sample required by the specified project parameter list, including additional volume for composite pH. Accommodate any laboratory quality control samples.
2. Divide the required final composite volume by the number of hours of discharge obtained from client. (This can be 24 for continuous discharge, 16 if facility operating two shifts or even 8 for single shift operations.) This is the minimum aliquot needed from each bottle of discharge.
3. Refer to Section 8.1 Basic Compositing Procedure.

8.3 Flow Weighted Composites

Flow weighted composites are suitable for discharges that demonstrate variations in flow rate over the monitoring period. A means of determining flow rate for each hour of discharge is needed to accomplish a flow-weighted composite. Hourly discharges are determined by recording hourly meter readings from fixed plant meters or by physically measuring the flow rate using primary and secondary flow monitoring devices.

8.3.1 Hourly Water Meter Compositing

Projects using hourly metered flow aliquot determinations rely on the principle that metered water into a facility minus metered or calculated deductions equals the discharge flow rate at the monitoring point. The most common complication encountered with hourly meter reading projects is missed meter readings for significant periods of time. Reasons for missed readings vary from uninformed clients, forgetfulness, or lack of staffing during certain hours. Refer to Section 8.4 for common anomalies. Missed meter readings may require a resample for another 24-hour period. Always consult the project administrator with questions concerning missed water meter readings

1. Obtain hourly "Meter Reading Record Sheet" from client.
2. Enter required header information and recorded hourly meter readings into client site-specific spreadsheet template found on the network.
3. Determine the final composite sample volume required by the specified project parameter list, including additional volume for composite pH.
4. Enter this volume in the designated cell within the spreadsheet. The flow data spreadsheet automatically calculates the individual hourly sample aliquots based on the hourly flow rates.
5. Submit flow data spreadsheet to project administrator, or other designated personnel, for review. Once the spreadsheet is initialed by the project administrator (or designated employee), composite the sample according to Section 8.1 Basic Compositing Procedure.

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8.3.2 Measured Flow Compositing

When Pace FSD performs flow monitoring, hourly and total flow data is obtained using automatic monitoring equipment. Primary and secondary flow-monitoring devices obtain a flow rate that directly correlates to the point of sample collection. Primary devices include weirs and flumes of various types. Secondary devices include various models of flow meters, such as the Isco 4230.

1. Enter required header information into client site-specific spreadsheet found on the network.
2. Using the hourly flow meter printout, enter the hourly gallons per minute (GPM) or other project specified flow rate data. If the hourly flow meter printout is compromised or data is collected from client's meter via RTD, enter values obtained from downloading data to Flowlink®.
    a. Flowlink® averages the discrete readings obtained by the flow meter into one summary interval value over a specified period of time. This summary interval value will be displayed with a time stamp representing the beginning or the end of the interval chosen. Isco instruments use the end time stamp to define the summary interval.
    b. Follow Quick Start procedure for complete Flowlink® process.
3. Determine the final composite sample volume needed by the specified project parameter list, including additional volume for composite pH. Enter this volume in the designated cell within the spreadsheet. The spreadsheet automatically calculates the individual hourly sample aliquots based on the hourly flow rates.
   Submit flow data spreadsheet to project administrator, or other designated personnel, for review. After the project manager verifies and initials spreadsheet, follow Section 8.1 Basic Compositing Procedure.

8.3.3 Diurnal Variation Compositing

Diurnal variation compositing is typically used for hourly discharges from hospitals. Diurnal variation composites are based on typical observed flow rates during certain periods of the day. Regulatory bodies determine and provide aliquots for diurnal variation. A peak flow hour is determined and is assigned a value of 1.0. The remaining hourly flows are determined as a percentage of peak flow. Obtain current diurnal flow percentages from site's current discharge permit.

8.4 Trouble Shooting Anomalies

Discuss discrepancies with client contact and/ or project administrator. Document inconsistencies, assumptions, and deviations on appropriate data sheets.

Anomaly #1: Sample was obtained during hours when no meter readings were recorded.

Calculate the average flow rate during the hours meter readings were not obtained. Determine the volume discharged during the non-recorded hours by subtracting the last recorded meter reading prior to the missed readings from next recorded meter reading. Divide this volume by the number of hours missed to determine the average flow rate for these hours. Proportion flow according to facility's production schedule.

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Anomaly #2: Sample was not obtained during hours when no meter readings were recorded.

Calculate hourly metered water use and disregard discharge during non-operating hours, provided the volume of water discharged during the non-operating, non-recorded hours is not greater than 10% of the total water discharged over the 24 hour period. Low flow rates during non-operating hours will be assumed to be leaking valves, toilets, etc. Technicians should question non-recorded water usage that exceeds the 10% factor to determine if it can be attributed to lawn water usage, water softener regeneration, or a significant plant operation such as the filling of a large tank overnight. Technician should also consider the validity of an unusually high aliquot volume for the startup hour on the following day.

Anomaly #3: Sample is not collected during hours of recorded meter readings indicating flow.

Calculate the discharge capture percentage onsite to determine if a minimum of 75% of the discharge was captured. A discharge capture of less than 75% may require resetting equipment and recording meters for an additional 24 hour period.

8.5 Hand Calculation of Composite Aliquots

Hourly sample aliquots based on hourly flow rates are determined for a composite without the use of a computer or spreadsheet by the following procedure.

1. Determine the total discharge volume over the 24-hour period by subtracting the first hourly reading from the last hourly reading. Apply relevant multipliers and convert to gallons if necessary (1 cubic foot = 7.481 gallons).
2. Determine the total composite volume in mL needed for project's sampling parameters.
3. Divide the total composite volume (Step 2) by the total discharge volume (Step 1). This is the composite factor.
4. Determine the flow volume for each hour by subtracting hourly meter reading 1 from hourly meter reading 2, hourly meter reading 2 from hourly meter reading 3 and so on until a discharge volume for each hour is obtained. Apply relevant multipliers and convert to gallons if necessary.
5. Multiply each hourly discharge volume (Step 4) by the composite factor (Step 3). The result will be the aliquot proportion in mL to be taken from the related bottle.
6. Use the aliquot volumes to composite samples.

9.0 CHAIN-OF-CUSTODY AND SAMPLE CHECK-IN

A chain-of-custody (COC) (Attachment 4) provides legal documentation of sample custody from collection through analysis. Field personnel or client representatives document on a COC every sample in custody for transportation, storage, transfer to another party, or check-in for analysis, either internally or by another laboratory. The COC is completed legibly with indelible ink following procedures in the most current revision of the Pace SOP *Documentation of Field Activities*, S-FSD-Q-004, or its equivalent replacement.

A chain-of-custody requires the following information:

- Client information
- Project name / Project number
- Requested turnaround time
- Unique sample identification
- Date and time collected
- Sample type (matrix)
- Preservatives used

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- Number of sample containers
- Requested analysis
- Sampler name and signature
- Additional sampler remarks or special requirements (if applicable)
- Relinquished signature, affiliation, time, date

The project administrator may provide a partially completed chain-of-custody to the field technician. The technician is responsible for adding the remaining information as the project progresses, including any abnormalities or deviations from normal or specified collection procedures or conditions. Sample information is recorded at time of collection. If a preprinted COC is not provided for a project, the technician is responsible for completing all required information on the COC.

The relinquishment of all samples, either to an intermediate handler or to the point of analysis, must be documented on the COC. All samples submitted for analysis by Pace FSD or by an external laboratory are accompanied by a completed chain-of-custody. Submitted samples must comply with laboratory's sample acceptance policy. If collection and analysis is performed by the same individual, it is noted on the COC that the sample was not relinquished to any other party. Sample results generated by Pace FSD are kept in their respective project folder.

10.0 REFERENCES

The most current issue of the reference document available shall be employed for compliance requirements related to this manual.

10.1 40 CFR Part 136, Environmental Protection Agency (EPA); Guidelines Establishing Test Procedures for the Analysis of Pollutants.

10.2 *Isco Open Channel Flow Measurement Handbook*. Teledyne Isco, Inc.

10.3 *Groundwater Example Sampling Protocol for Monitoring Well*. (2007). Minnesota Pollution Control Agency.

10.4 Field Guidance Manual. (1998) Minnesota Pollution Control Agency: Ground Water and Solid Waste Division.

10.5 *Determination of Turbidity by Nephelometry, Method 180.1, Revision 2.* (August 1993). Environmental Protection Agency (EPA).

10.6 *Calibration of Field Instruments, Standard Operating Procedure, Revision 2.* (January 2010). Environmental Protection Agency (EPA).

10.7 *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures*; Federal Register, Vol. 77, No. 98 (May 18, 2012). Environmental Protection Agency (EPA).

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11.0 REVISIONS

Document Number	Description of Change	Date
PM-FSD-004-rev.06	All document: Updated Inc. to LLC; updated "NIST-traceable" to "traceable to national standards maintained by NIST" where applicable. Section 4.2.7: Added BUuck controller instructions; subsequent sections renumbered. Section 5.4: Added sentence addressing undisturbed time period in first paragraph; added and edited a. and b. of Step 4. Section 6.6: Added barometric pressure requirements for dissolved oxygen measurements; added clause about calibration units; updated ICV and CCV requirements from mg/L to %; updated specific meter procedures to reflect new procedures. Attachment 1: Updated to current year. Attachment 4: Updated to current version. Attachment 12: Deleted Bore Volume Chart; now included on new Attachment 12. Updated to Quick Reference Tables. Attachment 13: Deleted Domestic Well Log; superseded by GW Well Sampling FDLS in Attachment 11; replaced with Vehicle Equipment Lists. Attachment 14C: Updated Dissolved Oxygen Field Data Sheet Table I: Removed; replace with Attachment 13. Table II: Removed; replaced with Attachment 13. Table III: Removed Oxygen Solubility Table; no longer applicable to dissolved oxygen procedure. Table IV: Removed pH Buffer-Temp Relationship Chart; now included on Attachment 12. Table V: Removed Zobell Solution-Temperature Relationship Chart; now included on Attachment 12.	June 15, 2017

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Field Services Division

ATTACHMENT 1
Project Record

2017

Client Name _____ Project Name _____
Facility Location _____ Field Prjoject No. _____
Client P.O. No. _____ Lab Project No. _____
Monitoring for Week of: _____

Project Lead _____ Project Manager _____

Project Task	Comments	By	Date
1. Project Proposal			
2. Permit on File at Pace			
3. Sampling Date(s)			
4. Create Project Number			
5. Project Expenses Submitted			
6. Profile Initiated/Confirmed			
7. Review Project Scope			
8. Print CoC and Labels			
9. Project Preparation			
10. Sampling Completed			
11. Grabs Collected			
12. Hourly pH Taken			
13. pH-Field Complete			
14. Samples Submitted to Lab			
15. Verify FDS & Project Completion			
16. Field Data Entered/Printed			
17. Project Expenses Submitted			
18. Data Pkg Assembled/Submitted			
19. SAF Review			
20. Field Data Review			
21. Lab Data Received			
22. Email Field/Lab Data to Client			
23. Email Final Report to Client			
24. Verify Charges & Invoice			
25. Update Schedule & Workbook			
26. Hard Copy of Report Sent			
27. Invoice Sent & Logged			

Comments and Notes:

Process Codes
PreSampling Task
Sampling Task
Post Sampling Task

Responsibilities:
Project Manager
Project Lead
Support Services

Tasks may be situationally reassigned

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Pace Analytical Field Services Division

ATTACHMENT 2

Wastewater Monitoring Client Data Sheet

Event

FDS P/N 00-00-0000

Project/Client Name: _____
Facility Designation: _____
Facility Address: _____

Facility Contact: _____
Phone: _____
Alternate Contact: _____
Phone: _____
Site Experienced: _____

Report Contact: _____
Phone: _____
E-mail: _____
Schedule Contact: _____
Phone: _____
E-mail: _____
Reg. Permit No.: _____
Expiration Date: _____
No. Techs On-site: _____
Lab Profile No.: _____

Schedule Preference: _____

Directions to Facility: _____

	Monitoring Sites	Analytes	Equipment
SP-01)			

Compositing Information:
SP-01) _____

Total Facility Discharge Determination Method:

	Meter ID	Units	Multiplier
1			
2			
3			
4			
5			
6			

Project Comments:

Field Comments: _____

Tech Labor Basis:
1.25 Hrs SP,DM,SA
0.75 Hrs OP, OT
1.75 Hrs Travel (TT

Printed: 5/7/2014

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Field Services Division

Project Specific Site Information

Requested By : _____ Project Manager: _____

Client Name: _____

Client Report Contact: _____ Alternate On Site Contact: _____

Facility Address: _____

_____

Directions: _____

_____

Phone Number: _____ Fax Number: _____

Client Project Reference: _____ Client Purchase Order Number: _____

Laboratory Profile No : _____ EPIC Project No: _____

Project Goal/Objective

_____

_____

_____

_____

_____

Equipment Requirements

_____

_____

_____

_____

Laboratory Analysis

Specify Special Requirements ie@ Turnaround Times,PRL,Method Requirements,ect,,,

_____

_____

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ATTACHMENT 4

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

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Page: of

Section A Required Client Information:

Company:	
Address:	
Email To:	
Phone:	Fax:
Requested Due Date/TAT:	

Section B Required Project Information:

Report To:	
Copy To:	
Purchase Order No.:	
Project Name:	
Project Number	

Section C Invoice Information:

Attention:	
Company Name:	
Address:	
Pace Quote Reference:	
Pace Project Manager:	
Pace Profile #:	

REGULATORY AGENCY

NPDES GROUND WATER DRINKING WATER
UST RCRA OTHER
SITE LOCATION: NC GA IL IN OH SC W OTHER

Filtered (Y/N)

Requested Analysis:

Pace Project No. / Lab I.D.

Section D Required Client Information

SAMPLE ID
One Character per box.
(A-Z, 0-9 / ,-)
Sample IDs MUST BE UNIQUE

Valid Matrix Codes

MATRIX	CODE
DRINKING WATER	DW
WATER	WT
WASTE WATER	WW
PRODUCT	P
SOIL/SOLID	SL
OIL	OL
WIPE	WP
AIR	AR
TISSUE	TS
OTHER	OT

ITEM #	MATRIX CODE	SAMPLE TYPE G=GRAB C=COMP	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Residual Chlorine (Y/N)	Pace Project No. / Lab I.D.
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		

Additional Comments:

RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME

SAMPLE CONDITIONS

	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N		Y/N

Temp in °C | Received on Ice | Custody Sealed Cooler | Samples Intact

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: DATE Signed (MM / DD / YY)

Inst pH = _____ s.u. Comp pH = _____ s.u.

Date: _____ Date: _____

Time: _____ Time: _____

Analyst: _____ Analyst: _____

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Sample Label

Pace Analytical Services, Inc.
1700 Elm Street, Suite 200
Minneapolis, MN 55414
(612) 607-1700

Client/Project: _____

Sample ID: _____

Date Collected: _____ Time: _____

Collected By: _____

Analyses: _____

Preservative: _____

Comments: _____

Pace Analytical
Field Services Division

PROJECT PRODUCTS FORM

Client Name: _____ **Project No:** _____ **Dept No:** _____

Date: _____ **Emp. Initials:** _____ **PM:** _____

Air / FTIR

Qty.	Item Description	Cost	Qty.	Item Description	Cost
	Air Analysis - EPA 15			Reagent Set - EPA 11	
	Air Supplies - 1/4" Teflon™ Tubing			Reagent Set - EPA 202	
	Air Supplies - 3/8" Teflon™ Tubing			Reagent Set - EPA 23	
	Air Supplies - Large Inert Bag			Reagent Set - EPA 26	
	Air Supplies - Poly Tubing			Reagent Set - EPA 29	
	Air Supplies - Small Inert Bag			Reagent Set - EPA 5	
	Air Supplies - X-Large Inert Bag			Reagent Set - EPA 6/8	
	Ambient - Air Monitoring Calibration kit			Reagent Set - Misc. Complex	
	Canister Rental/Cleaning			Reagent Set - Misc. Simple	
	Chromatography - GC/FID			Sampling Train - EPA 0030 (VOST)	
	Chromatography - GC/FPD			Sampling Train - EPA 11	
	FTIR - Gas Phase - Daily Rate			Sampling Train - EPA 16A	
	FTIR - Gas Phase - Disc. Rate			Sampling Train - EPA 16C	
	FTIR - Gas Phase - Weekly Rate			Sampling Train - EPA 17	
	Gas - Duo Gas Cal Set of 3 & Zero			Sampling Train - EPA 2	
	Gas - FID Combustion Set (H2/Air)			Sampling Train - EPA 201A	
	Gas - FTIR Cal Set (Ethylene & 0)			Sampling Train - EPA 202	
	Gas - Purge Nitrogen			Sampling Train - EPA 23	
	Gas - Single Gas Cal Set of 3 & Zero			Sampling Train - EPA 25	
	Gas - Tri Gas Cal Set of 3 & Zero			Sampling Train - EPA 26	
	Gas Analyzer - 3 Gas Set			Sampling Train - EPA 29	
	Gas Analyzer - CEM Lab			Sampling Train - EPA 3	
	Gas Analyzer - Chiller			Sampling Train - EPA 5	
	Gas Analyzer - Chiller FHR Dedicated			Sampling Train - EPA 8	
	Gas Analyzer - CO - EPA 10			Sampling Train - Macro CR	
	Gas Analyzer - Data Acquisition			Sampling Train - Midget CR	
	Gas Analyzer - Heated Line			Sampling Train - NCASI 94.02	
	Gas Analyzer - Heated Line FHR Dedicated			Sampling Train - NCASI 98.01	
	Gas Analyzer - Multigas Portable			Sampling Train - Vacuum Chamber	
	Gas Analyzer - NOx - EPA 7E			Sorbent Cartridge - Macro	
	Gas Analyzer - O2/CO2 - EPA 3A			Sorbent Cartridge - Midget	
	Gas Analyzer - Portable FID or PID			Reporting - Extra CD or DVD (Initial)	
	Gas Analyzer - Portable O2			Reporting - Extra Hard Copy (Initial)	
	Gas Analyzer - SO2 - EPA 6C			Reporting - Republished CD/DVD	
	Gas Analyzer - THC - EPA 25A			Reporting - Republished Hard Copy	
	Gas Sampler - Personnel Pump (IH)				

Groundwater			Wastewater		
Qty.	Item Description	Cost	Qty.	Item Description	Cost
	GW Analysis - Ferrous Iron Kit Xcel			Installation - Weir Fabrication	
	GW Analysis - HNU Model 101			Liquid Sampling - Bailer Rental	
	GW Analysis - Manganese Kit Xcel			Liquid Sampling - Coliwasa	
	GW Analysis - Methane Meter			Liquid Sampling - Peristaltic Pump	
	GW Analysis - OVA Model 128			SW Sampling - Pond Sampler	
	GW Analysis - Sonde Field Parameters			WW Analysis - Conductance Meter	
	GW Analysis - Turbidity Meter			WW Analysis - Dissolved O2 Meter	
	GW Sampling - AMS Coring			WW Analysis - Hach Chlorine Kit	
	GW Sampling - Deep Well Std.			WW Analysis - pH Field Test	
	GW Sampling - Deep Well Xcel			WW Analysis - pH Meter	
	GW Sampling - Eckmann Dredge			WW Analysis - Recording pH Meter	
	GW Sampling - Kemmerer Sampler			WW Safety - Confined Space Equipment	
	GW Sampling - Shallow Well			WW Safety - Davit Arm	
	GW Sampling - LLHg Kit			WW Sampling - Flow Metering Insert	
	GW Supplies - 0.45 µm Filter Std.			WW Sampling - Isco Flow Meter	
	GW Supplies - 0.45 µm Filter Xcel			WW Sampling - Isco Sampler	
	GW Supplies - Disposable Bailer			WW Sampling - Meter Master Flow	
	GW Supplies - Sediment Filter			WW Sampling - Weir/Flume Insert	
	GW Supplies - Teflon Lined Poly			WW Supplies - 3/8" Teflon Tubing	
	Soil Sampling - Auger			WW Supplies - 3/8" Tygon Tubing	

Shared

Miles	Item Description	Cost	Miles	Item Description	Cost
	Mileage - Van/Passenger			Miscellaneous Equipment	
	Mileage - Truck/Trailer Combo			Project Supplies (non-standard)	
	Mileage - 4WD Utility Truck			Shipping (Dollars)	

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Field Services Division

Attachment 7

NPDES Field Log Sheet

Client Name: _____

Sampling Site: _____ Date Collected: _____

Pace Project No.: _____ Collected By: _____

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						Yes or No if Present		
Location	Time	Temp. (°C)	pH (s.u.)	MDL (mg/L)	Chlorine (mg/L)	Oil Film	Floating Solids	Foam

pH Meter Calibration
10 = _____
7 = _____
4 = _____

Solution Identification	
Buffer 10.0 Lot# _____	buffer 10.0
Mfg _____ Exp. Date _____	
Buffer 7.0 Lot# _____	buffer 7.0
Mfg _____ Exp. Date _____	
Buffer 4.0 Lot# _____	buffer 4.0
Mfg _____ Exp. Date _____	

Comments: _____

_____

_____

_____

ATTACHMENT 8

WasteWater Field Data Log Sheet

Pace Analytical® Field Services Division

Page ___ of ___ (monitoring point)

General Information

Client Name _____ Pace Project No. _____

Facility Location _____ Sample Dates _____ (setup) _____ (takedown)

On-Site Contact _____ Time _____ (setup) _____ (takedown)

Monitoring Point _____ Technician(s) _____ (setup) _____ (takedown)

Operating Hours _____

Sampling

Auto Sampler Model _____ Auto Sampler Unit No. _____

Auto sampler base chilled at set-up: ☐ *Yes* ☐ *No* ☐ *Not Applicable*

Conditions at the point of probe placement during set-up: *(check all that apply - use comments section for anomalies)*

☐ *Sufficient depth (full aliquot capture)* ☐ *Shallow depth (difficult aliquot capture)* ☐ *Sporadic flow* ☐ *No flow at setup*

☐ *Turbulent* ☐ *Sluggish* ☐ *Probe placement avoids **settled** solids (if no, explain)* _____

Aliquot Sampled: ☐ *Every 15 min, 4 samples/bottle for 24 hrs* ☐ *Other* _____

Auto sampler base chilled at take-down: ☐ *Yes* ☐ *No* ☐ *Not applicable*

Flow

Flow Meter Model _____

Flow Meter Unit No. _____

Primary Device _____

Measured Discharge (GPD) _____

Metered Plant Discharge (GPD) _____

	Setup	Takedown
Reference Head (FT)		
Meter Display (FT)		
Technician		
Date		

pH

Composite pH ☐ *Yes* ☐ *No* Meter ID _____

Hourly pH ☐ *Yes* ☐ *No* Meter ID _____

Continuous pH ☐ *Yes* ☐ *No* Meter ID _____ *(see continuous meter calibration archives for calibration results including CCV)*

Instantaneous pH ☐ *Yes* ☐ *No* Meter ID _____

Composite pH Measurement

Analyst	pH Result (s.u.)	Temp (°C)	Date (m/d)	Time

(see meter calibration archives for calibration results)

Continuing Calibration Verification (CCV)

Standard Info: Value	Standard Info: Mfg. / Lot No.	Standard Info: Exp.	Meter Value (s.u.)	Temp (°C)	Date (m/d)	Time

CCV: Pass / Fail — *CCV acceptable if ± 0.1 s.u. of buffer value*

Hourly pH Measurements

Analyst	Date	Analysis Start Time	Analysis End Time

(see meter calibration archives for calibration results)

Continuing Calibration Verification (CCV)

Standard Info: Value	Standard Info: Mfg. / Lot No.	Standard Info: Exp.	Meter Value (s.u.)	Temp (°C)	Date (m/d)	Time

CCV: Pass / Fail — *CCV acceptable if ± 0.1 s.u. of buffer value*

Instantaneous pH Measurements

Analyst	pH Result (s.u.)	Temp (°C)	Date (m/d)	Time

(see meter calibration archives for calibration results)

Continuing Calibration Verification (CCV)

Standard Info: Value	Standard Info: Mfg. / Lot No.	Standard Info: Exp.	Meter Value (s.u.)	Temp (°C)	Date (m/d)	Time

CCV: Pass / Fail — *CCV acceptable if ± 0.1 s.u. of buffer value*

Comments

Attach additional notes if necessary.

Lead Technician Signature: _____ Date: _____

Form Revised 05/08/17

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ATTACHMENT 8

Pace Analytical®
Field Services Division

WasteWater Field Data Log Sheet

Page ____ of ____
(meter log sheets)

Metered Water Use

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Meter ID	+ FT³ - GAL	+ FT³ - GAL
End (date) / (time)		
Begin (date) / (time)		
Difference		
Gallons		

Meter ID	+ FT³ - GAL	+ FT³ - GAL
End (date) / (time)		
Begin (date) / (time)		
Difference		
Gallons		

Meter ID	+ FT³ - GAL	+ FT³ - GAL
End (date) / (time)		
Begin (date) / (time)		
Difference		
Gallons		

Meter ID	+ FT³ - GAL	+ FT³ - GAL
End (date) / (time)		
Begin (date) / (time)		
Difference		
Gallons		

Meter ID	+ FT³ - GAL	+ FT³ - GAL
End (date) / (time)		
Begin (date) / (time)		
Difference		
Gallons		

Note: Cubic Feet (FT^3) X 7.481 = Gallons (GAL)

Form Revised 4/24/2012

Pace Analytical® Field Services Division

Grab Sampling Field Data Log Sheet

Page ___ of ___
(monitoring point)

General Information

Client Name _____ Pace Project No. _____

Facility Location _____ Project Name _____

On-Site Contact _____ Date(s) _____

Monitoring Point _____ Technician(s) _____

Meters

☐ pH Meter (ID): _____ ☐ Chlorine Meter (ID): _____

☐ Multi-Parameter Meter (ID): _____ ☐ Other (description/ID): _____

(see meter calibration archives for calibration results)

Sampling Equipment

☐ Not Applicable *(collected directly into sample bottle)*

☐ Transfer Container (circle type): *Glass / Plastic / Stainless*

☐ Bailer (circle type): *Stainless / Disposable*

☐ Trowel/Scoop ☐ Shovel / Post-Hole Digger

☐ Pump and Tubing (circle tubing type): *Tygon / Teflon / Other:* _____

☐ Other Device(s): _____

☐ COLIWASA (circle type): *Plastic / Glass*

☐ Pole Sampler

☐ Dredge

☐ Sediment Core Sampler ☐ Hand Soil Auger

pH

pH Measurement					Continuing Calibration Verification (CCV)						
					Standard Info						
Analyst	pH Result (s.u.)	Temp (°C)	Date (m/d)	Time	Value	Mfg. / Lot No.	Exp.	Meter Value (s.u.)	Temp (°C)	Date (m/d)	Time
(see meter calibration archives for calibration results)					CCV: Pass / Fail			*CCV acceptable if ± 0.1 s.u. of buffer value*			

Sample Collection Notes

Monitoring Point	Time	Results / Observations / Sample Characteristics
		Attach additional notes if necessary.

Samples chilled immediately after collection: ☐ *Yes* ☐ *Other* _____

Lead Technician Signature: _____ Date: _____

Form Revised 3/17/16

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Pace Analytical® Field Services Division

Client _____

Project _____

Date _____

Meter Reading Record Sheet

Meter _____ Meter _____

Units _____ Units _____

	Time	Meter Reading	Comments	Meter Reading	Comments	
1						1
2						2
3						3
4						4
5						5
6						6
7						7
8						8
9						9
10						10
11						11
12						12
13						13
14						14
15						15
16						16
17						17
18						18
19						19
20						20
21						21
22						22
23						23
24						24
25						25

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ATTACHMENT 11

Pace Analytical® Field Services Division

Well Sampling Field Data Log Sheet

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Well Description and Presampling Information

Client _____ **Project** _____ **Project No.** _____

Monitoring Point ID _____ Labeled _____

Inside Diameter _____ (inches) Key # _____ ☐ Locked ☐ Not Locked

Casing Material: ☐ PVC ☐ Steel ☐ Stainless Steel

Depth Measurement and Elevations (from top of well casing)

Top of Casing Elevation _____ Feet

Well depth _____ Feet

Static water level measurement before purging _____ Feet

Static water level measurement at time of sampling _____ Feet

Static Water Level Elevation Before Purging _____ Feet

Purge Method _____

Date Purged _____ Water Column _____ Feet

Time Purged _____ One Casing Volume _____ Gallons

Pump Rate _____ GPM / LPM Volume Purged _____ Gallons

Field Sampling Data

Date Sampled _____

Time Sampled _____

Sampling Equip. _____

Analyzed by _____

Field Parameter Measurements of Sample

pH _____ (units) D.O _____ (mg/l)

Spec. Cond. _____ (µmhos/cm) Turbidity _____ (NTU)

Temp. _____ (°C) Eh _____ (mV)

Other _____

Field Measurements Temp. Corrected: ☐ Yes ☐ No ☐ NA

Sample for Soluble Metals Filtered in Field: ☐ Yes ☐ No ☐ NA

Weather Conditions During Sampling: _____

Sample Description: _____

Observations: _____

Stabilization Test

Time	pH (units)	Specifc Conductance (µmhos/cm)	Temp (°C)	D.O. (mg/l)	Turbidity (NTU)	Eh (mV)	Volume Purged (cumulative gal)

Samples chilled immediately after collection: ☐ *Yes* ☐ *Other* _____

Form Revised: 03/10/2016

Name/Affiliation of Sampler(s): _____

Lead Technician Signature: _____ Date: _____

ATTACHMENT 12

Pace Analytical® Field Services Division

Quick Reference Tables
Water Monitoring

pH Buffers			
Temp (°C)	ERA (Guide 34)		
	4	7	10
0	4.000	Not Listed	10.32
5	3.998	7.056	10.25
10	3.997	7.038	10.18
15	3.998	7.022	10.12
20	4.015	7.018	10.06
25	4.00	7.000	10.00
30	4.025	6.979	9.97
35	4.022 (37 deg)	6.937	9.93
40	4.027	6.917	9.99
50	4.050	6.893	9.83

Source: Manufacturer Specs

pH Buffers		
Temp (°C)	Fisher	Ricca
	7	7
0	7.13	7.12
5	7.10	7.09
10	7.07	7.06
15	7.05	7.04
20	7.02	7.02
25	7.00	7.00
30	6.99	6.99
35	6.98	6.98
40	6.97	6.98
50	6.97	6.97

Source: Manufacturer Specs

Zobell's ORP Calibration Values		
Temp (°C)	YSI	Hach
-5	270.0	NA
0	263.5	NA
5	257.0	NA
10	250.5	243.5
15	244.0	236.0
20	237.5	228.5
25	231.0	221.1
30	224.5	NA
35	218.0	NA
40	211.5	NA
45	205.0	NA
50	198.5	NA

Source: Manufacturer Specs

Reporting Specifications	
Parameter	Nearest
pH	0.1 s.u.
Sp. Cond.	10 umhos/cm
Temp	0.5 °C
DO	0.1 mg/L
Eh/ORP	1 mV
Turb 0.0-1.0	0.05 NTU
Turb 1-10	0.1 NTU
Turb 10-40	1 NTU
Turb 40-100	5 NTU
Turb 100-400	10 NTU
Turb 400-1000	50 NTU
Turb >1000	100 NTU

Source: Pace Water Manual

Well Vol. Multiplication Factors	
Vol. of Water (gal) = Ft of Water X Factor	
Diameter (in.)	Factor*
1	0.0408
1 ½	0.0918
1 ⅝	0.108
2	0.163
3	0.367
4	0.653
5	1.02
6	1.468
8	2.61
10	4.08
*(Radius inches /12)^2 x 3.14 x 7.48	

Source: EPA

Well Stabilization Criteria	
pH	± 0.1
Sp. Cond.	± 5%
Temp	± 0.5 °C
DO	± 0.5 mg/L
Turbidity	Project Specific
ORP	Project Specific

Source: Pace Water Manual

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ATTACHMENT 13
(Page 1 of 2)

Groundwater Vehicle Checklist

Inside Cab		Truck Compartments	
Vehicle registration card		Latex gloves	
Vehicle insurance card		Monkey grip gloves	
Accident report procedure		YSI meter or Sonde	
Gas card		pH standards	
First aid kit / Fire extinguisher		Eh standard	
MSDS book		Conductance standard	
Clipboard		DO meter	
Client/project information		DO membrane kit	
Forms binder		Flow through cell	
Groundwater manual		Flow through cell connections	
Labels and Chain-of-Custodies		DI water in gallon amber	
Calculator		Well sounder	
Pens		Downrigger and tripod	
Project keys		Stainless steel bailers	
Watch		Stainless steel bailer check valves	
Cell phone		Disposable bailers	
Jack		Peristaltic pump tubing (25 feet)	
Jumper cables		Peristaltic pump and battery	
Paper towels		Metals filters	
Ice/snow scraper		2" Grundfos pump and cable	
		Grundfos control box	
Toolbox		1/2" Grundfos adapter	
Flathead screwdriver (large & small)		Grundfos pump tubing	
Phillips screwdriver		Plastic tarp	
Hammer		Coolers/ice	
Crescent wrench (large & small)		Baggies	
Ratchet socket set (3/16"-3/4")		Sample bottles	
Open end wrench set (3/16"-1")		Graduated bucket	
Utility knife		Bolt cutter	
Pliers		Monkey wrench	
Wire cutter		Bladder pump & controller	
Crimper and crimps		Generator	
Tape measure		Extension cord	
Duct tape		Oil	
Electrical tape		Trash container	
Batteries (all types)		Scissors	
Flashlight			
Full set of Allen wrenches		**Miscellaneous**	
Needle nose pliers		Hard hat	
Treble hook		Safety glasses	
Cable ties		Rain gear	
		Coveralls	
		Boots	

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ATTACHMENT 13 - (Page 2 of 2)

Wastewater Safety and Vehicle Equipment Checklist

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Manhole General Equipment
Pick
Sledge Hammer
Crow bar
Various lengths of conduit
Various lengths of 2x4's
Hooks (4) & Harnesses (4)
Rope
Spare pen's & sharpies
Babbit Rite

ISCO Sampler
Battery
Tubing (at least 50 feet)
Cable ties
Pump tubing

ISCO Flow Meter
Battery
Bubbler paper roll
Stainless steel tubing

pH meter(s)
Analytical Measurements, YSI Sonde
pH meter paper roll
Calibration buffers (7,10, DI)

Manhole Entry Equipment
Air Toxicity Meter
Spare toxicity meter batteries
Tripod, Winch, Lanyard
Blower with battery
Bucket with Rope
Rubber hip boots
Long rubber gloves

Personnal Protective Equipment (PPE)
Latex and leather gloves
Hard hats (2)
Highway vest
Safety glasses
Steel toe boots
Coveralls
Hearing protection
Winter, carharts
jacket, hat, insulated boots & gloves

Cab
Accident Report package
Gas card
Hand sanitizer
Fire extinguisher
First Aid Kit
Map
MSDS Book
Gas receipts taken out
Cab clean
Data sheet pack

Miscellaneous
Hand saw
Duct tape
Masking tape
Electrical tape (3)
Extension cord
Highway cones (7)
Kim wipes
Assorted nails
Helmet head lamp
Medium large mirror

Vehicle Maintenance
Jumper cables
Windshield washer fluid
Ice scraper
Oil
Paper Towels
Isco batteries taken out
Back of vehicle clean
Empty trash

Tool Box
Flathead screwdriver (2)
Phillips screwdriver (3)
Heavy duty flathead screwdriver
Hammer
Large adjustable wrench
Small adjustable wrench
Ratchet socket set
Utility knives (2)
Pliers
Tape measure
C-clamps (2)
Flashlight
Full probes (2)
Level
Engineer ruler
Weighted probes (4)
Batteries (for flashlight)

Grabs
Sample Containers
Rope & Bailer
Pond Sampler
Cooler with Ice
Sample labels & COC

Pace Analytical® Field Services Division

ATTACHMENT 14A

pH Calibration and Verification Sheet

Page _____ of _____

Technician(s) _____

Meter Make _____ Meter ID _____

Initial Calibration (IC)

Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
IC Slope (%) _____	Pass / Fail / NA		*(Acceptable Slope: 92-102%)*		

Initial Calibration Verification (ICV)

Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Pass / Fail	*(ICV acceptable if ± 0.1 s.u. of buffer value)*				

Continuing Calibration Verification (CCV)

See Below ☐ See Field Data Log Sheet ☐ See Raw Data (continuous pH) ☐

Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Pass / Fail	*(CCV acceptable if ± 0.1 s.u. of buffer value (or ± 0.5 s.u. for continuous pH))*				
Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Pass / Fail	*(CCV acceptable if ± 0.1 s.u. of buffer value (or ± 0.5 s.u. for continuous pH))*				
Buffer Value | Manufacturer | Lot No. | Exp. Date	Meter Value	Temp (°C)	Date	Time	Initials
Pass / Fail	*(CCV acceptable if ± 0.1 s.u. of buffer value (or ± 0.5 s.u. for continuous pH))*				

Form Revised: 2/29/2016

Lead Technician Signature: _____ Date: _____

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Pace Analytical®
Field Services Division

ATTACHMENT 14B

pH and Conductivity
Field Calibration and Verification Sheet

Technician(s) ____________________ Date __________ Page _____ of _____

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pH

Meter Make ____________________ Meter ID ____________________

Initial Calibration (IC)

Buffer Value	Manufacturer	Lot No.	Exp. Date	Meter Value	Temp (ºC)	Time	Initials
Buffer Value	**Manufacturer**	**Lot No.**	**Exp. Date**	**Meter Value**	**Temp (ºC)**	**Time**	**Initials**
Buffer Value	**Manufacturer**	**Lot No.**	**Exp. Date**	**Meter Value**	**Temp (ºC)**	**Time**	**Initials**

IC Slope (%) __________ Pass / Fail *(Acceptable Slope: 92-102%)*

Initial Calibration Verification (ICV)

Buffer Value	Manufacturer	Lot No.	Exp. Date	Meter Value	Temp (ºC)	Time	Initials

Pass / Fail *(ICV acceptable if ± 0.1 s.u. of buffer value)*

Continuing Calibration Verification (CCV)

See Below ☐ See Field Data Log Sheet ☐ See Raw Data (continuous pH) ☐

Buffer Value	Manufacturer	Lot No.	Exp. Date	Meter Value	Temp (ºC)	Time	Initials

Pass / Fail *(CCV acceptable if ± 0.1 s.u. of buffer value)*

Conductivity

Initial Calibration (IC)

High Standard Value: __________

Mfg. __________
Lot No. __________
Exp. Date __________

Meter Result (µS/cm)	Temp (°C)

Initial Cal. Verification (ICV)

Low Standard Value: __________

Mfg. __________
Lot No. __________
Exp. Date __________

Meter Result (µS/cm)	Temp (°C)

Meter Make __________ ID __________
IC/ICV Time __________
Initials __________
ICV: Pass / Fail
(ICV acceptable if ± 5% of standard value or 10 µS/cm, which ever is greater)

Continuing Calibration Verification (CCV)

Time __________ Initials __________ Std. Value __________ ☐ High Standard ☐ Low Standard
Result (µS/cm) __________ Temp (°C) __________ CCV: Pass / Fail *(CCV acceptable if ± 5% of standard value or 10 µS/cm, which ever is greater)*

Time __________ Initials __________ Std. Value __________ ☐ High Standard ☐ Low Standard
Result (µS/cm) __________ Temp (°C) __________ CCV: Pass / Fail *(CCV acceptable if ± 5% of standard value or 10 µS/cm, which ever is greater)*

Time __________ Initials __________ Std. Value __________ ☐ High Standard ☐ Low Standard
Result (µS/cm) __________ Temp (°C) __________ CCV: Pass / Fail *(CCV acceptable if ± 5% of standard value or 10 µS/cm, which ever is greater)*

Form Revised: 11/13/2013

Lead Technician Signature: ____________________ Date: __________

Pace Analytical®
Field Services Division

Redox and Dissolved Oxygen

Field Calibration and Verification Sheet

Technician(s) ______________________ Date ____________ Page _____ of _____

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Redox (ORP)

Initial Calibration (IC)		Initial Cal. Verification (ICV)		
Calibration Std. Value (mV)		Secondary Source Std. Value (mV)		Meter Make ____________ ID __________
Mfg. ____________ Lot No. ____________ Exp Date ____________		Mfg. ____________ Lot No. ____________ Exp. Date ____________		IC/ICV Time ____________ Initials ____________
Meter Result (mV)	Temp (°C)	Meter Result (mV)	Temp (°C)	*ICV: Pass / Fail* *ICV acceptable if ± 10 mV of standard value.*

CCV Time ____________ Initials ____________ Std. Value ____________ ☐ Calibration Standard ☐ Secondary Source Standard
Result (mV) ____________ Temp (°C) ____________ CCV: Pass / Fail *(CCV acceptable if ± 10 mV of standard value)*

CCV Time ____________ Initials ____________ Std. Value ____________ ☐ Calibration Standard ☐ Secondary Source Standard
Result (mV) ____________ Temp (°C) ____________ CCV: Pass / Fail *(CCV acceptable if ± 10 mV of standard value)*

CCV Time ____________ Initials ____________ Std. Value ____________ ☐ Calibration Standard ☐ Secondary Source Standard
Result (mV) ____________ Temp (°C) ____________ CCV: Pass / Fail *(CCV acceptable if ± 10 mV of standard value)*

Dissolved Oxygen (D.O.)

Initial Calibration (IC)	Initial Cal. Verification (ICV)	
% Saturation	% Saturation	IC/ICV Time ____________ Initials ____________
Ambient Temp: ________ (°C) Barometric Pressure ________ (in. Hg) Barometric Pressure ________ (mm Hg) Calibration Value[1] ________ (%)	Ambient Temp: ________ (°C) Barometric Pressure ________ (in. Hg) Barometric Pressure ________ (mm Hg) Calibration Value[1] ________ (%)	DO Meter ID ____________ Barometer ID ____________
IC Meter Result (%)	ICV Meter Result (%)	ICV: Pass / Fail *(ICV/CCV acceptable if ± 2% of Calibration Value)*

[1] Calibration Value (% Saturation) = (Measured BP / Sea Level BP) X 100. Sea Level BP = 760 mm Hg or 29.92 inches Hg.

CCV Time ____________ Initials ____________ Barometric Pressure ____________ (in. Hg / mm Hg)
Calibration Value ____________ (%) Result ____________ (%) CCV: Pass / Fail *(CCV acceptable if ± 2% of Calibration Value)*

CCV Time ____________ Initials ____________ Barometric Pressure ____________ (in. Hg / mm Hg)
Calibration Value ____________ (%) Result ____________ (%) CCV: Pass / Fail *(CCV acceptable if ± 2% of Calibration Value)*

CCV Time ____________ Initials ____________ Barometric Pressure ____________ (in. Hg / mm Hg)
Calibration Value ____________ (%) Result ____________ (%) CCV: Pass / Fail *(CCV acceptable if ± 2% of Calibration Value)*

Form Revised: 02/03/17

Lead Technician Signature: ______________________ Date: ______________

Pace Analytical® Field Services Division

ATTACHMENT 14D

Chlorine and Chloramines Field Data Sheet

Page _____ of _____

Technician _____ Meter Make _____ ID. _____

Low Standard

Mfg. _____

Lot No. _____

Exp Date _____

High Standard

Mfg. _____

Lot No. _____

Exp Date _____

Reagents

Total - Ampul	Mfg. _____ Lot No. _____ Exp Date _____
Total - Powder	Mfg. _____ Lot No. _____ Exp Date _____
Free	Mfg. _____ Lot No. _____ Exp Date _____

Standard	Blank (mg/L)	Std. 1 Value (mg/L)	Std. 2 Value (mg/L)	Std. 3 Value (mg/L)	ICV & CCV results should fall within given tolerance limits given with each standard kit.
ICV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail
CCV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail

Standard	Blank (mg/L)	Std. 1 Value (mg/L)	Std. 2 Value (mg/L)	Std. 3 Value (mg/L)	ICV & CCV results should fall within given tolerance limits given with each standard kit.
ICV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail
CCV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail

Total and Free Chlorine

Collection			Analysis						
Location	Date	Time	Total Chlorine (mg/L)	Date	Time	Free Chlorine (mg/L)	Date	Time	Total Chloramines Calculated (mg/L)

Speciated Chloramines

Collection			Analysis						
Location	Date	Time	Date	Mono (NH_2Cl) (mg/L)	Time	Di ($NHCl_2$) (mg/L)	Time	Tri (NCl_3) (mg/L)	Time

Notes & Comments

Attach additional notes if necessary.

Lead Technician Signature: _____ Date: _____

Form Revised 12/30/2013

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ATTACHMENT 14E

Pace Analytical® Field Services Division

Chlorine Calibration Verification Sheet

Page _____ of _____

Technician _____ Meter Make _____ ID. _____

Low Standard

Mfg. _____

Lot No. _____

Exp Date _____

High Standard

Mfg. _____

Lot No. _____

Exp Date _____

Reagents

Total - Ampul	Mfg. _____ Lot No. _____ Exp Date _____
Total - Powder	Mfg. _____ Lot No. _____ Exp Date _____
Free	Mfg. _____ Lot No. _____ Exp Date _____

	Zero Std. (mg/L)	Std. 1 Value (mg/L)	Std. 2 Value (mg/L)	Std. 3 Value (mg/L)	ICV & CCV results should fall within given tolerance limits given with each standard kit.
Standard					
ICV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail
CCV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail

	Zero Std. (mg/L)	Std. 1 Value (mg/L)	Std. 2 Value (mg/L)	Std. 3 Value (mg/L)	ICV & CCV results should fall within given tolerance limits given with each standard kit.
Standard					
ICV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail
CCV	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Meter Value (mg/L)	Date _____ Time _____ Pass / Fail

Notes and Comments

Attach additional notes if necessary.

Lead Technician Signature: _____ Date: _____

Revised 2/29/2016

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ATTACHMENT 14F

Pace Analytical® Field Services Division

Turbidity Calibration and Verification Sheet

Technician(s) _____ Date _____ Page ____ of ____

Meter Make _____ Meter ID _____

	Std. 1 Value (NTU)	Std. 2 Value (NTU)	Std. 3 Value (NTU)	Std. 4 Value (NTU)	Standard Info
STANDARDS					Mfg. _____ Lot No. _____ Exp. Date _____

	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	
IC					IC Time _____ Initials _____ IC: Pass / Fail

	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	
ICV					ICV Time _____ Initials _____ ICV: Pass / Fail

ICV acceptable if ± 5% of standard value.

	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	Meter Value (NTU)	
CCV					CCV Time _____ Initials _____ CCV: Pass / Fail
CCV					CCV Time _____ Initials _____ CCV: Pass / Fail
CCV					CCV Time _____ Initials _____ CCV: Pass / Fail

CCV acceptable if ± 5% of standard value.

Comments _____

_____

_____

_____

Lead Technician Signature: _____ Date: _____

Form Revised: 11/14/2013

Attach additional notes if necessary.

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QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services – Field Services Division
1700 Elm Street SE
Minneapolis, MN 55414
(612) 607 - 1700

Effective date of last signature

APPROVAL

Donald B. Stock, QEP, General Manager, Technical Director
(612) 607-6370

Date 9/19/17

Richard A. Smith, Quality Assurance Officer
(612) 607-6367

Date 9/19/17

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COVER PAGE CONTINUED

DOCUMENTATION OF PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
	Quality Assurance Manager	September 19, 2018

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Table of Contents

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1.0 INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"

Pace Analytical Services LLC - Mission Statement

1.1 Introduction to Pace

Pace Analytical Services, LLC (Pace) is a privately held, legally identifiable, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy, radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2 Statement of Purpose

To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3 Quality Policy Statement and Goals of the Quality System

1.3.1 Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with applicable regulatory standards, such as NELAC, ISO 17025, and ASTM and is in accordance with the stated methods and customer requirements. Pace has developed and implemented a consistent quality policy and system that defines and documents its quality policies, quality objectives, and commitment to quality in each of its laboratories and service centers. This quality system enables it to continually improve and monitor its ability to deliver its scope of services. The overall objective of this quality system is to provide reliable data through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.3 Pace FSD operates under and maintains its documented quality system. The quality policy is disseminated by the Pace FSD Quality Assurance Office and is understood, implemented, and maintained at all levels within Pace FSD. Pace management demonstrates its commitment to quality by providing the resources, including facilities, equipment, and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system.

1.3.4 All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

1.3.5 All Pace personnel comply with and carry out activities to meet the requirements of all current applicable state, federal, and industry standards, and are required to perform all tests in accordance with regulatory authorities, stated methods and customer requirements.

1.4 Pace Analytical Services Core Values

1.4.1 The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**
- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5 Code of Ethics and Standards of Conduct

1.5.1 Code of Ethics:

1.5.1.1 Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2 Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3 Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4 Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources.

1.5.2 Standards of Conduct

1.5.2.1 Data Integrity

The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2 Confidentiality

Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for a period of two years thereafter.

Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and client information, inventions, materials composition, etc.

1.5.2.3 Conflict of Interest

Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3 Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4 Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5 Compliance: All employees are undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethics Policy statement.

1.6 Anonymous Compliance Alertline

1.6.1 An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environmental where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2 Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3 Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4 Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5 Website: www.lighthouse-services.com/pacelabs.

1.6.6 Email: reports@lighthouse-services.com (must include company name with report).

1.7 Organization

1.7.1 Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure. Pace Analytical Services, LLC - Minnesota Field Services Division (Pace FSD) is clearly defined as a stand-alone organization within the Pace system so that confidence in its judgment and integrity is maintained at all times. Pace is organized so its facilities and resources meet the requirements of relevant regulatory standards, such as NELAC, ISO 17025, and ASTM.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. A General Manager (GM) directly manages the Field Services Division. The Quality Assurance Officer (QAO) reports directly to the General Manager or to the highest level of local management, however named, that routinely makes day-to-day decisions regarding operations. The QAO also receives guidance and direction from the corporate Director of Environmental Quality.

1.7.4 The General Manager bears the responsibility for Pace FSD operations and serves as the final, local authority in all matters. In the absence of the General Manager (and an Assistant General Manager), the Quality Assurance Officer serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the GM until the GM is available to resume the duties of the position. In the absence of the GM and QAO, management responsibility is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the GM or QAO. The most senior department manager in charge may include the Administrative Business Manager at the discretion of the General Manager.

1.7.5 A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The General Manager or Quality Assurance Officer has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.6 The Quality Assurance Officer has the responsibility and authority to ensure the Quality System is implemented and followed at all times. The QAO has access to the highest levels of management at which decisions are made on policies affecting Pace FSD. The relationships between the quality manager, technical operations, and support services are clearly defined. In circumstances where the established level of quality is not being met or personnel are not following the policies set forth in this Quality Assurance Manual, the Quality Assurance Officer has the authority to halt operations should he or she deem such an action necessary. The QAO will immediately communicate the halting of operations to the GM and keep him or her posted on the progress of corrective actions. In the event the GM and QAO are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.7. The lab is required to appoint deputies for key managerial personnel. These deputies must be documented for auditing purposes.

1.7.8 The technical staff of Pace FSD is generally organized into the following functional groups:

- Air Operations
- Field Analytical Operations
- Water Operations

Appropriate support groups are present within each functional group.

1.7.9 The organizational structure for Pace FSD is listed in Attachment II. In the event of a change in General Manager, Quality Manager, or Technical Director, Pace FSD will notify its accrediting authorities per their required individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next schedule revision.

1.8 Pace FSD Job Descriptions and Functions

Pace FSD is organized so that staff members are aware of both the extent and limitations of their responsibilities, and to provide adequate supervision of technical staff by persons familiar with relevant methods and sampling procedures. Pace FSD authorizes personnel to operate particular types of equipment, perform certain types of testing and calibration, to issue reports, and provide opinions and interpretations of results. Due to the small size of Pace FSD, personnel may perform job functions for which the individual is qualified but are not directly specified in his or her job description. Current job descriptions for all personnel, including managerial, technical, and key support personnel are maintained in appropriate administrative files. Basic job functions are summarized below.

1.8.1 Senior General Manager

1. Oversees all functions of all operations within their designated region;
2. Oversees development of local General Managers within their designated region;
3. Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
4. Oversees preparation of budgets and staffing plans for all operations within their designated region;
5. Ensures compliance with all applicable state, federal and industry standards.
6. Works closely with Regional Sales Management.

1.8.2 General Manager (Local)

1. Oversees all functions of Pace FSD.
2. Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation.
3. Prepares budgets and staffing plans.
4. Monitors the Quality System and advises the Quality Assurance Officer accordingly.
5. Ensures compliance with all applicable state, federal, and industry standards.

1.8.3 Quality Assurance Officer

1. Oversees implementation and operation of the Pace FSD Quality System and reports directly to the General Manager.
2. Ensures communication takes place at all levels within Pace FSD regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system.
3. Responsible for ensuring compliance with any accreditation standards, such as ISO/IEC 17025, ASTM, etc.
4. Monitors Quality Assurance policies and Quality Control activities to ensure established standards of quality are achieved and is responsible for reporting this level of compliance to the GM on a quarterly basis.

5. Maintains records of quality control data and evaluates data quality.
6. Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or client representatives.
7. Reviews and maintains records of proficiency testing results.
8. Maintains quality documents and the document control system.
9. Assists in development and implementation of appropriate training programs.
10. Provides technical support to operations regarding methodology and project QA/QC requirements.
11. Maintains certifications from federal and state programs.
12. Ensures compliance with all applicable state, federal, and industry standards.
13. Maintains training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
14. Monitors corrective actions.
15. Maintains the currency of the Quality Assurance Manual.

1.8.4 Technical Director

1. Responsible for overall technical operations, including providing technical guidance in the review, development and validation of new methodologies.
2. Demonstrate competence in air emissions testing.
3. Monitors the standards of performance in quality assurance and quality control data.
4. Monitors the validity of analyses performed and data generated.
5. Reviews tenders, contracts, and QAPPs to ensure the facility can meet the data quality objectives for any given project
6. Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
7. Provides technical guidance in the review, development and validation of new methodologies.

1.8.5 Business Development Manager

1. Responsible for developing growth and identifying market potentials.
2. Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with clients.
3. Responds to inquiries or complaints from customers and regulatory agencies.
4. Interfaces between clients and personnel to achieve client satisfaction.
5. Ensures compliance with all applicable state, federal, and industry standards.

1.8.6 Department Manager

1. Oversees the day-to-day production and quality activities of their assigned department.
2. Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied.
3. Assesses data quality and takes corrective action when necessary.
4. Approves and releases technical and data management reports.
5. Ensures compliance with all applicable state, federal, and industry standards.

1.8.7 Administrative Business Manager

1. Responsible for financial and administrative management for Pace FSD.
2. Provides input relative to tactical and strategic planning activities.
3. Organizes financial information so the facility is run as a fiscally responsible business.
4. Works with staff to confirm appropriate processes are in place to track revenues and expenses.
5. Provide ongoing financial information to the General Manager and the management team so they can better manage their business.
6. Utilizes historical information and trends to accurately forecast future financial positions.

7. Works with management to ensure that key measurements (mileposts) are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios.
8. Works with General Manager to develop accurate budget and track on an ongoing basis.
9. Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments.
10. Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.8 Project Manager

1. Coordinates daily activities including taking orders, reporting data and analytical results.
2. Serves as the primary technical and administrative liaison between customers and Pace FSD.
3. Communicates with operations staff to update and set project priorities.
4. Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.).
5. Works with customers and other appropriate staff to develop project statements of work or resolve problems of data quality.
6. Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers, and maintain client records.
7. Mediation of project schedules and scope of work through communication with internal resources and management.
8. Responsible for preparing routine and non-routine quotations, reports, and technical papers.
9. Interfaces between customers and management personnel to achieve client satisfaction.
10. Manages large-scale complex projects.
11. Supervises less experienced project managers and provides guidance on management of complex projects.

1.8.9 Client Coordinator

1. Performs project initiation and scheduling.
2. Prepares routine quotations and reports.
3. Coordinates personnel, supplies, and equipment resources.
4. Coordinates sample analysis with laboratories.
5. Performs oversight of project set-up, sample receipt, project status, and ensuring samples are collected, analyzed, documented, reported, and invoiced in conformance with client requirements.
6. Provides client follow-up to include client satisfaction and additional services.

1.8.10 Project/Support Coordinator

1. Responsible for preparation of project specifications and provides technical/project support.
2. Coordinates project needs with other department sections and assists with proposal preparation.
3. Prepares routine proposals and invoicing.
4. Responsible for scanning, copying, assembling, and binding final reports.
5. Purchases necessary supplies and services.
6. Other duties include filing, maintaining forms, processing mail, maintaining training database and data entry.

1.8.11 Field Technician

1. Prepares and samples according to published methods, Pace Quality Assurance Manual and/or customer directed sampling objectives;
2. Capable of the collection of representative environmental or process related air samples;
3. Use computer software to compile, organize, create tables, create graphics and write test reports;
4. Reviews project documentation for completeness, method compliance and contract fulfillment;
5. Train less experienced environmental technicians and provide guidance on sampling and analysis;

6. Responsible for project initiation and contact follow-up;
7. Develop sampling plans and prepare test plan documents.

1.8.12 Field Analyst

1. Analyzes field samples according to published methods, procedure manuals, Quality Assurance Manual, and/or client directed sampling objectives.
2. Capable of the collection and analysis of representative environmental or process related air samples.
3. Proficient in a variety of analytical tests; specifically on-site gas-phase organic and inorganic compounds by extractive fourier transform infrared spectroscopy (FTIR).
4. Train less experienced staff and provide guidance on FTIR sampling and analysis.
5. Assist in reporting tasks and project management responsibilities.
6. Perform back-up support for manager tasks, such as reporting needs and client concerns.

1.8.13 Safety/Chemical Hygiene Officer

1. Maintains the Chemical Hygiene Plan.
2. Plans and implements safety policies and procedures.
3. Maintains safety records.
4. Organizes and/or performs safety training.
5. Performs safety inspections and provides corrective/preventative actions.
6. Assists personnel with safety issues.

1.8.14 Sales Associate

1. Meet sales goals, expand client list, and increase revenues.
2. Learn and perform various computer software programs for analysis of field samples (i.e., identify market opportunities through use of internet, associations, etc.)
3. Research government regulations for Air, Wastewater, and Groundwater.
4. Actively participate in trade shows, industry associations, sales presentations, and professional organizations.
5. Conduct mailings, make cold calls, and generate leads.
6. Prepare quotes and proposals and track sales calls and quotes for reporting purposes.
7. Collaborate with field and lab staff for project pricing and scheduling.

1.8.15 Program Director/Hazardous Waste Coordinator (or otherwise named)

1. Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
2. Maintains complete records of waste disposal including waste manifests and state reports;
3. Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.;
4. Conducts a weekly inspection of the waste storage areas of the laboratory.

1.8.16 Equipment Services Specialist

1. Evaluates field equipment for proper functioning and diagnoses errors or other technical problems to determine solutions to maintain instruments in good operating condition.
2. Performs scheduled and unscheduled maintenance, calibrations, and performance validations of field equipment according to documented procedures.
3. Maintains complete documentation of maintenance, calibration, and performance verification activities.
4. Packages, labels, and ships equipment to outside maintenance or calibration services, when needed.

1.9 Orientation and Training

Pace has a policy and procedures for providing training to personnel. Requirements for the education, training, and skills of Pace FSD employees are defined. Training is relevant to an employee's present and anticipated tasks and is organized so that staff members are aware of both the extent and limitations of their responsibilities. Pace FSD implements policies and procedures to identify training needs and evaluate the effectiveness of training. Each employee performing specific tasks is qualified on the basis of appropriate education, qualification, training, experience, examination, and demonstrated skills. Pace FSD provides appropriate supervision of trainees and all technical staff by personnel experienced in methods and procedures relevant to the type of work being performed and/or evaluation of results.

1.9.1 Much of Pace FSD employee training is managed through a web-based training system. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by trainer
- Training checklists/worksheets (e.g., from LMS new hire workbooks)
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Reading Standard Operating Procedures (SOPs) and Procedure Manuals (PMs)
- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan
- Core training modules
- Quality system training modules
- Data Integrity/Ethics training
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

1.9.2 Department Managers/Supervisors are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision, and the date on which competency was confirmed. The files also include documentation of capability (see Section 3.4 for details on Demonstration of Capability requirements). Training documentation files for each staff member are maintained by the Pace FSD Quality Office in hardcopy format or within the web-based training system.

1.9.3 All procedures and training records are maintained and available for review during facility audits. Additional information can be found in the most current revision of Pace FSD SOP *Orientation and Training*, S-FSD-Q-005, or its equivalent replacement.

1.10 Safety and Waste

It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his or her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the Safety Manual and Chemical Hygiene Plan. Pace FSD is able to provide documentation or otherwise demonstrate upon request compliance with applicable local, state, and federal requirements governing health and safety, transportation, and other relevant requirements.

1.11 Security and Confidentiality

Security is maintained by controlled access to facility buildings. Exterior doors to facility buildings remain either locked or continuously monitored by Pace staff. Pace FSD controls access to and use of areas affecting the quality of tests and calibration activities.

Additional security is provided where necessary, (e.g., specific secure areas for sample, data and client report storage), as requested by customers or cases where national security is of concern. These areas are lockable within the facilities, or are in secure offsite storage. Access is limited to specific individuals or their designees.

All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12 Communications

Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

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2.0 SAMPLE HANDLING

Pace implements procedures for chain-of-custody, transportation, receipt, handling, protection, storage, retention, and disposal of samples, sampling media, and calibration materials to protect the integrity of the sample or calibration material and to protect its interests. Pace FSD has procedures and appropriate facilities to avoid deterioration, loss or damage to sample, sampling media, or calibration material during storage, handling, and preparation.

2.1 Project Initiation

Prior to accepting new work, Pace FSD reviews its performance capability to confirm that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Client needs, certification requirements, and data quality objectives are defined and appropriate environmental sampling procedures or test methods are assured to meet project requirements by project administrators or sales representative. Team members, sales, and project management staff review current equipment capacity, personnel availability and training, procedures capability, and projected work load. Together, the department management staff and sales management determine whether or not Pace FSD can accept the new project via written, electronic, and/or verbal correspondence.

Additional information regarding specific procedures for reviewing new work requests can be found in the most current revision of the Pace FSD SOP S-FSD-Q-022 *Requests, Proposals, Contracts, and Project Initiation* or its equivalent replacement.

2.2 Sampling Materials and Support

Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VI. Note that all analyses listed are not necessarily performed at all Pace laboratories and additional laboratory analyses not included in these tables may be performed. Pace may provide pick-up and delivery services to customers when needed.

2.3 Field Services

Pace Analytical has a large Field Services Division based in their Minneapolis facility as well as limited field service capabilities in some of the other facilities. Field Services provides comprehensive nationwide service with offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services - FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater, and Drinking Water Monitoring
- Stormwater and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

The role of Pace FSD within its parent company is clearly defined, including a unit specific quality program. Pace Field Services operates under the Pace Corporate Quality System with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.4 Chain of Custody

2.4.1 A chain-of-custody (COC) provides the legal documentation of sample custody from time of collection to completion of analysis.

2.4.2 Field personnel or client representatives must complete a chain-of-custody form for every sample in custody for transportation, storage, transfer to another party, or check in for analysis, either internally or by another laboratory. Upon collection or receipt of samples from a client representative, field personnel record any abnormalities or deviations from normal or specified collection procedures or conditions. Samples are submitted to the designated laboratory following any handling instructions provided with the item and accompanied by a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If a sample does not conform to the description provided on a chain-of-custody, if information recorded is not specified in sufficient detail, or if there is doubt as to the suitability of a sample, Pace then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.4.3 The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the original entry and initialing and dating the change. All transfers of samples are recorded on the chain-of-custody in the "relinquished" and "received by" sections. All information except signatures is printed.

2.4.4 Additional information can be found in procedure manuals or in the most current revision of Pace FSD SOP *Documentation of Field Activities,* S-FSD-Q-004, or its equivalent replacement.

2.5 Sample Collection and Acceptance Policy

2.5.1 In accordance with regulatory guidelines, Pace complies with the following sample collection policy for all samples.

2.5.2 If the samples do not meet the criteria outlined below, Pace FSD is required to document all non-compliances, contact the customer, and either re-collect the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the customer.

2.5.3 Sample Collection and Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents, that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.

- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- Have started the cooling process, when necessary, as soon as possible after collection.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2oC of the required temperature, or within the method-specified range. For samples with a required temperature of 4oC, samples with a temperature ranging from just above freezing to 6oC are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
- Samples for drinking water analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).
- Some specific clients may require custody seals. For these clients, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

2.5.4 Upon sample receipt at Pace laboratories, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.6. Sample Log-in

A sample submitted to a Pace FSD laboratory for analysis is inspected with all sample information on the chain-of-custody is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 12:01 AM as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

Laboratory sample labels are printed from the LIMS and affixed to each sample container.

Additional information can be found in *SOT-ALL-C-001 Sample Management* or its equivalent revision or replacement.

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2.7 Sample Storage

2.7.1 Storage Conditions

All samples and sample fractions are stored in the manner specified by applicable procedure or method, applicable regulatory requirement, or client specifications until submission to the designated analytical laboratory or until disposal. All extracts, leachates and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.7.1.1 Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross-contamination. When a sample must be stored or conditioned under specified environmental conditions, these conditions are maintained, monitored, and recorded. Pace FSD transports samples with ice, when needed, to obtain the appropriate cooling temperature specified by parameter preservation requirements.

2.7.2 Temperature Monitoring

Samples are taken to an appropriate storage location (ambient, refrigerator, freezer) for temporary holding prior to submission to the designated analytical laboratory. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

The temperature of each refrigerated storage area is maintained at ≤6°C (but above freezing) unless state, method, or program requirements differ. The temperature of each freezer storage area is maintained at <-10°C unless state, method, or program requirements differ. Additional information, including corrective actions for temperatures outside of acceptance limits, can be found in the most current revision of Pace FSD SOP *Monitoring of Walk-in Coolers and Ovens*, S-FSD-E-001, or its equivalent replacement.

2.7.3 Hazardous Materials

Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.7.4 Foreign/Quarantined Soils

Foreign soils and soils from USDA regulated areas must be adequately segregated to enable proper sample disposal. The USDA requires these samples to be treated by an approved procedure. Additional information regarding USDA regulations and sample handling can be found in the SOP for Regulated Soil Handling S-ALL-S-003, or its equivalent revision or replacement.

2.8 Sample Protection

Pace facilities are operated under controlled access to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted at all times.

Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken. Samples will be secured in vehicles when left unattended.

Upon client request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. In these special circumstances, Pace FSD will arrange for storage and security that protect the

condition and integrity of the secured items or portions concerned. For example, some projects may require internal chain-of-custody protocols.

Pace FSD ensures, to the extent practical, that environmental conditions do not invalidate results or adversely affect the required quality of testing procedures. Any environmental conditions that can affect the results of a test or calibration are documented on field data sheets. Pace FSD, to the extent practical, avoids concurrent activities that are incompatible with collection or analysis of quality data and takes measures to avoid cross-contamination. Any excess portion of reagent, standard, or sample poured from its original container is not returned to its container but rather disposed of in the appropriate waste receptacle to prevent contamination of the remaining material. Measures to ensure good housekeeping practices in all testing or analysis locations are conducted and special procedures are taken when necessary.

2.9 Subcontracting of Work

Pace FSD makes every effort to satisfy client requests and relevant regulatory requirements when performing work. When subcontracting work becomes necessary, whether inside or outside the Pace network, a competent subcontractor that complies with all accreditations and regulations applicable to the project is used. When evaluating a subcontractor for competence, Pace FSD maintains a register of all subcontractors it uses and a record of evidence of compliance to applicable regulations for the work in questions. When necessary, Pace FSD may evaluate subcontractors through audits, quality control measures, or proficiency testing. Work performed under specific protocols may involve special considerations. Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories.

Customers are notified in writing of the intention to subcontract any portion of the work in question. When appropriate, Pace FSD obtains client approval of subcontracting. Pace FSD acknowledges responsibility to the client for subcontractor's work, except in instances where the client or regulatory authority specifies the subcontractor to be used. Work performed under specific protocols may involve special considerations.

Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

Contact and preliminary arrangements are made between the Pace Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract agreement include:

- Method(s) of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

All subcontracted sample data reports are sent to the Pace FSD Project Manager, who will provide a copy of the subcontractor's report to the client when requested.

Any Pace Analytical work sent to other labs within the Pace network is handled as subcontracted work and all final reports are labeled clearly with the name of the laboratory performing the work. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

Additional information can be found in the most current revision of Pace FSD SOP *Subcontracting of Work*, S-FSD-Q-011, or its equivalent replacement.

2.10 Sample Retention and Disposal

2.10.1 Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.10.2 Typically, EPA Method 5 samples are retained for a minimum of one year; the minimum time for all other samples is 45 days from receipt of the samples. Samples requiring thermal preservation may be stored at ambient temperature when the hold time is expired, the report has been delivered, and/or allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.10.3 Non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangement will be made for disposal by an approved contractor.

2.10.4 Additional information can be found in SOP SOT-ALL-W-002 *Waste Handling and Management* and SOP SOT-ALL-C-001 *Sample Management* or the equivalent revisions or replacements.

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3.0 FIELD SAMPLING AND ANALYTICAL CAPABILITIES

3.1 Method Sources

Pace FSD is capable of collecting and analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Pace FSD uses test methods, including methods for sampling, that meet the needs of the client and which are appropriate to the objectives of the project. Pace FSD uses appropriate methods and procedures for all testing performed including sampling, analysis, handling, transport, storage and preparation of items to be tested or calibrated, and an estimation of the measurement uncertainty, where appropriate, as well as statistical techniques for analysis of test data. When practical, methods established in international, regional, or national standards are used. The latest valid editions of methodologies are applied (unless inappropriate or impossible to use) from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, A2LA, Standard Methods, and State Agencies. Section 11 is a representative listing of general analytical protocol references. Pace discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the collection and analysis of samples. When necessary, Pace FSD supplements methods with additional detail to ensure consistent, accurate application.

In the event of a client specific need, instrumentation constraint or regulatory requirement, Pace reserves the right to use valid versions of methods that may not be the most recent edition available.

Methods for air emission testing performed for compliance purposes are currently defined by applicable regulations. Alternative or deviations from these methods are detailed in test protocol, or test report, along with any authorizations for the alternatives or deviations.

Project specific information is outlined in a test plan or equivalent document. A test plan, or equivalent document, is used for all Pace FSD projects and is the primary source of information on testing and quality procedures for a project. Test plans are communicated to all personnel participating in the test project prior to the start of the project. All procedures and activities specified in the test plan meet the requirements of applicable regulatory standards, and, at a minimum, address the following:

- Objectives and summary of test
- Description of test location, operating conditions, and process to be tested
- Description of test matrix
- Site address and sampling locations
- Methods/procedures to be used, including any alternatives or deviations from applicable regulations
- Major test equipment to be used
- Number of tests to be performed and sampling duration of each monitoring period
- Process data to be collected and by whom
- Applicable QC procedures including audits and field blanks
- Report format and requirements, including any due dates
- Facility specific entry and safety requirements
- Responsibilities of test personnel
- Tentative test schedule

3.2 Procedure and Method Documentation

The procedures and methods used by Pace FSD personnel are documented in Standard Operating Procedure (SOP) and Procedure Manuals (PMs) to ensure consistency throughout the sample and field data collection process. SOPs contain detailed steps regarding policies and procedures routinely performed by Pace FSD personnel within several functional groups. PMs contain pertinent information required to successfully perform the primary duties pertaining to a specific functional group within Pace FSD. SOPs and PMs may be supplemented by other training materials that further detail how activities are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

The required contents for SOPs and PMs are specified in the most current revision of Pace FSD SOP, *Preparation of Standard Operating Procedures and Procedure Manuals*, S-FSD-Q-001, or its equivalent replacement.

3.3 Analytical Method Validation

In some situations, Pace FSD develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, or when Pace FSD develops a method or modifies a method, Pace FSD validates the method prior to applying it to client samples and takes appropriate actions to ensure the applicable requirements are addressed before and during the execution of the method. Introduction of test and calibration methods developed by Pace FSD are systematically planned and assigned to qualified personnel equipped with adequate resources. Method validity is established by meeting the requirements of any relevant regulatory authority and criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. Pace FSD documents these requirements, the validation procedure, the results obtained, and a statement as to the usability of the method. The minimum requirements for method validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest. Any non-standard method used for a project will be subject to agreement with the client and any relevant regulatory authority and will include a clear specification of the purpose and client requirements. These methods will be described in test protocol and reported in the same ways as standard methods with any validation data included in the test protocol.

3.4 Demonstration of Capability (DOC)

Pace FSD insures the competence of those personnel who operate specific equipment, perform tests and/or calibrations, evaluate results, and sign calibration certificates and testing reports. Employees complete Demonstration of Capability (DOC) for major job functions prior to performing the operation independently. Demonstration of capability is evaluated by experienced personnel with documentation maintained in the respective staff member's training files. The trainer will determine the need and extent of training; the trainer will assess and document completed training and capability by signature on appropriate training document. Unacceptable performance will require further training and demonstration of capability for relevant procedures.

Pace FSD collects and documents performance data from all relevant sources. When an established, published, or validated test method that has not been previously performed by Pace FSD is required for a project, Pace FSD takes appropriate actions to ensure the applicable requirements are addressed before and during the execution of the method. Experienced personnel with competency in similar methods will perform the work after a thorough review of the selected method.

3.5 Regulatory and Method Compliance

Pace understands that expectations of our customers commonly include the assumption that field work and the results produced will satisfy specific regulatory requirements. Therefore, Pace attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the Chain of Custody submitted with samples.

Pace makes every effort to detect regulatory or project plan inconsistencies, based upon information from the client, and communicate them immediately to the client in order to aid in the decision-making process. All project plans are updated as development of the project proceeds with effective communication maintained amongst all personnel involved. Pace FSD informs the client when the method proposed by the client is considered to be inappropriate or out-of-date. Pace will not be liable if the client chooses not to follow Pace recommendations.

It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the work performed and the usability of data produced by our facilities. Customers are notified within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

3.6 Pace FSD Certifications

3.6.1 Accreditation

Pace Analytical Services, LLC – Field Services Division has three national accreditations:

- ASTM D7036 – Standard Practice for Competence of Air Emission Testing Bodies (AETB)
- The NELAC Institute (TNI) – General Requirements for Field Sampling and Measurement Organizations (FSMO)
- ISO 17025 – General Requirements for the Competence of Testing and Calibration Laboratories.

Compliance to these standard provides assurance of producing on a consistent basis accurate, reliable field testing data with documented quality.

3.6.2 Qualified Individuals

Pace Analytical Services, LLC – Field Services Division encourages its employees to obtain Qualified Source Testing Individual (QSTI) certification. The QSTI program, developed by the Source Evaluation Society (SES), allows individuals to demonstrate and be recognized for his or her knowledge and experience of stationary source air emissions testing. The QSTI program satisfies certain accreditation standard requirements, such as those for ASTM.

Pace FSD provides Qualified Individuals to oversee and supervise test projects. Projects are scheduled so that only Qualified Individuals supervise a test appropriate to their certification. At least one Qualified Individual who is qualified in each method employed for that project is on-site at all times during the project. The qualification credentials of each Qualified Individual are available on-site. Pace FSD maintains a signed statement by all its Qualified Individuals that all test projects performed under his or her supervision will conform to Pace FSD's Quality Assurance Manual and to the requirements of ASTM D-7036.

4.0 QUALITY CONTROL PROCEDURES

4.1 Quality Control Samples

4.1.1. The quality control samples described in this section are analyzed as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or final reports must be appropriately qualified.

4.1.2. Quality control samples must be processed in the same manner as associated client samples.

4.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

4.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the Quality Assurance Officer.

4.2 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system. The method blank is a sample of a matrix similar to the batch of associated samples that is free of the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

4.3 Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis. The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes. A Laboratory Control Sample may also be referred to as Laboratory Fortified Blank, Spiked Blank, or QC Check Sample.

4.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch unless the MS is actually used as the LCS. A matrix spike (MS) is a sample prepared by adding a known quantity of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

A Matrix Spike Duplicate (MSD) is a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte. An MSD may also be referred to as Spiked Sample Duplicate or Fortified Sample Duplicate.

Client samples specifically designated as Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples, or samples with adequate volume or weight, are spiked and are prepared and analyzed in the same manner as the original samples. An MS/MSD set is processed at a frequency specified in a particular method or as determined by a specific client.

4.5 Surrogates

Surrogates are compounds unlikely to be found in environmental samples that reflect the chemistry of the target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

4.6 Sample Duplicate

A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific client. The sample and duplicate are evaluated against the method or laboratory-derived criteria for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

4.7 Internal Standards

Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

4.8 Field Blanks

Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. A clean sample container with appropriate preservative, if any, is filled in the field and is then analyzed as a normal sample. Field blanks may also be referred to as rinseate blanks or equipment blanks. The laboratory analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.9 Trip Blanks

Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the laboratory. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.10 Limit of Detection (LOD)

Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent.

4.11 Limit of Quantitation (LOQ)

A Limit of Quantitation (LOQ) is the minimum level, concentration or quantity of a target variable that can be reported with a specified degree of confidence.

4.12 Estimate of Measurement Uncertainty

4.12.1 Pace FSD can provide an estimation of uncertainty associated with testing methods it performs. The estimate quantifies the error associated with any given result at a 95% confidence interval. In the absence of a regulatory or client specific procedure, Pace FSD bases this estimation on the recovery data obtained from the Laboratory Control Samples approach or the Root Sum of Squares approach. When approved test methods with stated uncertainties are used, a reference to where an estimate of uncertainty or the method can be found is

sufficient. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived is contained in the most current revision of Pace FSD SOP *Estimation of Measurement Uncertainty*, S-FSD-Q-023, or its equivalent replacement.

4.12.2 The measurement of uncertainty is provided only on request by the client, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

4.13 Proficiency Testing (PT) Studies

Pace FSD participates in relevant and available third-party proficiency testing programs provided by organizations administering acceptable proficiency tests at a frequency sufficient to assure competence of all methods/procedures, materials/matrices, and activities on its Scope(s) of Accreditation. If external proficiency testing is not available or relevant, Pace FSD will assure the quality of activities through sufficient internal proficiency testing, performance evaluations, or other quality control checks. The results of any proficiency testing are used to assess the effectiveness of the quality program.

Additional information can be found in the most current revision of Pace FSD SOP *Proficiency Testing*, S-FSD-Q-021, or its equivalent replacement.

4.14 Rounding and Significant Figures

4.14.1 Rounding

Pace FSD uses the following guidelines for rounding numbers:

If the figure following the digit to be retained is less than five, that figure is dropped and the retained digits are not changed. (e.g., For three significant figures: 2.544 is rounded to 2.54.)

If the figure following the digit to be retained is equal to or greater than five, that figure is dropped and the last retained digit is rounded up. (e.g., For three significant figures: 2.546 is round to 2.55, and 2.545 is rounded to 2.55.

Data is compared to reporting limits and MDLs, if applicable, to determine if qualifiers are needed before a rounding step occurs.

Rounding performed in Excel and other computer programs will be carried out according to the software's standard settings.

4.14.2 Significant Figures

In general, Pace FSD reports data using significant figures to avoid ambiguity and to reflect the level of accuracy justified by the measurement techniques used to obtain the data. Results may be reported using a level of significant figures which take into account possible continued use of the data beyond our report.

Additional information can be found in the most current revision of Pace FSD SOP *Significant Figures and Rounding Rules*, S-FSD-Q-014, or its equivalent replacement.

5.0 DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1 Document Management

Additional information can be found in the most current revision of Pace FSD SOP Document Control and Management, S-FSD-Q-002, or its equivalent replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

Pace has established and maintains procedure for controlling and managing all documents of the quality system. These managed documents include instructions on the use and operation of all relevant equipment, on handling of equipment, and on the preparation of instruments used for testing, where the absence of such instructions could jeopardized the results of the work in question.

A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. Copies of all quality systems documentation provided to DoD for review must be in English.

Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 Document Numbering.

All managed quality documents relevant to the work of Pace FSD, including the quality manual, instructions, standards, manuals and reference data, are maintained current and are readily available to personnel. Document control procedures are sufficient to preclude the use of invalid or obsolete documents. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

SOPs, specifically, are available to all lab staff via the Learning Management System (LMS) which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Printing of electronic documents may be allowed in certain instances; the wording UNCONTROLLED WHEN PRINTED must be marked on any printed copy from an electronic file and be handled as an uncontrolled copy.

5.1.1 Quality Assurance Manual (QAM)

The Quality Assurance Manual - Field Services Division (QAM - FSD) describes all aspects of the quality system for Pace FSD. The QAM - FSD is based on the Quality Assurance Manual template distributed by the Corporate Environmental Quality Department. The local management personnel modify the necessary and permissible sections of the base template and then all applicable staff sign the Quality Assurance Manual. The Pace QAO is then in charge of distribution to employees, external customers, or regulatory agencies and maintaining a distribution list of controlled document copies. The QAM - FSD is reviewed on an annual basis by appropriate personnel and revised accordingly. This includes the incorporation of applicable changes from the Quality Assurance Manual template provided by the Corporate Quality Department.

5.1.2 Standard Operating Procedures (SOPs)

5.1.2.1 SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

5.1.2.2 All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

5.1.2.3 Additional information can be found in the most current revision of Pace FSD SOP *Preparation of Standard Operating Procedures and Procedure Manuals*, S-FSD-Q-001, or its equivalent replacement.

5.1.3 Procedure Manuals

5.1.3.1 Procedure Manuals document routine procedures and detailed descriptions of activities performed by a specific functional group within Pace FSD to ensure consistency throughout the sample and field data collection process. Procedure manuals may include procedures regarding equipment preparation and set-up, sampling procedures, sample handling, field analysis, and task chronology.

5.1.3.2 Procedure manuals are reviewed every two years at a minimum (a more frequent review may be required by state or federal agencies or customers). A review of the document does not necessarily constitute a re-issue of a new revision. Documentation of this review and any applicable revisions are made in the last section of each procedure manual. This provides a historical record of all revisions.

5.1.3.3 All copies of superseded procedure manuals are removed from general use and one copy of each procedure manual is archived for audit or knowledge preservation purposes. This ensures that all Pace FSD employees use the most current version of each PM and provides the Quality Assurance Officer with a historical record of each PM.

5.1.3.4 Additional information can be found in the most current revision of Pace FSD SOP *Preparation of Standard Operating Procedures and Procedure Manuals*, S-FSD-Q-001, or its equivalent replacement.

5.2 Document Change Control

Changes to managed documents are reviewed and approved in the same manner as the original review unless specifically designated otherwise. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

All controlled copies of the previous document are replaced with controlled copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised of a revision release and any necessary training is scheduled.

Additional information can be found in the most current revision of Pace FSD SOP *Document Control and Management*, S-FSD-Q-002, or its equivalent replacement.

6.0 EQUIPMENT AND MEASUREMENT TRACEABILITY

Pace FSD implements policies and procedures for the selection, purchasing, reception, and storage of reagents, services, and consumable supplies it uses that affect the quality of the work it performs. Purchased supplies, reagents, and consumable materials affecting the quality of work performed are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the test or calibration. Purchasing documents for items affecting the quality of work performed contain data indicating the services or supplies ordered. These documents are reviewed and verified that the item received was as ordered and complies with any specified requirements. Records of actions taken to check compliance is maintained.

Each Pace facility has access to all items, sampling, measurement, and test equipment, including support equipment, required for the correct performance of the relevant analytical testing or field procedures performed Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. Pace FSD has established a calibration program for key quantities or values of the instruments where these properties have a significant effect on the results of a project. This section details some of the procedures necessary to maintain traceability and perform proper calibration of instrumentation and support equipment. See Attachment IV for a list of equipment used at Pace FSD.

6.1 Standards and Traceability

Pace FSD uses reference materials traceable to certified reference materials when possible. Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials are carried out, as far as technically and economically practicable, in accordance with documented procedures and schedules. Pace FSD confirms or establishes a new calibration at an interval established by relevant regulations. Each Pace facility retains all pertinent information for standards, reagents and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation and use.

Upon receipt, all purchased standard reference materials when not assigned a lot number are recorded into a standard logbook or database, and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, preparation date, expiration dates, preparer's initials, and a unique identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

All prepared standard or reagent containers include the new solution identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

For containers that are too small to accommodate labels that list all of the above information associated with a standard, the minimum required information will be Pace standard ID, concentration, and expiration date. This assures that no standard will be used past its assigned expiration date.

If a second source standard is required to verify an existing calibration or spiking standard, this standard is obtained from a different manufacturer or from a different lot unless client specific requirements state otherwise.

Pace FSD uses procedures for safe handling, transport, storage, and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

Additional information concerning standards and reagent traceability can be found in the most current revision of Pace FSD SOP *Standard and Reagent Management and Traceability*, S-FSD-Q-018, or its equivalent replacement.

6.2 General Analytical Instrument Calibration Procedures

Pace FSD uses an established program and procedures to calibrate its equipment. This program includes a schedule of calibration and appropriate methods to insure only properly calibrated equipment is used in test projects. All equipment and instruments used for tests and/or calibrations, including subsidiary measurements, having a significant effect on the accuracy or validity of the test or calibration are calibrated or performance checked before being placed into service to ensure proper functioning and verify that relevant standard specifications and project requirements are met.

All equipment and applicable software used for testing, calibration, or sampling is capable of achieving the accuracy level required of the project and complies with any relevant regulatory specifications. Calibrations and performance checks are performed using established methods at scheduled intervals sufficient to ensure the equipment continually meets specification in cases where relevant regulations do not specify a calibration interval. Pace ensures that equipment is operated by trained and authorized personnel.

All calibrations are performed by, or under the supervision of, experienced personnel using established methods at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated. Pace FSD ensures that when it uses equipment outside its permanent control all necessary requirements and standards for the work in question are met. Relevant records are obtained with any rented or loaned equipment.

Analyte specific calibrations are performed by the method. When a specific method is unavailable, a similar method is employed to develop calibration and reporting limit procedures.

Pace FSD takes precautions to ensure malfunctioning or inoperative equipment is not used on a project. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets necessary specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported in accordance with the calibration laboratory's recommendations to prevent breakage.

In the event that recalibration of a piece of equipment indicates it may have been malfunctioning during the course of sampling or analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the Quality Assurance officer. If the investigation casts doubt on the validity of results already transmitted to the client, the client is notified in writing within 30 days. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

6.3 Support Equipment Calibration and Verification Procedures

All support equipment is calibrated or verified over its entire range of use. When required by the procedure, method, or project, references traceable to national standards maintained by NIST are used for calibration or performance verification. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The facility maintains records to demonstrate the correction factors applied to working thermometers.

On each day of use, walk-in coolers and ovens are checked in the expected use range with references traceable to national standards maintained by NIST in order to ensure the equipment meets relevant specifications. These checks are documented appropriately. Other support equipment is checked using documented procedures prior to use to ensure relevant specifications are met.

6.3.1 Analytical Balances

Stationary balances are calibrated by an external service provider on an annual basis using reference weights traceable to national standards maintained by NIST and undergo performance verification by Pace FSD staff on each day of use with reference weights traceable to national standards maintained by NIST bracketing the range of use. Stationary balance calibration weights are ASTM Class 1 or other class weights that have been calibrated against a standard weight traceable to national standards maintained by NIST and are re-certified every five years, at a minimum, against a reference traceable to national standards maintained by NIST. Some accrediting agencies may require more frequent checks.

Mobile balances undergo performance verification by Pace FSD on an annual basis using reference weights traceable to national standards maintained by NIST and do not require performance verification prior to use.

If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the appropriate logbook and/or is maintained on file in the Pace FSD Quality Office.

Additional information can be found in the most current revision of Pace FSD SOP *Performance Verification and Calibration of Analytical Balances*, S-FSD-E-003, or its equivalent replacement.

6.3.2 Thermometers

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers traceable to national standards maintained by NIST are provided for initial calibration and are re-certified, at a minimum, every 3 years with equipment traceable to national standards maintained by NIST.

Working thermometers are compared with the reference thermometers annually according to Pace FSD procedures. Each thermometer is individually numbered and assigned a correction factor based on the reference source traceable to national standards maintained by NIST. In addition, working thermometers are visually inspected by personnel prior to use and temperatures are documented.

Thermometer inventory and calibration data are maintained in the Pace FSD Quality Office.

Additional information can be found in the most current revision of Pace FSD SOP *Calibration of Thermometers*, S-FSD-E-002, or its equivalent replacement.

6.3.3 pH/Electrometers

Meters are calibrated each day of use with fresh buffer solutions. Calibration information is recorded in associated logbook or on data sheets. For specific meter procedures, refer to the most current revision of the Pace FSD Procedure Manual, *Water Monitoring*, PM-FSD-W-004, or its equivalent replacement.

6.3.4 Barometers

Pace FSD uses a Princo Fortin type mercurial barometer (Princo 2) as a reference barometer. The Princo 2 was calibrated to a near zero correction by comparison with a Fortin type mercurial barometer traceable to national standards maintained by NIST by the manufacturer prior to purchase. Following the installation at Pace, the Princo 2 barometer was verified against a barometer traceable to national standards maintained by NIST and certified by an ISO/IEC 17025 accredited body to confirm its accuracy. The barometer should never go out of calibration if not damaged and used correctly.

Each field barometer is checked and (if necessary) calibrated on a semi-annual basis against the reference barometer by a qualified service technician. All information pertaining to barometer maintenance and calibration is recorded in the appropriate logbook and/or is maintained on file in the Pace FSD Quality Office.

Additional information can be found in the most current revision of the Pace FSD SOP *Calibration of Barometers*, S-FSD-E-004, or its equivalent replacement.

6.4 Instrument/ Equipment Maintenance

The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

The Pace FSD GM and department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, coordinate instrument repair and maintenance, and ensure associated computer programs are up-to-date. Analysts and technicians have a primary responsibility to perform routine maintenance.

To minimize downtime and interruption of work, planned maintenance is routinely performed on each analytical instrument. Pace FSD also provides for safe handling, transport, storage, and use of equipment to ensure proper functioning and in order to prevent contamination or deterioration. Up-to-date instructions on the use and maintenance of equipment are available to staff at the location where the equipment is used.

Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment requiring calibration is labeled, or otherwise identified, to indicate its status. This label or other identification includes, at a minimum, the date of the last calibration, the date when recalibration is due, and any applicable correction factors. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. Pace FSD maintains records of equipment and its software significant to testing or calibrations performed. These records include the following:

- The name of the equipment and its software
- The manufacturer name, type, and serial number

- Approximate date received and date placed into service
- Condition when received (new, used, etc.)
- Copy of any manufacturer manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

All instrument maintenance is documented in the appropriate (hardcopy or electronic) maintenance log.

When maintenance is performed to repair an instrument problem, the item will not be placed into service until it has demonstrated proper functioning. Verification that the instrument has been returned to an in-control status will be documented in the appropriate maintenance log. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance log, if different from the date(s) of the maintenance.

Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab, if possible.

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7.0 CONTROL OF DATA

Pace FSD retains records of original observations, derived data, and sufficient information to establish an audit trail, calibration records, and a copy of each test report or calibration certificate issued for a defined period. Results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user. All data undergo a review process prior to being reported to the customer. This section describes procedures used by Pace for translating raw data into accurate, final sample reports and Pace data storage policies.

7.1 Results Processing

All field measurements, observations, and testing data are recorded on appropriate field data sheets following the procedures in the most current revision of Pace FSD SOP *Documentation of Field Activities*, S-FSD-Q-004, or its equivalent replacement. Records for each test activity contain sufficient information to facilitate, if possible, identification of factors affecting uncertainty and to enable the test activity to be repeated under conditions as close as possible to the original. Project records include the identity of personnel responsible for the sampling and the performance of each test activity. All observations, data, and calculations are recorded at the time they are made and are identifiable to a specific task. Errors made while recording information are corrected by drawing a single line through the mistake, not erasing, deleting, or making illegible, and inserting the correct entry. The date and initials of the person making the correction are inserted near the error. Automated system printouts and other project documentation are labeled appropriately and are maintained in the respective project file. All records are kept in accordance with applicable storage and archival policies and procedures.

The primary analyst or technician is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, and noting non-conformances in logbooks and as footnotes or narratives. The primary analyst or technician must be clearly identified in all applicable logbooks, spreadsheets, or other data records.

Standardized spreadsheets are used to compile raw field data, analytical results, and pertinent process parameters to formulate final test results. Prior to implementation, spreadsheets are verified through secondary validation, formula cells are locked and password protected to mitigate unauthorized edits. Specialized and single use data reduction devices or calculations require secondary review before resultant data is released.

The primary analyst or technician then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain of custody forms, and logbook copies.

Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst or technician may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2 Data Verification

Data verification is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any discrepancies are properly documented.

Analysts and technicians performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst or technician initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. The completed data

package is then sent to a designated qualified reviewer (this cannot be the primary analyst or technician). Criteria have been established to qualify someone as a data reviewer. Both the primary analyst or technician and reviewer must be clearly identified on all applicable data review checklists. To perform a secondary data review, an individual must meet at least one of the following:

1. Have a current Demonstration of Capability (DOC) on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or[See Note]
2. Have a DOC on file for a similar method/technology and they have an SOP Acknowledgment form on file for the method/procedure being reviewed; or,[See Note]
3. Supervise or manage a Department and have an SOP Acknowledgment form on file for the method/procedure being reviewed; or,
4. Have significant background in the department/methods being reviewed through education or experience and have an SOP Acknowledgment form on file for the method/procedure being reviewed.

Note: Reviewer status must be approved personally by the Quality Assurance Officer or General Manager in the event that this person has no prior experience on the specific method or general technology.

This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods and protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation.

Once the data have been technically reviewed and approved, authorization for release of the data is indicated by initialing and dating the Project Completion Record or otherwise initialing and dating the data. The project administrator examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

7.3 Data Reporting

7.3.1 All data segments pertaining to a particular Pace FSD project are collected, reviewed, and compiled into a clear, accurate, unambiguous, and objective final report in accordance with the analytical and sampling procedures, methods, and needs of the project. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

7.3.2 Final reports are prepared according to the level of reporting required by the client and can be transmitted to the customer via hardcopy or electronic deliverable. Refer to the most current revision of Pace FSD SOP *Reporting*, S-FSD-Q-017, or its equivalent replacement, for details on reports and deliverables.

7.3.3 Any changes made to a final report after issue are re-issued with the designated of "Revised" or equivalent wording and meet any relevant regulatory requirements. Pace must keep sufficient archived records of all reports and revisions. For higher levels of data deliverables, a copy of all applicable raw data is sent to the client along with a final report of results. When necessary, opinions and interpretations can be included in reports if the statements are clearly marked as such and the bases for the opinions and interpretations are documented. Pace FSD can report results in a simplified way for internal clients or when agreed upon with external clients. When possible, the Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

7.3.4 Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

Additional information can be found in the most current revision of Pace FSD SOP *Reporting*, S-FSD-Q-017, or its equivalent replacement.

7.4 Data Security

All data including electronic files, logbooks, data sheets, calculations, project files and reports, and other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

7.5 Data Archiving

Pace FSD has established and maintains procedures for handling and storing quality and technical records. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. Access to archived data is documented and controlled by Pace FSD administrative staff and the off-site storage facility.

In the case of electronically stored documents and records, Pace FSD provides equivalent measures to avoid loss or change of original data. Records that are computer-generated have either a hard copy or electronic backup copy to prevent unauthorized access and changes. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

In the event of a change in ownership, accountability or liability, reports of work performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6 Data Disposal

Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports that are disposed.

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8.0 QUALITY SYSTEMS AUDITS AND IMPROVEMENTS

8.1 Internal Audits

8.1.1 Responsibilities

The Quality Assurance Officer is responsible for managing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since audits represent an impartial assessment of facility functions, internal audits are carried out by qualified personnel who, whenever practical, are functionally independent of the operation audited. The auditor must be trained, qualified and familiar enough with the objectives, principles, procedures and operation of the quality system to be able to perform a thorough and effective evaluation. The Quality Assurance Officer evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other applicable quality programs.

Additional information concerning audits can be found in the most current revision of Pace FSD SOP *Audits*, S-FSD-Q-009, or its equivalent replacement.

8.1.2 Scope and Frequency of Internal Audits

8.1.2.1 The complete internal audit process consists of the following four sections: 1) Raw Data Reviews, 2) traditional Quality Systems internal audits (including SOP and method compliance), 3) Final Report Reviews, and 4) Corrective Action Effectiveness Follow-up.

8.1.2.2 Internal systems audits are conducted yearly at a minimum. The scope of an audit may include the evaluation of an individual department or specific quality related system. Internal audits will assess field performance, final reports, and over-all system functioning. Deficiencies found during internal audits are linked to specific performance issues.

8.1.2.3 Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

8.1.2.4 Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

8.1.2.5 Pace FSD, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

8.1.3 Internal Audit Reports and Corrective Action Plans

A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the Pace FSD Quality Assurance Officer writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

When audit findings cast doubt on the effectiveness of the operations or on the validity and integrity of work performed, Pace FSD will take timely corrective action and notify the customer in writing within three business days, if investigations show that results may have been affected.

Additional information concerning audits can be found in the most current revision of Pace FSD SOP *Audits*, S-FSD-Q-009, or its equivalent replacement.

8.2 External Audits

Pace FSD may be audited regularly by regulatory agencies to maintain certifications and by customers to maintain appropriate specific protocols.

External audit teams to the company review Pace FSD to assess the effectiveness of quality systems. The Quality Assurance Officer and other staff host the audit team and assist in facilitation of the audit process. After the audit, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for Pace FSD. Pace staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the QAM, who provides a written response to the external audit team. The QAM follows-up with staff to ensure corrective actions are implemented and that the corrective action was effective.

8.3 Annual Managerial Review

A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

The managerial review must include the following topics of discussion:

- Policy and procedure suitability
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventative actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Client feedback, including complaints
- Recommendations for improvement
- Other relevant factors, such as quality control activities, resources, and staffing

This managerial review must be documented for future reference by the Quality Assurance Officer and copies of the report are distributed to staff. Results should feed into the FSD planning system and should include goals, objectives, and action plans for the coming year. Pace FSD shall ensure that any actions identified during the review are carried out within an appropriate and agreed timescale.

Additional information can be found in the most current revision of Pace FSD SOP *Management Review and Quarterly Quality Reports*, S-FSD-Q-010, or its equivalent replacement.

8.4 Client Feedback

8.4.1 Pace FSD actively seeks feedback, both positive and negative, from clients as part of its continuous improvement program. Feedback solicited will include the entire range of a client's interaction with Pace FSD. Methods of solicitation may vary and include:

- Electronic survey, online or email
- Mailing survey
- Verbal
- Written statement with report

8.4.2 Pace personnel will relay verbal feedback to the Quality Assurance Officer. The QAO reviews and conveys any feedback received, solicited or unsolicited, to the appropriate party, as needed. Any feedback determined to be a complaint by the receiving party, or QAO, is addressed according to procedures documented in the most current revision of Pace FSD SOP *Inquiry and Complaint Resolution*, S-FSD-Q-007, or its equivalent replacement. Solicitation records are maintained in the Pace FSD Quality Office. All feedback is discussed during the annual management review and is evaluate and used to improve the management system, testing activities and customer service.

8.4.3 In addition, Pace FSD cooperates with customers or their representatives to clarify customer requests and to monitor performance in relation to the work being performed for the customers. This cooperation may include providing the customer reasonable access to relevant areas to observe activities performed.

8.4.4 Customer service is an important aspect to Pace's overall objective of providing a quality product. Good communication should be provided to the customer's throughout projects. Pace FSD should inform the customer of any delay or major deviations in the performance of analytical tests.

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9.0 CORRECTIVE ACTION

Additional information can be found in the most current revision of Pace FSD SOP *Corrective and Preventative Action*, S-FSD-Q-008, or its equivalent replacement.

During the process of sample collection, handling, preparation and analysis, or during review of quality control records, or during reviews of non-technical areas, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of client complaints, personnel errors, deficiencies in quality control, method or procedure deviations, or other unusual circumstances. The Quality System of Pace FSD provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using a system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved

9.1 Client Complaints

Inquiries, both written and verbal, regarding submitted data, field activities, and technician actions, will be assessed by the individual receiving the inquiry and determined to be a minor inquiry or a complaint. An inquiry is deemed minor, if it does not question the validity and integrity of work performed and is resolved with a simple answer over the phone or through email without changes to submitted data, excluding negligible grammatical corrections. An inquiry is deemed a complaint, if the inquiry questions the validity and integrity of work performed or changes are required to submitted data. Complaints require the completion of a Complaint Report Form by the individual receiving the inquiry. All complaints are investigated, documented, and addressed with an appropriate response, which may include corrective action.

9.2 Deviation and Nonconformance

Pace FSD implements policies and procedures when any aspect of its testing or calibration activities does not conform to its own procedures or agreed requirements of the applicable client. Deviation from test plans or other project specific documents occur only if the deviation is documented, technically justified, authorized, and accepted by the client or any relevant regulatory authority. Do to the inherent variability in field activities, Pace FSD relies heavily on the expertise and professional judgment of its personnel when evaluating and addressing deviation and nonconformance events occurring during field activities. When a deviation has been recognized, an evaluation of the significance of the nonconforming work is made. Impact on the method/procedure, resulting data, and client requirements is determine and documented to address the deviation. If necessary, the client is notified and work is halted or re-done. Pace FSD grants authority to management to halt work and withhold testing reports when nonconforming work is identified. When an evaluation of a nonconformance indicates that the nonconforming work could recur or that there is doubt about the compliance of Pace FSD operating within its own policies and procedures, the corrective action procedure is initiated.

The following items are examples of deviations or nonconformance that warrant some form of documentation:

- Quality Control data outside of acceptance criteria
- Sample collection deviations
- Sample handling deviations
- Instrument failures (including calibration failure)
- Sample preparation or analysis errors
- Sample contamination
- Errors in client reports
- Audit findings (internal and external)
- Proficiency Testing (PT) sample failures

- Client complaints or inquiries

Minor deviations will be documented in field notes or as a comment or footnote in the final report. Nonconformance will be documented on a Nonconformance and Corrective Action Form. This documentation must include any affected samples, observations, or results, and the name of the project administrator and the client name. Any known causes of the nonconformance will also be recorded.

All nonconformances are evaluated by the Pace FSD Quality Assurance Officer for the need for corrective action. This evaluation may include reviewing data, reports, and interviewing involved personnel. Corrective action will be initiated when necessary. Where the identification of deviations or nonconformance casts doubts on Pace FSD's compliance with its own policies and procedures, or on its compliance with applicable regulations, Pace FSD will ensure that the appropriate areas of activity are audited as soon as possible.

Additional information can be found in the most current revision of Pace FSD SOP *Documentation of Deviation and Nonconformance*, S-FSD-Q-006, or its equivalent replacement.

9.3 Corrective Action

Corrective Action (CA) is essential in the elimination of the source of an occurrence that indicates weakness or inconsistency within the quality or technical systems of Pace FSD in order to prevent recurrence. Pace FSD implements policies and procedures and designates appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the quality system or technical operations have been identified. Where needed, Pace identifies potential corrective actions and selects and implements the action(s) most likely to eliminate the problem and prevent recurrence. CA may result from client complaints, personnel errors, method or procedure deviations, audit findings, proficiency testing failures, management, or other undesirable situations. All corrective action and decisions about the acceptability of nonconforming work are taken within an appropriate time period and to a degree appropriate to the magnitude and risk of the identified problem.

The Quality Assurance Officer will review and assess all nonconformances, complaints, responses to audit findings or other undesirable situations to determine the need for corrective action. If a process or procedure is determined to be deficient or detrimental to the validity and integrity of the work performed by Pace FSD, the QAO will document it on a Nonconformance and Corrective Action Form. The corrective action process will follow these general steps: define the nonconformance problem, assign personnel to investigate, determine the root cause of the problem, develop a solution to the problem, and implement and verify the solution. Pace FSD documents and implements any required changes resulting from corrective action investigations. Results of the corrective action are monitored to ensure the changes have been effective.

9.4 Preventative Action

Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems including technical, managerial, and quality. These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems are discovered. These improvements can be made within a department or lab-wide.
- Annual managerial reviews - part of this accreditation-required review is to look at all processes and procedures used by the lab over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews - any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

When improvement opportunities are identified or if preventive action is required, Pace can develop, implement, and monitor preventive action plans.

10.0 GLOSSARY

3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	Specified limits place on characteristics of an item, process, or service defined in requirement documents.
Accreditation	The process by which an agency or organization evaluates and recognizes an entity as meeting certain predetermined qualifications or standards, thereby accrediting the entity.
Accuracy	The agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
ASTM: American Society for Testing and Materials	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analyte	The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
Audit	A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours (Eight hours for South Carolina). An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Calibration	A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Range	The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	A substance or reference material used for calibration.
Chain-of-Custody (COC)	Record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, collector, time of collection, preservation, and requested analyses.
Calibration	The process of verifying a calibration by analysis of standards and comparing the results with

Verification	the known amount.
Continuing Calibration Verification (CCV)	The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit).
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of nonconforming data.
Corrective Action	The action taken to eliminate the causes of a nonconformity, defect, or other undesirable situation in order to prevent recurrence.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Quality Objective (DOQ)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
Document Control	Procedures to ensure that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • **Non Potable Water:** (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts). • **Drinking Water**: Delivered (treated or untreated) water designated as potable water. • **Water/Wastewater**: Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, & industrial influents/effluents. • **Sludge**: Municipal sludges and industrial sludges. • **Soil**: Predominately inorganic matter ranging in classification from sands to clays. • **Waste**: Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes.

Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Finding	An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.
Holding Time	The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the re-quirements of the method in use or at a frequency specified in the method.
Initial Calibration Verification (ICV)	A standard obtained or prepared from a source independent of the source of the standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is performed after the initial calibration.
Internal Standards	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
Laboratory Control Sample (LCS)	(however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
Limit of Detection (LOD)	A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility.
Limit of Quantitation (LOQ)	The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix	The substrate of a test sample.
Matrix Spike (MS) (spiked sample or fortified sample)	A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike	A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of

Duplicate (MSD)	precision of the recovery of each analyte (sometimes referred to as Spiked Sample Duplicate or Fortified Sample Duplicate).
Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis) systematically presented in the order in which they are to be executed.
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Limit of Detection (LOD); defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
National Institute of Standards and Technology (NIST)	A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.
Preservation	Any conditions under which a sample must be kept in order to maintain the chemical and/or biological integrity of the sample.
Procedure	A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Sample	A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing Program	The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures required by a specific project.
Quality Assurance (QA)	An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Control (QC)	The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance	A formal document describing the detailed quality control procedures by which the quality

Project Plan (QAPP)	requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin • **Chemical Waste**: A product or by-product or an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Random Error	The EPA has established there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, printouts of chromatograms, instrument outputs, and handwritten records.
Reference Standard	Standard used for the calibration of working measurement standards in a given organization or at a given location.
Reporting Limit (RL)	The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL.
Sampling	Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels (concentrations) of a variable of interest.
Standard	A substance or material with properties known with sufficient accuracy to permit its use to evaluate the same property in a sample.
Standard Blank	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Operating Procedure (SOP)	A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.

Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Traceability	The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measured (i.e., the concentration of an analyte).
Unregulated Contaminate Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminates in drinking water.
Validation	The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.
Verification	Confirmation by examination and objective evidence that specified requirements have been met.

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11.0 REFERENCES

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.
- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
- Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988.
- National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent.
- TNI (The NELAC Institute) Standards; most recent version.
- UCMR3 Laboratory Approval Requirements and Information Document, version 2.0, January 2012.

12.0 REVISIONS

The Pace FSD Quality Assurance Office file an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Revision 4.0	Intermediate section numbers added to Section 1.3, 1.5, 4.13, 8.5 to assist in reference. Section 1.3.4: Deleted examples of regulatory bodies; substituted with "regulatory authorities" per corporate template. Section 1.5.4: Added anonymous hotline number per corporate template. Section 1.8.3: Added #2 and edited #4 to Quality Manager responsibilities per corporate template. Section 1.8.14: Addition of Sales Associate job description; missing from previous version. Section 1.10: Added anonymous hotline number to last paragraph per corporate template; removed reference to number in employee handbook. Section 2.4: Edited to more closely match COC section in FSD procedure manuals. Section 2.5: Last bullet point modified to reflect Minnesota Laboratory Sample Acceptance policy regarding samples in cooling phase. Section 4.13: Added sentence regarding rounding occurring after comparing results and RL and MDL limits per corporate template. Section 5.1.2: Edited sentence regarding review procedure when no revisions needed per corporate template. Section 6.3.1: Certification period for NIST traceable weights extended from one year to five years per corporate template. Section 6.4: Changed 'logbook' to 'log' and added (hardcopy or electronic). Section 7.1: Added statement regarding identifying primary on results documents per corporate template; added section regarding spreadsheet validation and use in results processing to match FSD procedure. Section 7.2: Added statement regarding identifying primary and reviewer on data review documents per corporate template. Section 7.3, #11: Added statement regarding identification on report of samples not meeting test plan or regulatory requirements per corporate template. Section 8.1.2: Added two bullet points: "data records and non-technical documents" and "sample receipt and management practices" per corporate template. Section 8.3: Added SOP reference to current FSD document. Section 8.3: Deleted items to match current MR/QRR SOP. Section 8.5.3: Added last sentence per corporate template. Section 8.5.4: Added section per corporate template. Section 10.0: Added definitions for Acceptance Criteria, Accreditation, Calibration Range, Calibration Standard, CCV, CEM, ICV, Matrix, NIST, Procedure, Proficiency Testing Program, QAPP, Technical Director, and Validation; edited definitions for Analyte, Calibration, Document Control, Holding Time, LOD, MSD, and Quality System per Corporate Template. Section 11: Added TNI to reference list. Attachment IIA and IIB: Updated with most current structures. Attachment III: Updated equipment table. Attachment IV: Updated floor plan. Attachment V: Updated FSD SOP list and edited titles within document as necessary. Attachment VII: Updated bottle type, holding time, preservation requirements per revised corporate bottle-prep SOP; added statement at top of table.	March 21, 2013

Document Number	Reason for Change	Date
Revision 5.0	Reference to Corporate Template is Rev.17 issued April 2014. Cover page: Updated addresses. Section 1.1: Removed "industrial hygiene testing" and "explosives" from testing list per corporate template. Section 1.3.1: Added "that is fully compliant with applicable regulatory standards" per corporate template. Section 1.8.1: Added number 6. Section 1.8.4: Added number 6 regarding serving as manager in times of absence per corporate template. Section 1.8.11: Matched header and points to those in corporate template. Section 1.8.15 Added Hazardous Waste job description. Section 1.10: Added "Data" to header per corporate template. Section 1.13: Added Communication section per corporate template. Section 2.5: Updated bullet points to match corporate template. Section 2.6: Added new Sample Log-in section per corporate template; all subsequent sections renumbered. Section 2.7.2: Added "and above freezing" per corporate template. Section 2.7.4: Corrected SOP number to reflect MN document. Section 2.8 and 2.9: Updated for clarity. Section 2.10: Added 45 day minimum retention time per corporate template. Section 5.3: Corrected Management of Change SOP number. Section 6.3.1: Updated calibration weight class per corporate template; changed mobile balance calibration period to annual per current procedure. Section 6.4: Added last sentence to last paragraph "In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab, if possible." Section 7.2, #1: Added SOP acknowledgement to reviewer requirement. Section 7.3: Updated list to match corporate template. Section 7.6: Added last sentence regarding disposal per corporate template Section 8.4: Added "Management and" per corporate template. Section 9.0: Updated to include new nonconformance and corrective action combo form; added last sentence per corporate template. Section 10.0: Added definition for 'Unregulated Contaminate Monitoring Rule (UCMR).' Attachment IIA & B: Updated with current versions. Attachment IV: Updated floor plan. Attachment V: Deleted IT SOPs; retired by corporate: info now disseminated through training guides; Added Contingency and Emergency Procedure Plan document. Attachment VIII: Updated preservation table per corporate template.	April 21, 2014
Revision 6.0	Cover Page: Added "Effective date of last signature" Section 1.7: Added section numbers to each paragraph. Section 1.7.7: Added statement regarding deputies assigned for key positions. Section 1.12: Replaced 'Sample Custodian' to 'department supervisors and field technicians/analysts'. Section 2.6: Deleted LIMS codes; redundant information from SOP. Section 6.3.1: Edited to remove brass reference weight use for mobile balances. Section 6.3.4: Edited calibration interval to reflect change from quarterly to semi-annual. Section 8.3: Added 'or designee'. Section 12.0: Removed wording regarding filing paper copy. Attachment IIA: Updated FSD Organizational Chart. Attachment IIB: Updated Corporate Organizational Chart Attachment III: Updated Equipment List Attachment V: Updated SOP List. Attachment VII: Updated Preservation Table to match corporate table.	June 11, 2015

Document Number	Reason for Change	Date
Revision 7.0	Section 1.8.16: Added Equipment Services Specialist job title and major responsibilities. Section 2.9: Added clause regarding COC. Section 3.3: Reworded sentence regarding minimum requirements for method validation. Section 3.6: Updated to current accreditation status. Section 8.2: "report" replaced with "response" through section. Attachment IIA: Updated FSD Organizational Chart. Attachment IIB: Updated Corporate Organizational Chart Attachment III: Changed to Map; Updated outline. Attachment IV: Changed to COC. Attachment V: Changed to Equipment List; Updated to current status. Attachment VI: Changed to SOP list; Updated to current status. Attachment VII: Updated.	June 23, 2016
Revision 8.0	All document: Updated Inc to LLC; replaced PASI with Pace. All document: Replaced NIST-traceable to "traceable to national standards maintained by NIST." All document: Grammatical and formatting changes that do not affect policy and procedures. Cover Page: removed corporate approval signature lines per Corporate template. Section 1.1, 1.3: Edited for clarity and to reduce redundancy. Section 1.4: Removed definitions from Pace Core Values list. Section 1.5.2: Rearranged Standard of Conduct; moved Data Integrity (previous Section 1.10) and Confidentiality sections to subsections of Standard of Conducts; match language to Corporate Template 19.0. Section 1.5.5: Replaced Compliance section to match language of Corporate Template 19.0; subsequent sections renumbered. Section 1.6 Added section addressing Anonymous Compliance Alertline. Section 1.7.1, 1.7.2, 1.7.3, 1.7.9: Edited for clarity, to reduce redundancy, and to match language to Corporate Template 19.0. Section 1.9: Edited for clarity, to reduce redundancy, and to match language to Corporate Template 19.0.; combined training lists. Section 1.10: Added "and Waste" to Section title; replaced "health" with "waste compliance." Section 1.12: Edited for clarity, to reduce redundancy, and to match language to Corporate Template 19.0. Section 2.1: Project Initiation moved from Section 2.3; Edited for clarity, to reduce redundancy, and to match language to Corporate Template 19.0. Section 2.4: Edited for clarity, to reduce redundancy, and to match language to Corporate Template 19.0; added section numbering. Section 2.5: Edited bullet points for clarity, to reduce redundancy, and to match language to Corporate Template 19.0; added section numbering; added bullet list regarding items checked upon sample receipt. Section 2.6: Edited bullet points for clarity, to reduce redundancy, and to match language to Corporate Template 19.0; generic time changed to 12:01 AM. Section 2.7.2: Edited bullet points for clarity, to reduce redundancy with SOP, and to match language to Corporate Template 19.0. Section 2.7.3: Match language to Corporate Template 19.0. Section 2.8: Added clause regarding pour back of unused reagent, standard, sample. Section 2.10: Added section numbers; edited to match Corporate Template 19.0. Section 4.1: Added section regarding Quality Control Samples; subsequent sections renumbered. Section 4.12: Edited bullet points for clarity, to reduce redundancy with SOP, and to match language to Corporate Template 19.0. Section 5.0: Added section numberings. Section 5.1.1, 5.1.2, 5.1.3: Edited bullet points for clarity, to reduce	September 18, 2017

Document Number	Reason for Change	Date
Revision 8.0	redundancy with SOPs, and to match language to Corporate Template 19.0. Section 5.3: Deleted Management of Change section per corporate revision 19.0. Section 7.3: Edited bullet points for clarity, to reduce redundancy with SOP, and to match language to Corporate Template 19.0. Section 7.5: Edited to match Corporate Template 19.0; removed examples of documents included; changed length of reports are kept in event of change in ownership. Section 8.1, 8.2: Edited bullet points for clarity, to reduce redundancy with SOP, and to match language to Corporate Template 19.0. Section 8.3: Deleted Quarterly Quality Report section; subsequent sections renumbered. Section 10: Updated Glossary Terms. Attachment II: Updated FSD Organization Chart Attachment III: Relabeled Corporate Organizational Chart to Attachment 3; updated Corporate Organizational Chart. Attachment IV: removed floor plan attachment per corporate Revision 19.0; renumbered subsequent attachments; Update Equipment List. Attachment VI: removed COC to eliminate redundancy with other documents per corporate Revision 19.0. Attachment V: Updated Quality Document Log.	September 18, 2017
Revision 8.0	Periodic Review Signature Only; Updated FSD Org Chart. All other changes held for incorporation in MasterControl 10/2018.	September 19, 2018

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ATTACHMENT I

Quality Control Calculations

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

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ATTACHMENT I (CONTINUED)

Quality Control Calculations (continued)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

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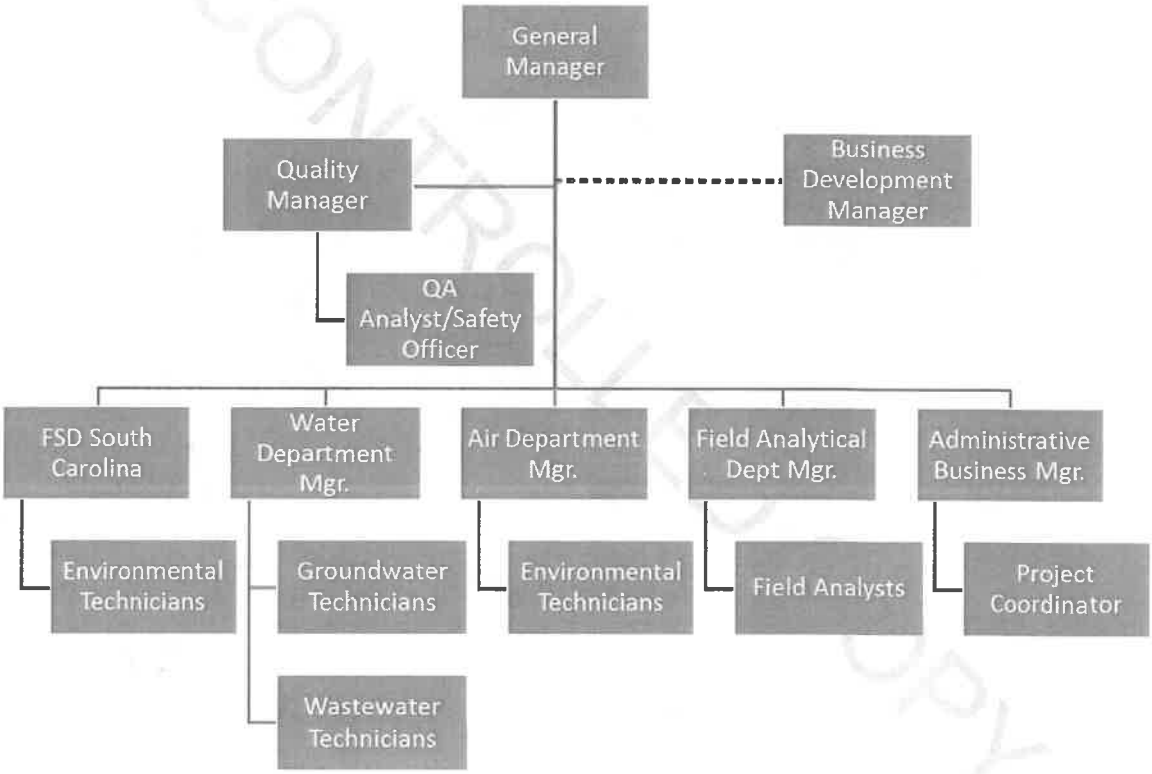
ATTACHMENT II

Field Services Division Organizational Chart
(Current as of issue date.)



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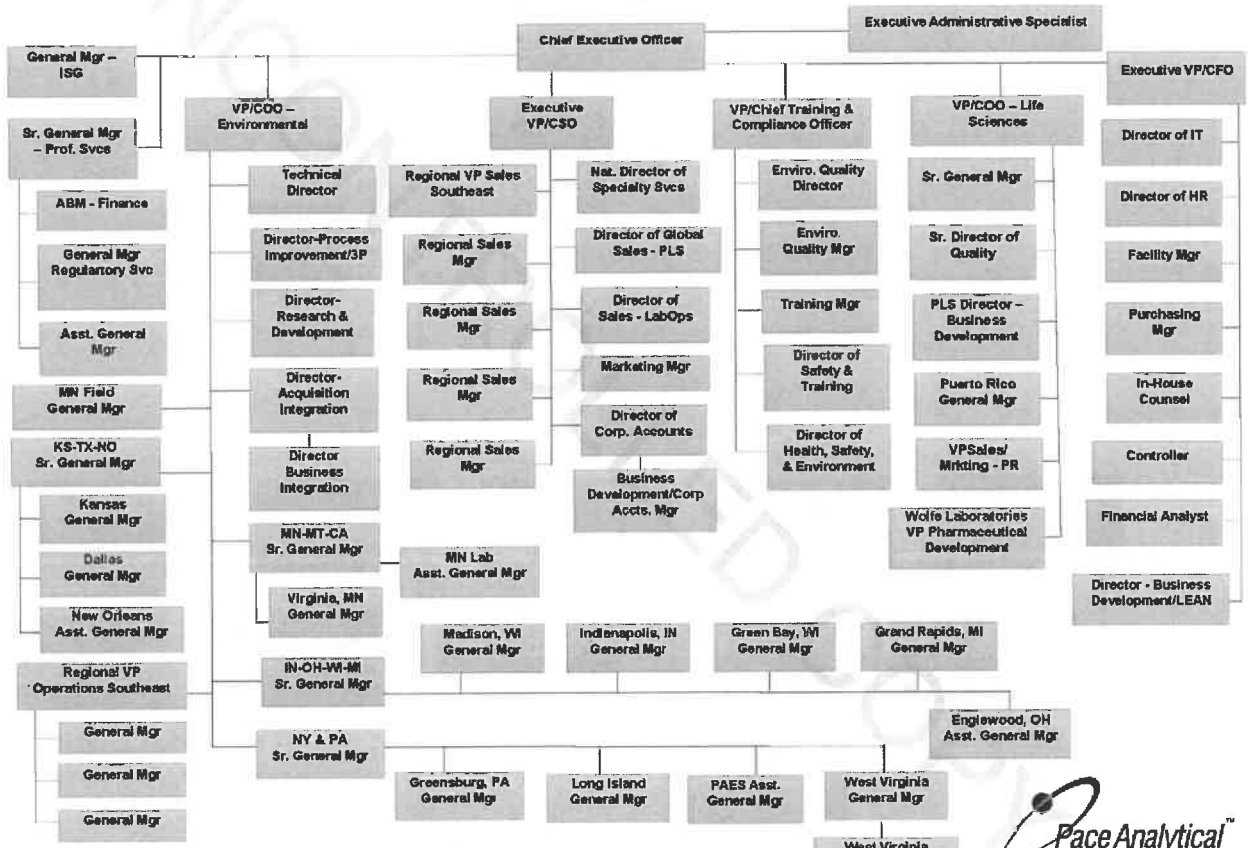
ATTACHMENT III
PACE – CORPORATE ORGANIZATIONAL CHART



Corporate/Management Staff
August 2017

ATTACHMENT IV – Major Equipment

(Current as of issue date.)

PACE – FIELD SERVICES DIVISION

Instrument	Manufacturer	Model	Quantity
Automatic Sampler	Isco	3700	23
Automatic Sampler	Isco	6700	5
Automatic Sampler	Isco	2900	3
Automatic Sampler	Isco	2700	3
Automatic Sampler	Isco	GLS	1
Flow Meter	Isco	4230	8
Stream Gauging Flow Meter	Global Water	FP-201	1
Flow Meter	Meter-Master	Model 100	4
Multi Parameter Sonde	YSI	600XL	3
650 MDS Display	YSI	650 MDS	1
Surveyor 4a	Hach Hydrolab	4a	1
pH Meter	Orion	230A	2
pH Meter	Orion	410A+	1
Logging pH Meter	Sper	850059	1
Logging pH Meter	Sper	80060	2
Turbidity Meter	WTW	350 IR	1
Turbidity Meter	HF Scientific	DTR 15CE	1
Turbidity Meter	HF Scientific	MicroTPI	1
Turbidity Meter	HF Scientific	20008	1
Turbidity Meter	Hach	2100P	1
Portable Colorimeter	Hach	Colorimeter II	2
Portable Colorimeter	Hach	DR/890	1
Portable Colorimeter	Hach	DR/3000	1
Ferrous Iron Kit	Hach	IR-18C	1
DO Meter	YSI	54A	1
DO Meter	YSI	51B	1
Multi Probe System	YSI	556 MPS	1
Multi Probe System	YSI	ProPlus	3
VFD Controller	Grundfos	Rediflo VFD	2
Low Flow w/Power Booster XL	Proactive	for SS Mega Monsoon XL	1
12 VDC Submersible Pump Controller	Pace	Generic	1
Submersible Pump	Grundfos	Rediflo 2	3
Submersible Pump	Proactive	SS Mega Monsoon XL	1
Submersible Bladder Pump	GeoPump	57000	2
12 VDC Submersible Pump	Whale	921 (Dual)	1
12 VDC Submersible Pump	Whale	921 (Single)	1

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Geopump Peristaltic Pump	Geotech	Easy Load 2	1
Masterflex Pump	Masterflex	7520-00	1
Masterflex Pump	Masterflex	n/a	1
Isco Portable Peristaltic Pump	Isco	PTP-150	1
Geocontrol Controller	Geotech	Pro	3
Geoguard Pneumatic Controller	Geoguard	5100	1
Water Level Meter	Keck	300	2
Water Level Meter	Keck	200	1
Water Level Meter	Solinst	101-150-P4	1
Water Level Meter	Heron Instruments	Dipper-T	1
Water Level Meter	Geotech	ET	1
Multi-Gas Meter	RKI Instruments	Eagle	2
Multi-Gas Meter	RKI Instruments	GX-2003	1
Oven	Fisher	Iso Temp	1
Oven	Fisher	Iso Temp 500	1
Oven	Lindberg/Blue (Thermo)	MO1450PSA-1	1
Hot Wire Anemometer	TSI	VELOCICALC 8350	1
Hot Wire Anemometer	Control Company	4330	1
Digital Vane Anemometer	Fisher/Control Company	Vane	1
Digital Vane Anemometer	Extech	Thermo-Anemometer	1
Total Hydrocarbon Analyzer	VIG Indutries	20-2	3
Total Hydrocarbon Analyzer	JUM	3-500	4
PID	RAE SYSTEMS	Mini Rae 2000	1
CO	Teledyne	TML30M	2
CO	Teledyne	TML30	1
CO	Teledyne	T300M	3
CO2/O2	Servomex	1440D	3
CO2/O2	Servomex	1440D1STD	1
CO2/O2	Servomex	SERVOPRO 1440	3
NOx	California Analytical	600-CLD	5
NOx/O2	Teledyne	TML-41-H-02	1
SO2	Teledyne API	100AH	1
SO2	Teledyne API	100EH	1
SO2	Teledyne	TML-50-H	1
SO2	Teledyne	T100H	3
O2/CO/NO/NO2/SO2	ECOM	J2KNPro	1
Toxic Vapor Analyzer	Thermo	TVA-1000B	1
Zero Air Generator	Teledyne	701	1
Calibrator	Teledyne	700E	1
Chiller	Universal Analyzer	1060MPV	1
Chiller	Universal Analyzer	1060PV	4

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Chiller	Universal Analyzer	1080	1
Chiller	Universal Analyzer	1050	1
Chiller	Universal Analyzer	3080	3
P-DAQ	Omega	DAQ-55	4
P-DAQ	Omega	DAQ-54	2
P-DAQ	Strawberry Tree	DATA Shuttle	1
O2	Teledyne	320 P-D	4
O2	Testo	327-1	1
O2	Nuvair	Pro O2	8
Orsat Analyzer	Burrell	INDUSTRO-B Gas Analyzer	2
Personal Pump	SKC	224-PCXR3	1
Personal Pump	SKC	224-PCXR7	4
Personal Pump	Gilian	HF3513A	3
DryCal Flow Meter	Bios	DC-Lite	7
DryCal Flow Meter	Bios	Defender 510L	1
Balance	Mettler	AE163	1
Balance	Mettler	AE200	1
Balance	Sartorius	LP620S	1
Balance	Ohaus	1500D	1
Balance	Sartorius	L2200P	1
Balance	Sartorius	CPA124S	1
Balance	Smart Weigh	Pro Pocket	16
Temperature Display Calibrator	Omega	CL23A	1
Temperature Display Calibrator	Altek	Series 22	1
Temperature Displays	Omega	HH-12	2
Temperature Displays	Omega	871A	5
Temperature Displays	Omega	HH-12A	1
Temperature Displays	Omega	HH-21	1
Temperature Displays	Omega	HH-12B	16
Temperature Displays	HOBO	UX120-014M	1
Temperature Displays	Fluke	51II	2
EDM	Air Flow	APM K5	2
EDM	Love	HM 28	1
EDM	Solomat Air Neo.	PDM205	8
EDM	Air Flow	PVM100	1
EDM	Omega	HHP102M	2
EDM	Fluke	992	5
EDM	Neotronics	PDM305	4
EDM	Dwyer	475 Mark III	2
Mercury Barometer	Princo	496	1
Digital Barometer	WeatherHawk	SM-18	12

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Digital Barometer	Motorola	Android	2
Digital Barometer	Apple	iPhone 6s	10
Mechanical Barometer	Thommen	TX-16	2
Pitot Tube - Standard	Dwyer	Misc.	30
Pitot Tube - S-Type	Dwyer	Misc.	30
Nozzle - Stainless Steel	Dwyer	Misc.	40
Nozzle - Glass	Dwyer	Misc.	30
Impinger	Dwyer	Misc.	30
Canister	Summa	1 Liter	50
Isokinetic Train	Pace	Pace	10
Isokinetic Train	Grasbey NuTech	2010	1
Isokinetic Train	Grasbey NuTech	2010A	1
Isokinetic Train	Environmental Supply	C-5000	2
Heated Line	n/a	100 FT	5
Heated Line	n/a	75 FT	2
Heated Line	n/a	50 FT	6
Heated Line	n/a	25 FT	4
Heated Line Controller	Omega	CM9122A	6
Heated Line Controller	Omega	CN9000A	2
Heated Line Controller	Omega	CM9122A	2
Heated Line Controller	Watlow	EZ Zone	1
Heated Line Controller	Fuji Electric	PXV3	4
Heated Line Controller	Omega	CN9500	2
Heated Line Controller	CAE	n/a	2
Heated Line Controller	Watlow	SD31	1
Heated Line Controller	Neptech, Inc.	n/a	4
Umbilical	Pace	Custom	13
Gas Dilution System	Millennium Inst.	200-A203	2
Wet Test Meter	Shinagawa	W-NK-5B	1
FTIR	MIDAC	I2001	4
FTIR	MIDAC	i2000	2
FTIR	MKS	2030	4
GC/FTIR	MAX	MAX	1
TDT Sampler	MAX	TDT Sampler	2
Micro GC	Agilent	3000 A	1
Temp Controller	Watlow	Series 96	6
Temp Controller	Watlow	EZ Zone	16
Pressure Transducer	SenSym	n/a	5
Pressure Transducer	Bourns	n/a	2
Pressure Transducer	MKS	n/a	6
Sample Modules	Pace	n/a	9

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High Volume Air Sampler	Tisch Environmental	5170-DL-BL	1
Van	Ford	E-250	2
Van	Chevrolet	Express	2
Van	Ford	Transit Connect	1
Pickup Truck	Ford	F-350 w/Utility Box	4
Pickup Truck	Ford	F350 Super Duty	1
Pickup Truck	Ford	F250 Crew Cab	3
Pickup Truck	Ford	F350 Crew Cab	2
SUV	Ford	Explorer	1
Trailer	Wells	Trailer	5
Trailer	Haulmark	MC612BS	1
Portable Generator	Commercial Mobile Power	CMM 7000/7HGJAE-2132C	1
Portable Generator	Honda	EU6500is	1
Portable Generator	Honda	EM4000SX	1

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ATTACHMENT V

STANDARD OPERATING PROCEDURES AND PROCEDURE MANUALS

PACE - FIELD SERVICES DIVISION

Document Number	Document Title
S-FSD-Q-001	Preparation of SOP and PM
S-FSD-Q-002	Document Control and Management
S-FSD-Q-003	Document Numbering
S-FSD-Q-004	Documentation of Field Activities
S-FSD-Q-005	Orientation and Training Procedures
S-FSD-Q-006	Documentation of Deviations and Nonconformance
S-FSD-Q-007	Inquiry and Complaint Resolution
S-FSD-Q-008	Corrective and Preventative Action
S-FSD-Q-009	Audits
S-FSD-Q-010	Management Review and Quarterly Quality Reports
S-FSD-Q-011	Subcontracting of Work
S-FSD-Q-012	Purchasing of Supplies and Services
S-FSD-Q-013	Receipt and Storage of Equipment and Supplies
S-FSD-Q-014	Significant Figures and Rounding Rules
S-FSD-Q-015	Reference to and Use of A2LA Accreditation Status and Symbol
S-FSD-Q-016	Equipment Handling, Maintenance, and Calibration
S-FSD-Q-017	Reporting
S-FSD-Q-018	Standards and Reagent Management
S-FSD-Q-019	Software and Spreadsheet Validation
S-FSD-Q-020	Use of STAC Terms and Logo
S-FSD-Q-021	Proficiency Testing
S-FSD-Q-022	Requests, Tenders, Contracts and Project Initiation
S-FSD-Q-023	Estimation of Measurement Uncertainty
S-FSD-Q-024	Evaluation and Qualification of Vendors
S-FSD-Q-024	Review of Data Deliverables
S-FSD-E-001	Monitoring of Coolers and Ovens
S-FSD-E-002	Calibration of Thermometers
S-FSD-E-003	Performance Verification and Calibration of Analytical Balances
S-FSD-E-004	Calibration of Barometers
S-FSD-E-005	Performance Verification and Calibration of Temperature Displays and Temperature Controllers
S-FSD-E-006	Calibration of Pitot Tubes
S-FSD-E-007	Performance Verification and Calibration of Electronic Digital Manometers (EDMs)
S-FSD-E-008	Calibration of Midget Modules

Document Number	Document Title
S-FSD-E-009	Calibration of Control Modules
S-FSD-E-010	Maintenance and Performance Verification of DryCal DC-Lite Air Flow Meter
S-FSD-E-011	Functional Testing of Thermocouples
S-FSD-E-012	Performance Verification of FTIR Pressure Transducers
S-FSD-E-013	Calibration of RKI Multi-Gas Detector
S-FSD-E-014	Performance Verification of Angle Gauges and Calipers
S-FSD-E-015	Millennium Diluter Performance Verification
S-FSD-E-016	Drager 5500 Oxygen Monitor
S-FSD-A-001	Qualified Individual Program
S-FSD-W-001	Chlorine MDL Study
S-ALL-S-001	Hazard Assessments
S-FSD-S-002	DOT Training
S-FSD-S-003	Waste Management Training Requirements
S-FSD-S-004	Air Quality Monitoring and Fume Hood Monitoring
S-FSD-S-005	Fall Protection Equipment Inspection
S-MN-S-003	Waste Handling and Management
PM-FSD-001	Administrative Manual
PM-FSD-002	Air Manual
PM-FSD-003	Field Analytical Manual
PM-FSD-004	Water Manual
Revision	Field Services Division Quality Manual
YEAR	Chemical Hygiene Plan/Safety Manual
MN Field	Contingency and Emergency Procedure Plan
Milan Field	Contingency and Emergency Procedure Plan
ASTM D7036	Standard Practice for Competence of Air Emission Testing Bodies
ISO/IEC 17025:2005(E)	General requirements for the competence of testing and calibration laboratories
TNI FSMO-V1-ISO-2014	General Requirements for Field Sampling and Measurement Organizations
I102	Responding to Deficiency Report
I105	Typical Steps in Preparing for the Accreditation Process
P113	A2LA Policy on Measurement Traceability for Life Sciences Testing Laboratories
R101	General Requirements: Accreditation of ISO/IEC 17025 Laboratories
R102	Conditions for Accreditation
R103	General Requirements: Proficiency Testing for ISO/IEC 17025 Laboratories
R103A	Annex to the A2LA General Requirements for Proficiency Testing
R105	Requirements When Making Reference to A2LA Accredited Status
R219	Specific Requirements: TNI Field Sampling and Measurement Organization (FSMO) Accreditation Program

ATTACHMENT VI

METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

PACE – FIELD SERVICES DIVISION

This table should be used as a guide; please confirm local regulations and verify specific laboratory requirements.

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	≤ 6°C	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6°C	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤ 6°C	14 Days
Alkylated PAHs		Water	1L Amber Glass	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	≤ 10°C	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	≤ 6°C; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	≤ 6°C	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	≤ 6°C	28 days
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	≤ 6°C	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	48 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; ≤ 6°C	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; ≤ 6°C	7/40 Days
Carbamates	8318	Solid	Glass	≤ 6°C	7/40 Days
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6°C, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	≤ 6°C; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C, $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	24 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)
Diesel Range Organics-Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics-Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics-TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics-	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
TPH DRO				present	
Diesel Range Organics-TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics-TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Diesel Range Organics-NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics-NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics-Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics-Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	30/45 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10°C	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6°C; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	≤ 6°C; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	≤ 6°C	14 Days
Gasoline Range Organics-Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics-	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Alaska GRO					
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Glyphosate	547	Water	Glass	≤ 6°C; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	≤ 6°C	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH ≥8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	30 Days from collection to extraction and 7 days from r extraction to analysis
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			being tested)		preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	40mL vials	HCl; or trisodium phosphate or benzalkonium chloride and ≤ 6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H2SO4 (must be preserved within 48 hours of collection)	6 months
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6°C	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6°C	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6°C	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides,	608	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl	Pest: 7/40 Days;

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Organochlorine (OC)				present	PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3: pH<2 HCl; ≤ 6°C	14/30 Days
Perchlorate	331	Water	Plastic/Glass	≥0-6°C, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and ≤ 6°C	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Purgeable Organic Halides	9021	Water	Glass; no	≤ 6°C	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
(POX)			headspace		
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics-Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	≤ 6°C	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	≤ 6°C	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, ≤ 6°C	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	≤ 6°C	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR3 Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR3 Hexavalent	218.7	Water	HDPE or	$Na_2CO_3/NaHCO_3/(NH_4)_2$	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Chromium			propylene	SO_4; pH>8	
UCMR3 Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR3 Hormones	539	Water	Amber glass	$Na_2S_2O_3$, 2-mercaptopyridine-1-oxide, sodium salt	28 Days
UCMR3 Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days
UCMR3 Volatiles	524.3	Water	40 mL amber glass vials	Ascorbic acid. Maleic acid pH~2	14 Days
UCMR3 1, 4 Dioxane	522	Water	Glass	Na_2SO_3 $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6°C	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	≤ 6°C	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	≤ 6°C with benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil	ASTM D3328 (prep); ASTM D5739	Product	10mL glass vials	≤ 6°C	N/A

Cl_2 = Chlorine
FP = Teflon® or other fluoropolymer

HCL = Hydrochloric Acid
H_2SO_4 = Sufuric Acid
HNO_3 = Nitric Acid
$Na_2S_2O_3$ = Sodium Thiosulfate
$NaHSO_4$ = Sodium bisulfate
NaOH = Sodium Hydroxide
NH_4Cl = Ammonium chloride
ZnOAc = Zinc Acetate

Note[1]: 5035/5035A vial kit contains the following:

- 2 vials, preserved by freezing ***or***
- 2 vials aqueous $NaHSO_4$, preserved at ≤6°C* ***and***
- 1 vial methanol preserved at ≤6°C* ***and***
- 1 vial unpreserved stored at ≤6°C*

Note[2] For Method 524.2, ascorbic acid is listed as a preservative when residual chlorine is suspected, unless gasses or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

Note[3] For Method 9315 and 9320, if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation and analysis.

Note[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR3 program.

UNCONTROLLED COPY

Assembly of the Purge Manifold Assembly (PMA)

Each PMA should include the following:

2-1′ sections of tubing, 1-1″ section of tubing, 1-four way valve, 1-two way valve, 1-unidirectional valve, 3-male slip adapters, 1-set of fittings and ferrules, 1-moisture filter and 1-60mL syringe *(Figure 1)*.

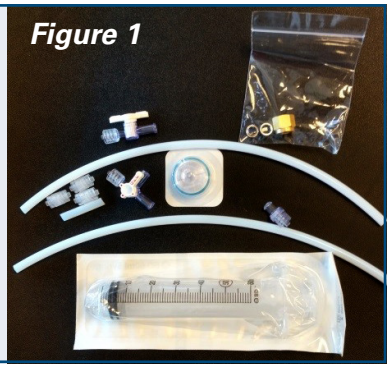
Figure 1

Assemble the manifold in accordance with *Figure 2* and *Figure 3*. Ensure that the orientation of the unidirectional valve matches the figures below or purging the manifold will not be possible.

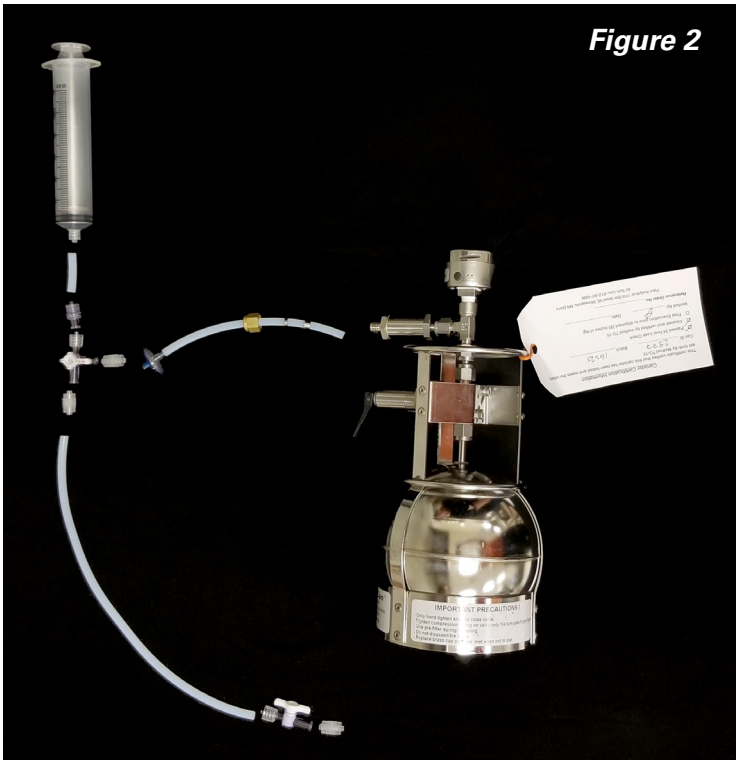
Figure 2


Figure 3

Purging the manifold

(General guidelines, subject to state or client specific guidelines).

1. **DISCLAIMER: Do not open canister until ready to collect sample.**
2. The purge manifold assembly contains four valves, including the canister valve. They are as follows:
   a. Canister valve – attached to the canister. Do not open until ready to collect sample.
   b. 4-way valve – attached to canister assembly via Teflon tubing. This valve has 3 directions "open" at a time with only one closed direction (indicted by the "OFF" tab).
   c. Unidirectional vale – Attached to the male luer fitting on the 4-way valve. This valve will allow volume to exit the assembly, but cannot be pushed back into the assembly in this direction. Once installed, the valve will be active.
   d. 2-way valve – This is the valve closest to the sampling point. It is used to close off the sampling point to allow purging of ambient air in the Teflon lines prior to sampling. It is closed when the white valve is perpendicular to the valve body.
3. After assembly, set the 2-way valve to the closed position. See *Figure 1*.
4. Turn the 4-way valve OFF tab to the direction on the 4-way valve that will allow flow in all three connected directions (the "OFF" tab should face in the only direction without a connection). See *Figure 2*.
5. Attach the syringe to the unidirectional valve with a 1" piece of Teflon tubing. Pull aliquots of the syringe out of the unidirectional valve until the gauge on the canister reaches the required vacuum level. See *Figure 3* and *Figure 4*.
6. Allow the canister and Purge Manifold to sit undisturbed for the desired amount of time, and read the canister gauge. If notable drops in pressure occur, inspect the system for potential leaks and retest. If no pressure change is observed, proceed to step 7. The manifold assembly has now been determined to be free of leaks.
7. Open the two way valve to allow air from the sampling point to fill the lines of the Purge Manifold Assembly. The canister pressure should return to 0.
8. (**Optional**) Using the syringe, purge the line with the desired aliquots of sampling point volume. Multiple purges may be necessary.
9. Open the canister to collect sample.


Figure 1

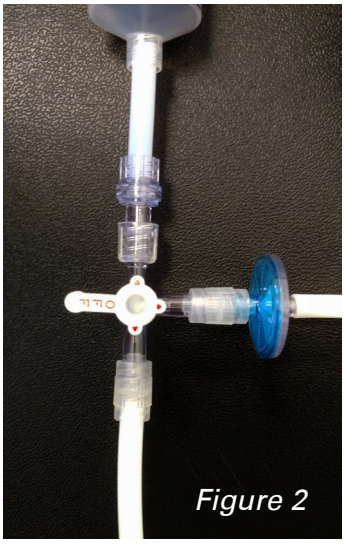
Figure 2

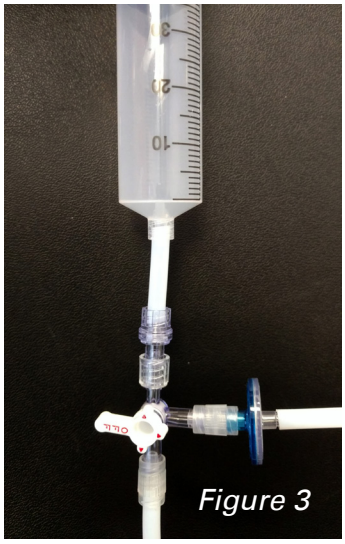
Figure 3


Figure 4

Appendix F

Barr Records Retention Guidelines

Project Records Retention Guidelines

The project file must provide a clear record of the project activities so someone looking at the files years later can retrace the thinking that led to the final product. This table provides general guidance on what should be kept. **Always take into account client-specific records-management requirements.**

	RETENTION PERIOD		
RECORD TYPE	***Temporary**	****Permanent**	**Notes:**
Client Agreements		✓	Save PDF copy in the project electronic folder, the hard copy original in the client-contract file
Client Communications			
▪ All communication dealing with scope, schedule budget		✓	
▪ Formal correspondence		✓	
Email Communications	*Note*: Email is a form of communication, not a record type. Retention is based on content.		
Computations			
▪ Model input		✓	Computations used for model input parameters document and validate the foundation of a computer modeling effort.
▪ Model output		✓	Due to size, it may be preferable to save it in an electronic format. If the input files have been saved, the output can be regenerated.
▪ Handwritten structural computations		✓	
Data	*Note:* In the course of a project, a variety of data in a wide range of formats is gathered and reviewed. Data essential to the recreation of project logic is kept permanently. Other data reviewed may be referenced and discarded.		
▪ Analytical Data		✓	Where applicable, refer to the project quality assurance plan for specific data retention requirements
Drawings		✓	
Models			
▪ Drafts	✓		
▪ Final		✓	
Barr Reports	Includes Barr generated reports, specifications, contract documents and deliverables		
▪ Drafts	✓		Drafts are generally not kept. Exceptions are drafts submitted to agencies, which are kept permanently.
▪ Final		✓	A hard copy of the final deliverable is sent to the Library for permanent storage.
▪ Duplicates	✓		Duplicate copies are discarded or sent to the client when no longer needed.
Third party generated resources			
▪ Aerial photos		✓	
▪ As Built Drawings		✓	Save only those that are relevant to project logic
▪ Manufacturer's cut sheets		✓	Save only those that are relevant to project logic
▪ Reports by other firms		✓	
▪ Published references		✓	Often referenced in the file and kept in the Library.
Project Invoicing Files		✓	Maintained by accounting
Proposal and related documentation		✓	
Project presentation boards	✓		

* **TEMPORARY** = kept until no longer needed, until superseded, or until project close-out
** **PERMANENT** = kept permanently in the project file

Appendix G

Barr Field Audit Checklist

BARR ENGINEERING COMPANY
FIELD AUDIT PROGRAM

FIELD AUDIT CHECKLIST

Site/Project Number: _____

Date of Audit: _____

Field Personnel

Name	Title
_____	_____
_____	_____

Auditing Personnel

Name	Title
_____	_____
_____	_____

1.0 Advance Preparation for Sampling

A. Coordination

1. Does the State, EPA or client need notification of sampling at this site Was that completed? ___

2. Were appropriate sample containers obtained from the laboratory? ___

3. Were sample containers received in good condition? ___

B. Purging and Samping Equipment

The Barr Engineering Company Field Work Check Lists provides a comprehensive overview of the items necessary for successful field event. Sections include: project reference material, miscellaneous tools and supplies, transportation, pumps, bailers, power supplies, documentation and labeling, decontamination, health and safety, other personal gear.

Has a Field Work Check List been completed for the event? ___

If no field work check list was completed, does the field technician have all the proper equipment to perform proper groundwater sampling operations based on the project specific requirements? ___

2.0 Preliminary Field Work

A. Water Level Measurements

1. Was the water level read to the nearest 0.01 foot? ___

2. Was a product interface probe necessary to measure LNAPL or DNAPL? ___

3. Was the water level recorded on the Field Log Data Sheet? ___

4. Was the water level verified with a second reading? ___

5. Was the water level marker decontaminated appropriately? ___

2.0 Sampling of Monitoring Wells

A. Well Purging/Stabilization

1. Verify the correct order of purging/sampling is being followed? ___

2. Field stablization parameters should be measured after several existing well volumes have been removed. Typically, between 3 and 5 well volumes are removed with stablization readings obtained after the third, fourth and fifth column volumes. Was this or equivalent completed? ___

3. Target stablization criteria is given below:

   Temperature +/- 0.1oC
   Specific Conductance (temperature corrected EC) +/-5%
   Dissolved Oxygen +/- 0.5 mg/L
   Redox Potential (-50 to +50) +/-20 mV
   (-100 to +100) +/- 40 mV
   (-200 to +200) +/- 60 mV
   Turbidity: < or = to 10 NTU or +/- 5% if > 10 NTU

   Was the target stabilization criteria met prior to sampling? ___

4. Was calibration of all field instrument completed and documented prior to sampling? ___

5. Was documentation completed as purging activities progressed? ___

6. For low-yielding wells, were they purged dry and allowed to recharge? ___

7. Were a minimum of 3 to 5 well volumes of removed? ___

8. If containerization of purge water is required was it performed? ___

9. Was care given to avoid placing clean sampling equipment (hoses, lines, etc.) on the ground or other potentially contaminated areas prior to use at the well? ___

10. Was appropriate or required purging equipment employed? (i.e., bladder, peristaltic, submerible pumps, clean disposable tubing, etc.) ___

11. Were decontamination procedures for non-dedicated equipment employed? ___

B. Sample Collection

1. Was a clean bailer and line used for sample collection? ___

2. Was the bailer slowly lowered into the well (minimizing aeration)? ___

3. Was the sampling completed "in-line" using dedicated equipment? ___

4. Were vehicles or generator running during sample collection? ___

5. Were the vehicles or generators downwind from the monitoring point? ___

5. Were new sampling gloves worn at the time of collection? ___

6. Were dirty gloves replaced as necessary? ___

7. Were containers filled in the correct order? (i.e., volatiles, semivolatiles, metals, general chemical) ___

8. Were samples filtered as necessary (0.45 micron)? ___

9. Were in-line filtered employed for dedicated wells? ___

10. Was a chain-of-custody completed at the monitoring point? ___

11. Were field QA/QC samples collected as required? ___

12. Were samples placed for "storage" within an acceptable time-frame and on ice (@4oC)? ___

13. Was all non-dedicated or disposable sampling equipment decontaminated as required? ___

Comments:

Appendix H

MPCA COC and Instructions

Minnesota Pollution Control Agency

MPCA Chain-of-Custody Form revision 2017.0328

*** indicates a required field**

Work Order Number: **COC Type:** **Page:** 1 of

Turnaround Time: **COC ID:**

FOR LAB USE ONLY

Lab Work Order Sticker

PROJECT/CLIENT INFO		**LABORATORY**
Facility Code:*	Program Code (MDH Lab Only):	Lab Name:
Project Name:*	Project Task Code:*	Address:
Project Manager:		
Potential Hazard?	If yes, add information to **Sampler Comments** field	EPA Lab ID:*

SAMPLE DETAILS

SAMPLE TYPES
Sample=Routine Sample
QC-FB=Field Blank Sample
QC-FR=Field Replicate Sample
QC-TB=Trip Blank Sample
QC-EB=Equipment Blank
Treated-Mid=Treatment system sample
Treated-Post=Treatment system sample

SAMPLING METHODS
G=Grab sample
CT=Composite, time-paced w/AS
CF=Composite w/AS
D-T=Discrete,time-paced w/AS
D-F=Discrete,flow-paced w/AS
SW-GAS=Gas Sampling
Unknown=Unknown

LAB MATRICES
DW=Drinking Water
NW=Nonpotable Water
SD=Soil/Solid
AR=Air
BL=Biological Material
OT=Other
TS=Tissue

FIELD MATRICES
Wtr-Ground=Groundwater
Wtr-Surf=Surface Water
Wtr-Drink=Drinking Water
QC-BLANK=Artificial Blank Water
Leachate=Leachate Sample
Air-Indoor=Indoor Air
Gas-Soil=Soil Gas

ANALYSIS REQUESTED

MN Location Identifier*	Field Name	Sample Type*	Start Date* (mm/dd/yyyy)	Start Time* 24 hr (hh:mm)	Depth Start	Depth End	Depth Units (m or ft.)	Sampling Method*	End Date (mm/dd/yyyy)	End Time 24 hr (hh:mm)	Lab Matrix*	Field Matrix*	AIS	Sampler Comments (filter volume, special handling, etc.)	# of Cont	PRESERV.	FF	ANALYSIS*	Lab Sample No.	#
																				1
																				2
																				3
																				4
																				5
																				6
																				7
																				8
																				9
																				10

Complete **ONLY** if Method is CT, CF, D-T, or D-F

Sampler's Name:*	**Phone#:**
Sampler's Signature:*	
Sampler's Organization:	

Billing Organization:	**Acct#:**
Address:	
Courier Name:	**Tracking#:**

Receiving Comments:

Relinquished By/Affiliation	**Date/Time**	**Accepted By/Affiliation**	**Date/Time**
(Sampler)			

Instructions for Filling out the MPCA Chain of Custody (COC) Form (revision 2017.0328)

***indicates a required field**

1. The MPCA COC is divided into six sections – 1) the header, 2) Project/Client Info, 3) Laboratory, 4) Sample Details, 5) Analysis Requested, and 6) the footer. All six sections must be filled out.

2. Header Section:
   This section contains the work order number, COC type, Turnaround Time, and COC ID
   a. **Work Order Number**: This usually is populated for COCs prepared for non-state laboratories. The Work Order number is obtained from the MPCA.
   b. **COC Type:** "Standard" for routine sample collection. Designate as "Civil" or "Criminal" for samples that likely will be used in court cases. The laboratory may have an additional charge to process "Civil" or "Criminal" COC forms. Most projects will use a "Standard" COC.
   c. **Turnaround Time:** "Standard", "**Rush**", or "**24 Hour**". The latter two choices must be bolded so they stand out on the COC. Note that some analyses cannot be performed within a 24-Hour turnaround time.
   d. **COC ID:** This field only is populated by the EDGE software produced by EarthSoft Inc.

3. Project/Client Info Section:
   This section contains general information about the project for which the samples were submitted.
   a. **Facility Code*:** Code is obtained from the MPCA Program staff and is required to load data correctly into the MPCA's database.
   b. **Program Code:** This is used for samples submitted only to the MDH laboratory. This 2-digit code is obtained from the MPCA.
   c. **Project Task Code*:** Code is obtained from the MPCA Program staff and is required to load data correctly into the MPCA's database.
   d. **Project Name*:** listed on the Work Order for the project or is available from Program staff.
   e. **Project Manager:** is the MPCA Project Manager listed on the work order.
   f. **Potential Hazard?:** Entered as "Y" or "N". A value of "Y" designates that the sample could be hazardous for the laboratory staff to handle. The sampler must enter an explanation in the "Sampler Comments" section for any "Y" values.

4. Laboratory Section:
   This section contains the contact information for the laboratory.
   a. **Lab Name:** List the name of the laboratory
   b. **Address:** List the address of the laboratory
   c. **EPA Lab ID*:** List the EPA provided lab identifier.

5. Sample Details Section:
   a. **MN Location Identifier***: the location identifier for the site. For most wells, this is Minnesota Unique Well Number. For streams, this is the MPCA stream identifier. For lakes, this is the DNR lake identifier. For Gas or Flares, this is the designator set by the landfill programs. Check with MPCA Program staff for instructions on the format for the location identifier.
   b. **Field Name**: the more common name associated with location. For wells this could be 'MW-1' or for streams this might include an associated biological monitoring station identifier.
   c. **Sample Type*:** designates the type of sample collected. Routine samples have a Sample Type code of "Sample". Trip blank samples have a Sample Type code of "QC-TB". There are many other choices for the Sample Type code. Choose as appropriate.
   d. **Start Date*:** the sample collection date in the mm/dd/yyyy format.
   e. **Start Time*:** the sample collection time in military or 24-hour clock format. For example, 0110 would be 1:10 am and 2200 would be 10:00 pm.
   f. **Depth**

i. **Start:** indicates the starting depth where the sample was collected. Used for integrated lake water samples and some selected programs. Check with the MPCA Program staff to verify if these designators are required.
ii. **End:** indicates the ending depth where the sample was collected. Used for integrated lake water samples and for selected programs. Check with the MPCA Program staff to verify if they are needed.
iii. **Units**: indicates the units used to determine depth. Use either meters (m) or feet (ft.)

g. **Sampling Method*:** indicates the sampling method used to collect the sample. Check with MPCA program staff on the appropriate code to use
h. **End Date**: the sample collection end date in mm/dd/yyyy. Used ONLY for composite sampling.
i. **End Time**: the sample collection end time in military or 24-hour clock format. For example, 0110 would be 1:10 am and 2200 would be 10:00 pm. Used ONLY for composite sampling.
j. **Lab Matrix*:** indicates to the laboratory the analytical method type to use. Commonly used lab matrix codes are listed on the COC form. Check with MPCA program staff on the appropriate code to use.
k. **Field Matrix*:** further qualifies the sample type. Commonly used field matrix codes are listed on the COC form. Check with MPCA program staff on the appropriate code to use.
l. **AIS:** entered as "Y" or "N". This identifies if any water comes from a source where aquatic invasive species (AIS) have been identified. If AIS is "Y", the lab must separate any remaining sample volume and provide special handling procedures to eliminate the organisms. The laboratories may charge an additional fee for this service.
m. **Sampler Comments:** If needed, this field can be filled in to provide additional information about the sample, such as the filter volume for chlorophyll a samples or any special handling that is required.
n. **# of Containers:** is the total number of containers for collected for a Location Identifier.

6. Analysis Requested Section
This section specifies the preservatives added to the samples and the analytical methods to be used.
a. **PRESERV.**: List which acid or base was used to preserve the sample. Do not list the concentration. If the sample was not preserved, list "None".
b. **FF:** Abbreviated for Field Filtered. Entered as "Y" or "N". This indicates if the sample was filtered by the collector in the field.
c. **ANALYSIS*:** Fill in the analyses that are requested for the Location identifier. Designate the analysis using the method source and number, such as EPA 524.2.
d. The Lab Sample No.: this field is populated by the laboratory when the samples during sample login.

7. Footer Section:
a. **Sampler's Name*:** Print the sampler's name in this section.
b. **Phone#:** List the sampler's contact phone number.
c. **Sampler Signature*:** the sampling staff sign the COC in this section.
d. **Sampler's Organization:** Print the sampler's organization in this section.
e. **Billing Organization/Acct#/Address:** If applicable**,** indicate the name, account number, and address for the organization that will be billed for the analysis.
f. **Courier Name:** If applicable, indicate the name of the courier company transporting the sample container(s).
g. **Tracking#**: If applicable indicate the tracking number of the sample shipping container.
h. **Relinquished By/Affiliation:** The sampling staff signs in this box and lists their affiliation when the samples are submitted to the courier for transport to the laboratory. The courier transporting the samples also signs in this box and lists their affiliation after the samples are transported to the laboratory and the date/time of release of the samples to the laboratory.
i. The laboratory staff will accept the samples by filling in the Accepted By/Affiliation box.
j. **Date/Time:** the date and time the samples are relinquished to the courier or laboratory.

8. **If using EDGE (the EQuIS Data Gathering Engine), information on the COC can be pre-populated.**

The Standalone COC template is being provided by the MPCA for use by labs and contractors when the EDGE field collection tool is not used to generate a COC.

This file can be completed electronically or can be printed and completed by hand. It is consistent with but does not have the complete functionality of the version of the COC that is embedded in EDGE.

The accompanying instruction sheet should be used, whether using the standalone or EDGE version. This will help ensure that all necessary information gets to those testing labs that are adopting the EQuIS Lab_MN format.

Stuart Arkley
MPCA
February 14, 2014

Appendix I

References

References

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