

***2013 Wild Rice and Water Quality Sampling  
Report***

***Dark River and Dark Lake***

***Prepared for  
United States Steel Corporation,  
Minnesota Ore Operations - Minntac***

***December 2013***

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Report***

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# 2013 Wild Rice and Water Quality Sampling Report

## December 2013

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## Executive Summary

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Barr Engineering Company (Barr) was retained by U. S. Steel Corporation (U. S. Steel) to conduct a wild rice survey of the Dark River and Dark Lake located downstream of the U. S. Steel Minntac tailings basin facility (see Figure 1 and Figure 2) as requested by Minnesota Pollution Control Agency (MPCA) in letters dated June 28 and July 3, 2013 (Appendix A). The scope provided by MPCA for the wild rice survey includes a field survey to determine if wild rice is present and collecting grab water quality samples for sulfate analysis at locations where wild rice is identified.

Barr conducted the wild rice survey field work in August 2013 using a protocol similar to the one used by the 1854 Treaty Authority, “Wild Rice Monitoring and Abundance in the 1854 Ceded Territory (1998–2008)” (reference (1)) and other vegetation plot data surveys designed to quantify in situ plant species (e.g., A Handbook for Collecting Vegetation Plot Data in Minnesota: The Relevé Method 2007 (reference (2))). Surface water quality sample collection methods followed Barr’s standard operating procedure (SOP), Collection of Surface Water Samples (Appendix B).

Barr observed wild rice in only three single-point locations (density 1) on the northwest side of Dark Lake (Figure 2). A grab water quality sample collected at this location exhibited a sulfate concentration of 176 mg/L (Figure 3). Additionally, Barr conducted plant identification and sampling as part of quality control and quality assurance measures during the survey.

# 1.0 Introduction

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United States Steel Corporation (U.S. Steel), Minnesota Ore Operations – Minntac (Minntac) is a taconite pellet production facility located near Mountain Iron, Minnesota. The National Pollutant Discharge NPDES/SDS Permit No. MN0057207, issued by the Minnesota Pollution Control Agency (MPCA), authorizes seepage discharges from the Minntac tailings basin facility. Surface seepage from the tailings basin to the Dark River Watershed is monitored at NPDES/SDS Permit Outfall SD001, which is considered to be the headwaters of Dark River.

In a letter dated June 28, 2013 (Appendix A), the MPCA requested that U. S. Steel develop information regarding the presence of wild rice in the following receiving water bodies located downstream of SD001:

- Dark River—from the tailings basin discharge at SD001 to Dark Lake
- Dark Lake

The letter outlined the requirements for a literature search, field survey, and water quality sampling and analysis to develop information regarding to the presence of wild rice.

In a letter dated July 3, 2013 (Appendix A), the MPCA refined the request for information and removed the request to conduct a literature search. U. S. Steel retained Barr Engineering (Barr) to conduct this work. This report summarizes the results of the wild rice and macrophyte surveys and water quality sample analysis (Section 2.2) conducted by Barr in August 2013.

## **2.0 Wild Rice Survey and Water Quality Sampling**

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In August 2013, Barr carried out wild rice field surveys in Dark River and Dark Lake and collected water quality samples in accordance with the wild rice survey request from MPCA (Appendix A). This section presents the methodologies and results of this survey and water quality sampling.

### **2.1 Wild Rice Survey Methodology**

#### **2.1.1 Purpose**

The purpose of wild rice surveys and water quality sampling at wild rice stands was to identify and characterize wild rice stands present in the study area and measure sulfate levels in the water at these stands within the project area.

The method used for the wild rice survey was similar to the one used in reference (1) and other vegetation plot data surveys designed to quantify in situ plant species (e.g., reference (2)).

#### **2.1.2 Qualitative Density Measurements**

A wild rice density rating scale of 1 to 5, used by the 1854 Treaty Authority (reference (1)), was applied to each observation of wild rice. The density rating was used to qualitatively assess the density of wild rice over a given area and relates ratings to the approximate percent coverage of wild rice. Table 2-1 relates wild rice density scale ratings to the approximate percent coverage of wild rice.

**Table 2-1 Wild Rice Density Scale**

<b>Wild Rice Density Classification</b>	<b>Description</b>
1	<10% Wild Rice Coverage
2	10–25 % Wild Rice Coverage
3	25–50 % Wild Rice Coverage
4	50–75% Wild Rice Coverage
5	>75 Wild Rice Coverage

### **2.1.3 Macrophyte Sampling Methods and Survey Staff Training**

As part of implementing quality control and quality assurance measures for wild rice surveys, Barr carried out plant identification training facilitated by in-house botanists and ecologists. The objectives of the training were to ensure that staff could distinguish wild rice from other macrophytes commonly growing in similar habitats in northern Minnesota and better assess wild rice habitat characteristics (including the presence and abundance of other macrophytes). Staff carried out macrophyte identification and density assessments in habitats where wild rice grows or could potentially grow. A subset of macrophyte observations included collection of plant specimens to verify field identification.

### **2.1.4 Water Quality Sample Collection Methods**

Barr staff followed Barr’s standard operating procedure (SOP), Collection of Surface Water Samples to collect water samples (Appendix B). A grab sample was collected at the location of the wild rice stand in Dark Lake (see Section 2.4). Analysis of the unfiltered sample for sulfate was conducted by ion chromatography (EPA 9056/EPA300.0).

## **2.2 Wild Rice Survey Results**

Figure 2 includes the extent of the wild rice survey; wild rice was identified in only three single-point locations (density 1) on Dark Lake. Table 2-2 provides a summary of 2013 wild rice survey outcomes and water quality sample analysis for sulfate. Photographs of wild rice within the study area are included in Appendix C.

**Table 2-2 Wild Rice Field Survey Outcomes**

<b>Water body</b>	<b>River/Stream reach or lake shoreline interval</b>	<b>Wild Rice Survey Outcomes</b>	<b>Sulfate (SO<sub>4</sub><sup>2-</sup> mg/L)</b>
Dark Lake	Northwest side of lake	Three single-point locations within 205 feet (Density = 1)	176 mg/L

### **2.2.1 Dark Lake**

Dark Lake was directly surveyed by kayak. The total paddled survey length was 3.7 miles.

Wild rice observations included:

- One point along the shoreline with 12 stems in an area 8 feet long by 2 feet wide
- One point with a single stem
- One point with 35 stems in an area 15 feet long by 4 feet wide

One water sample and five macrophyte voucher samples were also collected during the course of the survey. Water levels ranged from 20 inches to 21.5 inches deep where wild rice was present (water levels were recorded at minimum once per water body in locations where wild rice was identified).

### **2.2.2 Dark River**

Dark River was directly surveyed by kayak, from approximately one-quarter mile east of Timber Creek to Dark Lake. The total paddled survey length was 7 miles.

Dark River was typically observed to be between 10 and 25 feet wide with water clarity to about 103 cm. Substrate materials ranged from mucky in the deeper areas or pools to sandy in the shallower areas. Beaver dams and snags were prevalent along the river.

Four macrophyte voucher samples were taken during the survey.

There were no occurrences of wild rice growing on the Dark River.

## **2.3 Macrophyte Results**

Approximately 12 different species of macrophytes were observed in addition to wild rice; these are listed in Appendix D. The most frequently documented macrophytes include *Schoenoplectus acutus* (hardstem bulrush), *Nuphar lutea* (yellow pond-lily), *Sparganium* spp. (bur-reeds), and *Stuckenia pectinata* (sago pondweed). Appendix D provides a summary of macrophyte occurrences for Dark River and Dark Lake.

## **2.4 Water Quality Sample Analysis Results**

One grab water quality sample was collected at the location where a wild rice stand was observed in Dark Lake (sample number MT-BKB-01) which had a sulfate concentration of 176 mg/L. The location of this water quality sample is provided in Figure 3. The laboratory sulfate result was reviewed and found to be acceptable as reported by the laboratory.

## 3.0 Summary

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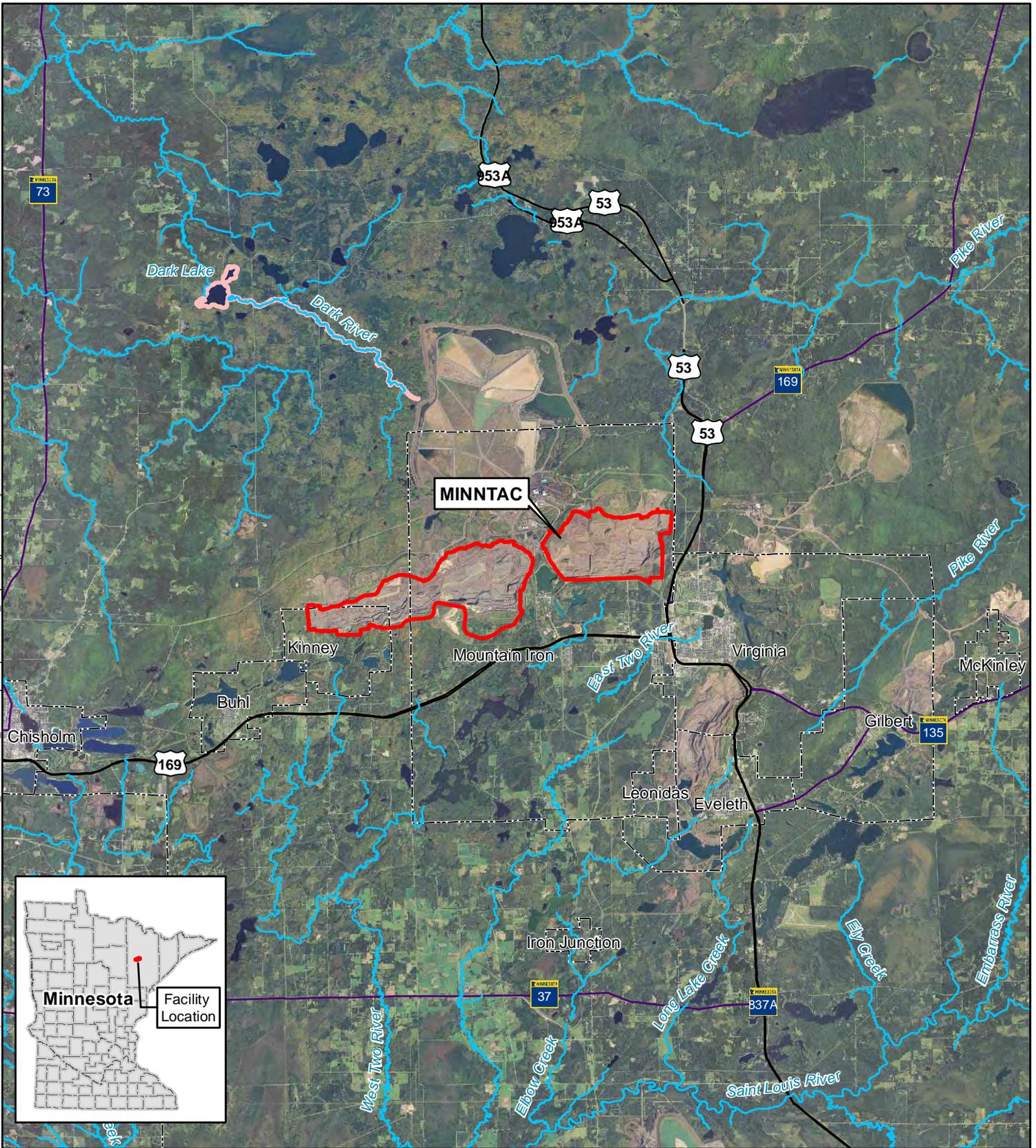
Wild rice was observed in three single-point locations on the northwest side of Dark Lake, all of which received a density rating of “1” (<10% coverage within the stand). The sulfate concentration in that location was 176 mg/L. Water levels of approximately 20 inches recorded at the wild rice locations are consistent with literature documenting optimal water levels for wild rice growth. No wild rice was found on Dark River. Identification of macrophytes within the study area with characteristics similar to wild rice was carried out as part of the survey. The most frequently documented macrophytes include *Schoenoplectus acutus* (hardstem bulrush), *Nuphar lutea* (yellow pond-lily), *Sparganium* spp. (bur-reeds), and *Stuckenia pectinata* (sago pondweed).

## 4.0 References

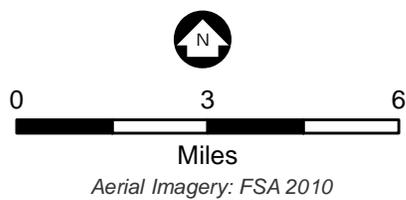
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1. **1854 Treaty Authority.** Wild Rice Monitoring and Abundance in the 1854 Ceded Territory (1998-2008). 2008.
2. **Minnesota Department of Natural Resources.** A Handbook for Collecting Vegetation Data in Minnesota: the Releve Method, second edition. [Online] 2013.  
[http://files.dnr.state.mn.us/eco/mcbs/releve/releve\\_singlepage.pdf](http://files.dnr.state.mn.us/eco/mcbs/releve/releve_singlepage.pdf).

## Figures

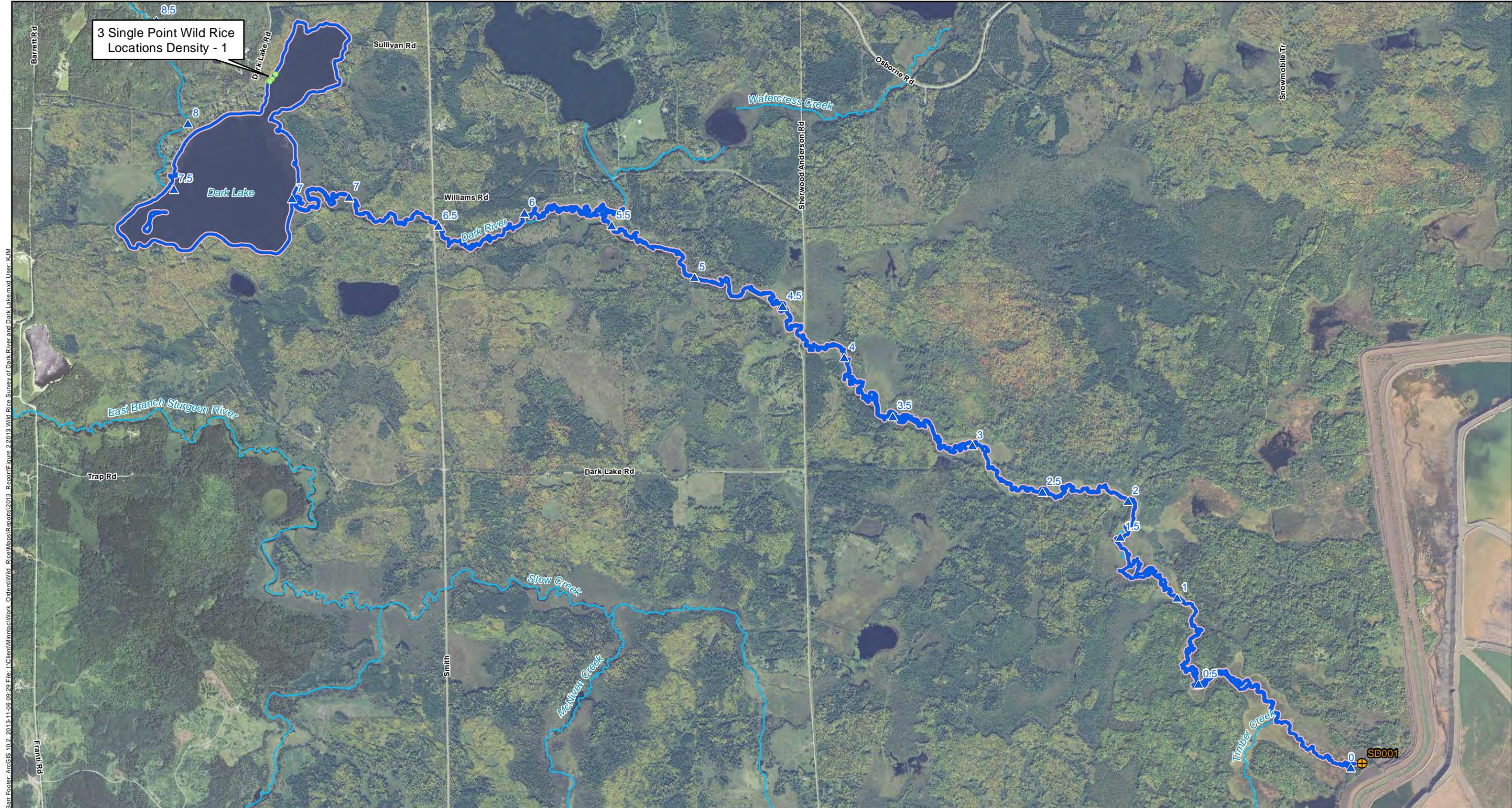


- Pit Boundary
- Rivers & Streams
- Study Area Water Bodies
- US Highway
- State Trunk Highway
- Municipal Boundary

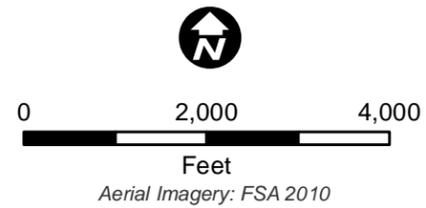


**DRAFT**  
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Figure 1

FACILITY LOCATION  
U. S. Steel - Minntac  
St. Louis County, MN

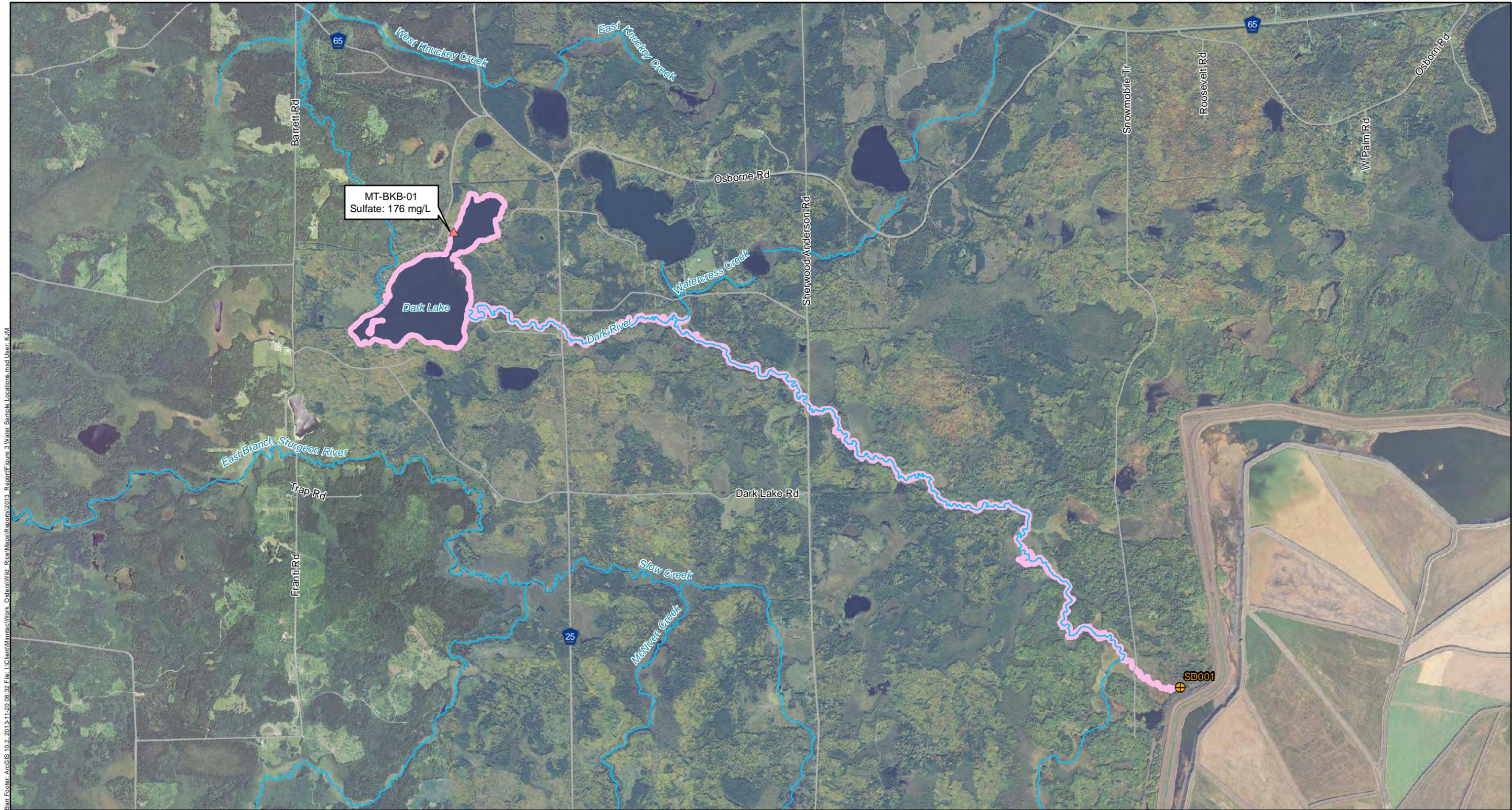


- Wild Rice Density in 2013**
- No Wild Rice Observed
  - 1 <10% Wild Rice Coverage
  - 2 10-25%
  - 3 25-50%
  - 4 50-75%
  - 5 >75% Wild Rice Coverage
- ▲ River Miles
- ⊕ Active Outfall
- Study Area Water Bodies



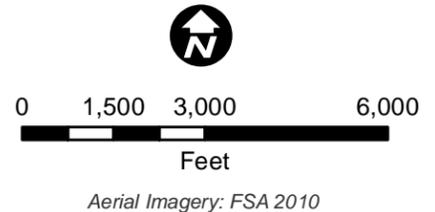
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Figure 2  
2013 WILD RICE SURVEY OF  
DARK RIVER AND DARK LAKE  
U. S. Steel - Minntac  
St. Louis County, MN



Barr, Fowler, ArcGIS 10.2, 2013-11-20 08:32 File: I:\Client\Minntac\Work\_Orders\Wild\_Rice\Maps\Reports\2013\_Report\Figure 3 Water Sample Locations.mxd User: KJM

- ▲ Sulfate Sample Location
- ⊕ Active Outfall
- ~ Study Area Water Bodies



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Figure 3  
 2013 WILD RICE WATER  
 QUALITY SAMPLE ANALYSIS RESULTS  
 U. S. Steel - Minntac  
 St. Louis County, MN

## **Appendices**

## **Appendix A**

### **MPCA Letters**



# Minnesota Pollution Control Agency

520 Lafayette Road North | St. Paul, Minnesota 55155-4194 | 651-296-6300

800-657-3864 | 651-282-5332 TTY | [www.pca.state.mn.us](http://www.pca.state.mn.us) | Equal Opportunity Employer

June 28, 2013

Ms. Chrissy Bartovich  
Director – Environmental  
U.S. Steel Corporation  
Minnesota Ore Operations  
PO Box 417  
Mountain Iron, MN 55768

RE: NPDES/SDS Permit No. MN00570207  
US Steel Corporation – Minntac Tailings Basin Area  
Request for Information on Wild Rice

Dear Ms. Bartovich:

The Minnesota Pollution Control Agency (MPCA) is beginning the process of reissuing the US Steel Minntac Tailings Basin National Pollutant Discharge Elimination/State Disposal System (NPDES/SDS) permit. One of the goals of the MPCA is to protect surface waters used for the production of wild rice. Over the last year, MPCA staff has been working to develop guidance to determine, on a case-by-case basis, what waters of the state are “used for the production of wild rice” and subject to the 10 mg/L sulfate standard under Minn. R. 7050.0224, subp. 2. Such waters may be located downstream of the Minntac Tailings Basin Area.

Due to concerns regarding the concentration of sulfate in the tailings basin and the resulting potential for impact to wild rice resources, the MPCA is requesting the company to gather additional information on the presence of wild rice waters downstream of the tailings basin discharges, including conducting a literature search to identify waters potentially containing wild rice followed by a field survey of those waters identified in the literature search. This information will be important for the permitting process to ensure that appropriate water quality standards are applied and to ensure that surface waters, including those used for the production of wild rice, are adequately protected.

We are requesting the company to include the following receiving waters on the west side of the tailings basin, including any adjacent wetland areas, in the literature search and, as appropriate, in the subsequent field surveys:

## MN0057207 US Steel Corporation - Minntac Tailings Basin Area

- Dark River – from the tailings basin discharge at SD001 to Dark Lake
- Dark Lake [A Minnesota Department of Natural Resources survey conducted on July 30, 2012 identified wild rice in the northeast bay of Dark Lake, (ID# 69-0790-00).]

The company should provide the following information to the MPCA:

1. A literature search for wild rice in the downstream receiving waters listed above potentially impacted by the discharges. Some data sources that may be used to determine the potential for wild rice impacts include Appendix B of the "2008 DNR Wild Rice Report," the most recent "DNR Wild Rice Harvester Survey," and the "1854 Treaty Authority List." For waters listed in the "DNR Wild Rice Report," Ann Geisen may be contacted at 218-833-8625 to gather all the available Department of Natural Resources (DNR) data on those sites. Information on any active or proposed DNR management activities designed to establish, protect, or enhance the wild rice resources of these waters would be helpful.
2. A field survey to observe whether wild rice is actually present in all waters potentially impacted by the discharges that were determined to have potential for wild rice, either based on the literature search above or those that have characteristics which may encourage wild rice production. When the field survey is conducted, it should be conducted by a qualified professional and should take into account the cyclic nature of the growth of this aquatic plant.
3. The company should collect at least one grab sample of water for sulfate analysis in each water where wild rice is found to be present.

The wild rice literature search and field survey work for at least those waters noted above should be conducted in 2013. The company may incorporate into its submittal information from previous wild rice survey work that may have been done in the recent past or work collaboratively with other actual or potential dischargers to these waters on gathering any new information.

On a related note, the MPCA has again this year contracted with University of Minnesota, Limnological Research Center / LacCore Facility staff to conduct field surveys at selected wild rice lakes and streams across the state. Dark Lake (69-0790-00) has been added to the list of potential waters to be sampled on three different occasions over the course of the 2013 field season. MPCA staff will keep you apprised of their initial survey findings once they have had an opportunity to sample Dark Lake. If you have any questions related to these field surveys please contact Gerald Blaha in the MPCA Environmental Outcomes Division at 651-757-2234.

We appreciate your cooperation in this matter. If you have any questions regarding this request, please contact me at 651-757-2405 or by e-mail at [stephanie.handeland@state.mn.us](mailto:stephanie.handeland@state.mn.us).

Sincerely,

Stephanie Handeland  
Hydrologist  
Water Section  
Industrial Division

SH:rm

cc: Tom Moe, US Steel Minntac  
John Thomas, MPCA Duluth Regional Office



# Minnesota Pollution Control Agency

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July 3, 2013

Ms. Chrissy Bartovich  
Director – Environmental  
U.S. Steel Corporation  
Minnesota Ore Operations  
PO Box 417  
Mountain Iron, MN 55768

RE: NPDES/SDS Permit No. MN00570207  
US Steel Corporation – Minntac Tailings Basin Area  
Updated Request for Information on Wild Rice

Dear Ms. Bartovich:

The purpose of this letter is to provide updated information to assist you with the wild rice survey request sent on June 28, 2013. A wild rice survey for the Dark River from the tailings basin to Dark Lake and Dark Lake was requested to provide information necessary for the permitting process. This information will be important for the permitting process to ensure that appropriate water quality standards are applied and to ensure that surface waters, including those used for the production of wild rice, are adequately protected.

The Minnesota Pollution Control Agency (MPCA) discovered wild rice has been identified in Dark Lake during a recent Call for Data in which the Minnesota Department of Natural Resources (MDNR) compiled and submitted a list of waters where wild rice has been identified. These waters are in addition to the list of wild rice waters noted in Appendix B of the MDNR report titled *"Natural Wild Rice in Minnesota – A rice Study document submitted to the Minnesota Legislature by the Minnesota Department of Natural Resources, February 14, 2008."* The recent MDNR list identified the presence of wild rice in Dark Lake (69-0790-00) during a July 30, 2012, survey of the northeast bay of the lake. There is no information related to density or aerial coverage from this survey.

We are requesting the company to include the following receiving waters on the west side of the tailings basin, including any adjacent wetland areas, in the subsequent field survey:

MN0057207 US Steel Corporation - Minntac Tailings Basin Area

- Dark River – from the tailings basin discharge at SD001 to Dark Lake
- Dark Lake [A Minnesota Department of Natural Resources survey conducted on July 30, 2012 identified wild rice in the northeast bay of Dark Lake, (ID# 69-0790-00).]

The company should provide the following information to the MPCA:

1. A literature search for wild rice in the downstream receiving waters listed above is not necessary.
2. A field survey to observe whether wild rice is actually present in all waters potentially impacted by the discharges that were determined to have potential for wild rice or that have characteristics which may encourage wild rice production. When the field survey is conducted, it should be conducted by a qualified professional and should take into account the cyclic nature of the growth of this aquatic plant. A measure of stand health such as a stem count or stem density should be determined where wild rice is present. Where wild rice is encountered, the acreage of wild rice and density of wild rice within that acreage should be determined. As an example, the 1854 Treaty Authority has developed methodology for determining stand density. Wild rice survey reports previously submitted to the MPCA have used an adaptation of this methodology. Please refer to the attached document "*Wild Rice Monitoring and Abundance in the 1854 Ceded Territory (Technical Report 10-01), January 2010*" for guidance on determining stand density.
3. The company should collect at least one grab sample of water for sulfate analysis in each water where wild rice is found to be present.
4. The MPCA Environmental Analysis and Outcomes Division Water Assessment and Environmental Information Section has developed a GIS map showing the location of known wild rice stands in the state. The map is used by MPCA Effluent Limit Unit staff to help evaluate the need for additional monitoring and/or limits in NPDES permits for discharges upstream of potential wild rice production waters. The following information and spatial data for all locations surveyed for wild rice should also be submitted with the final wild rice survey results. These data will supplement the existing data in our wild rice GIS map.
  - GIS point shape file (coordinates) of all wild rice locations with corresponding density factor/percentage of wild rice coverage data for each point/coordinate.
  - GIS point shape file (coordinates) of each grid sampling location.
  - GIS shape file (polygons) showing the extent of wild rice with corresponding acres data for each polygon.

Information to be included in the Final Report should also include the metadata for the above spatial data that describes the accuracy, production method, spatial reference system, and attributes of the dataset. Our preference is to receive metadata that follows the Minnesota Geographic Metadata Guidelines (MGMG) standard, but if it is not possible to produce metadata in the MGMG format, the Federal Geographic Data Committee (FGDC) format will suffice.

The wild rice field survey work for at least those waters noted above should be conducted in 2013 and submitted to the MPCA by December 31, 2013. The company may incorporate into its submittal information from previous wild rice survey work that may have been done in the recent past or work collaboratively with other actual or potential dischargers to these waters on gathering any new information.

Ms. Chrissy Bartovich

Page 3

July 3, 2013

On a related note, the MPCA has again this year contracted with University of Minnesota, Limnological Research Center / LacCore Facility staff to conduct field surveys at selected wild rice lakes and streams across the state. Dark Lake (69-0790-00) has been added to the list of potential waters to be sampled on three different occasions over the course of the 2013 field season. MPCA staff will keep you apprised of their initial survey findings once they have had an opportunity to sample Dark Lake. If you have any questions related to these field surveys please contact Gerald Blaha in the MPCA Environmental Outcomes Division at 651-757-2234.

We appreciate your cooperation in this matter. If you have any questions regarding this request, please contact me at 651-757-2405 or by e-mail at [stephanie.handeland@state.mn.us](mailto:stephanie.handeland@state.mn.us).

Sincerely,



Stephanie Handeland  
Hydrologist  
Water Section  
Industrial Division

SH:rm

cc: Tom Moe, US Steel Minntac  
John Thomas, MPCA Duluth Regional Office

## **Appendix B**

### **Collection of Surface Water Samples SOP**

# STANDARD OPERATING PROCEDURE

## Collection of Surface Water Samples

Revision 5

April 29, 2011

Approved By: Andrea Nord Andrea Nord 4-29-10  
Print QA Manager(s) Signature Date

Kim Johannessen Kim Johannessen 4-29-10  
Print Field Technician(s) Signature Date



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Annual Review of the SOP has been performed and the SOP still reflects current practice.	
Initials: <u>KSJ</u>	Date: <u>2/21/2012</u>
Initials: _____	Date: _____

# Standard Operating Procedures for the Collection of Surface Water Samples

## Purpose

The purpose of this procedure is to describe the collection of water samples for volatiles, semivolatiles, metals, inorganics, bacteria, and dioxin from surface water.

## Applicability

This procedure applies to the collection of surface water samples by the sampling technician(s).

## Definitions

**Headspace.** Air space between the container top and water sample level.

**Holding Time.** Time interval between sample collection and sample analysis.

**Sample Preservation.** The stability of analytes depends upon the proper preservation technique and preservation acceptance criteria as defined by EPA Title 40 of the Code of Federal Regulations and corresponding method criteria.

**Leachate** The liquid product produced when water percolates through any permeable material.

## Equipment

Sampler media	Nitrile Gloves
Pre-cleaned-certified Sampling Containers	Water Quality Meter
Coolers	Sample label
Ziploc® Baggy	Chain of Custody Form
Ice	Lead acetate test paper
Water-proof ink pen or pencil	Acetic acid buffer solution (pH of 4)

## References

Procedures for Ground Water Monitoring, Minnesota Pollution Control Agency Guidelines, September 2006.

EPA: Title 40 of the Code of Federal Regulations

## Discussion

- Surface water stations may include seep locations, lake sampling, influent and or effluent stream or river locations.
- Samples collected from a surface water location; water quality may vary from shore to shore. The sample should be integrated from top to bottom in the middle of the location.
- Samples collected in shallow water (less than 3 feet deep) should be collected at mid-depth, holding the container under the surface until filled. The mouth of the container

should face the flow. Samples can also be collected by the use of a peristaltic pump, with tubing attached to a telescoping pole for larger water bodies. The use of a pole allows access to the mid channel location without disturbing the bottom sediments. Refer to the SOP for peristaltic pump operation.

- When sampling extremely shallow water such as leachate seeps, care should be taken not to disturb the bottom sediments.
- When sampling shallow streams, collection should begin at the furthest downstream point and move upstream so that any disturbances caused by sampling will not affect the quality of the water sampled. When sampling deeper waters, such as rivers, collection should begin first at the upstream point, next to the downstream point, and finally to the sampling point closest to the apparent source of discharge, minimizing contaminants adhering to the sample apparatus.
- All unpreserved sample containers will be rinsed three times with sample water prior to collection as a precautionary measure to be sure containers are uncontaminated.
- Caution will be exercised in filling preserved containers to prevent loss of the preservative.

## **Responsibilities**

The environmental technician(s) is responsible for the proper collection of surface water samples; sample identification; quality control procedures; sample filtering and documentation.

## **Procedure**

1. Obtain sampling media-Approximately one week before the sampling event, the sample containers should be ordered from the laboratory.

**Note:** Container volume, type, and preservative are important considerations in sample collection. Container volume must be adequate to meet laboratory requirements for quality control, split samples, or repeat examinations. The container type or construction varies with the analysis required. The analytical laboratory will preserve the container before shipment. Preservation and shelf life vary; contact the laboratory to determine if an on-hand container is still useful.

2. Put on sampling gloves to protect the sample and skin.

**Note:** New sampling gloves should be used for each location.

3. Prepare sampling containers by filling out the label with the following information:
  - Project number
  - Location identification
  - Individual collecting the samples
  - Date and time of collection
  - Sample analysis (if required by the lab)

4. Remove cap from the first sample container. Make sure to collect low-level mercury and volatile samples prior to all other analytical method samples. Collect metals samples prior to cyanide samples.
5. Fill sampling container (do not overfill).

To ensure sample integrity, collect volatile samples first, then proceed to the least volatile method required for the site.

- A. Volatiles and WI Gasoline Range Organics (WIGRO) – Samples to be analyzed for volatile organics will be collected in two or three 40-ml vials with Teflon®-lined septum caps. Use caution because concentrated acid may be present. Do not rinse glass vials. Hold bottle in one hand, the cap right side up in the other. Allow a slow stream of water to run into the 40-ml vial. The vial should be held at an angle while filling to prevent water from falling directly to the bottom of the container and becoming overly disturbed. While holding the vial vertically, add the water sample until a small meniscus forms on the top of the sample container. Avoid air bubbles and overfilling the vial. Cap tightly, invert the bottle, and tap gently. If any air bubbles appear in the vial, discard and collect sample in a new vial. These samples will be cooled to approximately 4°C. After collecting the required number of vials, insert them in a zip-lock plastic bag and place in a cooler with ice.

If prescribed by site-specific situations a duplicate volatile sample may be collected and field checked with a pH indicator strip to assess the pH of the sample. If the pH is greater than 2, the laboratory will be instructed to reduce the holding time of that day's samples to the 7-day holding period used for unpreserved samples.

- B. Semivolatiles (includes: Pesticides, PCB, Herbicides, BNAs, Dioxin and Furans)– Samples to be analyzed for semivolatile organics will be collected in a 1-liter amber glass jar with a Teflon-lined septum cap for each fraction. Fill container slowly with a minimum headspace and cap tightly. Do not rinse glass containers. Place container directly in a cooler with ice. These samples will be cooled to approximately 4°C.
- C. WI Diesel Range Organics (WIDRO) – Samples to be analyzed for WIDRO are to be collected in a 1-liter amber glass jar with a Teflon-lined septum cap and preserved with 1:1 HCl to a pH or less than 2. Fill container slowly with a minimum of headspace and cap tightly. Do not rinse glass containers. Place container directly into a cooler with ice. These samples will be cooled to approximately 4°C.
- D. Other Organics – Containers may contain acid(s), use caution when handling. Fill containers completely minimizing headspace and avoiding spillage. Place container directly in a cooler with ice.
- E. Metals
  1. Total Metals – Samples to be analyzed for metals will be collected in a 500-mL or 1-liter polyethylene jar with a polyethylene-lined closure. These samples will be preserved in by the lab with a 1:1 (50%) solution of Nitric Acid to reduce the pH of the sample to less than 2.

2. Filtered Metals – Select the appropriate Corning filter size, either 250-ml or 500-ml volume (for further details regarding water sample filtration, see Standard Operating Procedures for Filtering Groundwater Samples). Pour filtered sample into metals sample container, minimizing headspace and avoiding spillage. Use caution handling metals containers because of nitric acid. Place directly in a cooler with ice.
  
- F. Oil and Grease by hexane extraction – Samples to be analyzed for Oil and Grease will be collected in a 1-liter glass jar with a Teflon-lined septum cap preserved to a pH or less than 2 with either 1:1 hydrochloric acid or 1:1 sulfuric acid. These samples will be cooled to approximately 4°C.
  
- G. Cyanide – Samples to be analyzed for cyanide will be collected in a 1-liter polyethylene container with a polyethylene cap and preserved with sodium hydroxide to pH greater than 12 and cooled to approximately 4°C. If elevated levels of sulfur components (i.e. sulfate, sulfide, sulfite, thiosulfate, thiocyanate, and aldehydes) are suspected, test water with a lead acetate test paper (previously moistened with 1-2 drops of acetic acid buffer solutions (pH of 4) to determine the presence of sulfur. If elevated concentrations of sulfur components are detected, contact the project's quality assurance manager, expedite sample shipment to the analytical laboratory on the same day of collection and coordinate rush analyses per the EPA guidelines.
  
- H. Collecting General Chemistry Samples – Samples to be analyzed for sulfate, chloride, carbonate, and bicarbonate will be collected in laboratory supplied containers (plastic or glass per the guidelines included in the analytical method(s)). These samples will be cooled to approximately 4°C.
  
- I. Bacteria – Plastic bottles or glass containers preserved with 10 mg of sodium thiosulfate are used for bacterial sample preservation. Care should be taken not to contaminate the container before collecting the sample. Fill the container within 1 inch of the top. This allows the laboratory to shake and mix the contents before analysis. Place directly in a cooler with ice and cool to approximately 4°C.  
**Note:** 6 hour technical holding time for analysis.

### **Collecting Quality Control Samples**

The effectiveness of the sample handling techniques is monitored by collecting both preserved and unpreserved field blank samples. For additional information, consult the Barr Engineering Co. SOP for the Collection of Quality Control Samples.

Field (or Masked) duplicate samples will be collected to measure relative sampling (and laboratory) precision. The ratio of quality control samples are generally 1 field blank/field duplicate per twenty samples, however, specific project requirements may be determined by the QAPP/SAP for the project. These samples are collected at the same time using the same procedures, equipment, and types of containers as the required samples. They are also preserved in the same manner and are either co-located or split and submitted for the same analyses as the native sample(s).

Trip blank samples are only applicable when sampling/analyzing for volatile organics. Their purpose is to determine if contamination has occurred as a result of improper sample

container cleaning, contaminated blank source water, sample contamination during storage and transport due to exposure to volatile organics, or other environmental conditions during sampling and analysis. The water will be free of contaminants. The trip blanks are prepared, sealed and labeled appropriately at the lab, and transported to the field in the same containers as the sample vials. These blanks are not opened in the field. They are transferred to the coolers designated for volatile sample storage and transport and accompany the samples to the analytical laboratory.

Field blank samples (or Rinsate Blanks) are used to evaluate the effects of sampling cross-contamination caused by inadequately decontaminated equipment. Their purpose is to determine if contamination has occurred as a result of improper equipment cleaning. Field blanks are prepared onsite by pouring analyte-free water through decontaminated sample collection equipment (bailer or pump) and collecting the rinsate in the appropriate sample container. The field blanks will be handled in the same manner as the sample group for which they are intended (i.e., blanks will be stored and transported with the sample group).

The volume of the sample obtained should be sufficient to perform all required analyses with an additional amount collected to satisfy the needs for quality control, split samples, or repeat examinations. The QA Staff should be consulted for any specific volume requirements.

The elapsed time between sample collection and initiation of each laboratory analysis will fall within a prescribed time frame. Holding times for samples required by this project are prescribed by EPA: Title 40 of the Code of Federal Regulations.

### **Water and Soil Sample Storage**

The samples will be bubble wrapped or bagged immediately after collection, stored in a sample cooler, packed on double bagged wet ice and accompanied with the proper chain of custody documentation. Samples will be kept cold (approximately 4°C) until receipt at the laboratory, where they are to be stored in a refrigerated area. Custody seals may be present, but at minimum, the coolers must be taped shut with three straps of fiberglass tape. All samples will be kept secured to prevent tampering. If sample coolers are left in a vehicle or field office for temporary storage, the area will be locked and secured. The coolers must be delivered to the laboratory via hand or overnight delivery courier in accordance with all Federal, State and Local shipping regulations.

**Note:** Samples may have to be stored indoors in winter to prevent freezing.

After collection, all samples should be handled as few times as possible. Samplers should use extreme care to ensure that samples are not contaminated. If samples are placed in a cooler, samplers should ensure that melted ice cannot cause sample containers to become submerged, as this may result in cross-contamination. Plastic bags, such as Ziplock® bags, should be used when small sample containers (e.g., VOC vials) are placed in coolers to prevent cross-contamination.

Some compounds can be detected in the parts per billion and/or parts per trillion range. Extreme care will be taken to prevent cross-contamination of these samples. A clean pair of new, disposable gloves will be worn for each sample location. Sample containers for source samples or samples suspected of containing high concentrations of contaminants are placed in separate plastic bags and coolers immediately after collecting, preserving and tagging.

Sample collection activities will proceed progressively from the least contaminated area to the most contaminated area (when known).

### **Disposal**

All waste generated by this process will be disposed of in accordance with Federal, State and Local regulations. When feasible, implement procedures to minimize environmental pollution.

### **Documentation**

The technician(s) will document the type and number of samples collected during each field event. All sample information will be documented in the field notebook, field log data sheet and chain-of-custody record.

### **Attachments**

- Attachment 1: Chain of Custody Form
- Attachment 2: Sample Label
- Attachment 3: Custody Seal – if applicable
- Attachment 4: Field Sampling Report
- Attachment 5: Field Log Data Sheet

# Attachment 1 Chain of Custody Form

<b>Chain of Custody</b> 4700 West 77th Street <b>BARR</b> Minneapolis, MN 55435-4803 (952) 832-2600										Number of Containers/Preservative										COC _____ of _____						
Project Number:										Water					Soil					Project Manager: _____  Project QC Contact: _____  Sampled by: _____  Laboratory: _____						
Project Name:										VOCs (HCl) #1 SVOCs (unpreserved) #2 Dissolved Metals (HNO <sub>3</sub> ) Total Metals (HNO <sub>3</sub> ) General (unpreserved) #3 Diesel Range Organics (HCl) Nutrients (H <sub>2</sub> SO <sub>4</sub> ) #4					VOCs (sred MeOH) #1 GRO, BTEX (sred MeOH) #1 DRCO (sred unpreserved) Metals (unpreserved) SVOCs (unpreserved) #2 % Solids (plastic vial, unpres.)											
Sample Origination State ____ (use two letter postal state abbreviation)																										
COC Number:										Total Number Of Containers																
Location	Start Depth	Stop Depth	Depth Unit (m./ft. or in.)	Collection Date (mm/dd/yyyy)	Collection Time (hh:mm)	Matrix		Type		QC	VOCs (HCl) #1	SVOCs (unpreserved) #2	Dissolved Metals (HNO <sub>3</sub> )	Total Metals (HNO <sub>3</sub> )	General (unpreserved) #3	Diesel Range Organics (HCl)	Nutrients (H <sub>2</sub> SO <sub>4</sub> ) #4	VOCs (sred MeOH) #1	GRO, BTEX (sred MeOH) #1	DRCO (sred unpreserved)	Metals (unpreserved)	SVOCs (unpreserved) #2	% Solids (plastic vial, unpres.)	Total Number Of Containers		
						Water	Soil	Grab	Comp.																	
1.																										
2.																										
3.																										
4.																										
5.																										
6.																										
7.																										
8.																										
9.																										
10.																										

Common Parameter/Container - Preservation Key										Relinquished By: _____		On Ice? _____		Date _____		Time _____		Received by: _____		Date _____		Time _____	
#1 - Volatile Organics = BTEX, GRO, TPH, 8260 Full List #2 - Semivolatile Organics = PAHs, PCP, Dioxins, 8270 Full List, Herbicide/Pesticide/PCBs #3 - General = pH, Chloride, Fluoride, Alkalinity, TSS, TDS, TS, Sulfate #4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN										Relinquished By: _____		On Ice? _____		Date _____		Time _____		Received by: _____		Date _____		Time _____	
										Samples Shipped VIA: <input type="checkbox"/> Air Freight <input type="checkbox"/> Federal Express <input type="checkbox"/> Sampler <input type="checkbox"/> Other: _____										Air Bill Number: _____			

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

H-RLG-STD-FORM-8/Chain of Custody Form 2009 RLG Rev. 09/01/09

Attachment 2  
Example - Sample label



Client \_\_\_\_\_

Project Number \_\_\_\_\_

Date: \_\_\_\_\_ Time \_\_\_\_\_

Preservative: \_\_\_\_\_

Sampled By: \_\_\_\_\_

Sample Location: \_\_\_\_\_

\_\_\_\_\_

Attachment 3  
Custody Seal – if applicable

<b>Custody Seal</b>			
Date _____	Project _____		
Signature _____	Container# _____	of _____	

Attachment 4  
Field Sampling Report



**FIELD SAMPLING REPORT**

---

**Date:**

**Project:**

**Contact:**

Barr Engineering Company  
4700 W. 77th Street  
Minneapolis, MN 55435-4803

**Field Sampling**

**Field Report**

Attachments:

- 
- 
- 
- 
- 

**Laboratory Analysis Status**

\_\_\_\_\_  
<Name inserts here>  
Environmental Technician

Document1

\_\_\_\_\_  
Barr Engineering Company · 4700 W. 77th Street · Minneapolis, MN 55435-4803 · 952/832-2600



Attachment 5  
Field Log Data Sheet

<b>Client:</b>			<b>Monitoring Point:</b>						
<b>Location:</b>			<b>Date:</b>						
<b>Project #:</b>			<b>Sample time:</b>						
<b>GENERAL DATA</b>			<b>STABILIZATION TEST</b>						
Barr lock:									
Casing diameter:		Time/ Volume	Temp. °C	Cond. @ 25	PH	ORP mV	D.O.	Turbidity Appearance	
Total well depth:*		NA							
Static well level:*									
Water depth:*									
Well volume: (gal)									
Purge method:									
Sample method:									
Start time:		Odor:							
Stop time:		Purge Appearance:							
Duration: (minutes)		Sample Appearance:							
Rate, gpm:		Comments:							
Volume purged:									
Duplicate collected:									
Sample collection by:									
Others present:			Well condition:						
MW: groundwater monitoring well			WS: water supply well		SW: surface water		SE: sediment		Other: sump
VOC	Semi-volatile	General	Nutrient	Cyanide	DRO	Sulfide			
Oil, grease	Bacteria	Total Metal	Filtered Metal		Methane	Filter			
Others:									

\* Measurements are referenced from the top of riser pipe, unless otherwise indicated.

## **Appendix C**

### **Study Area Photographs**



Photo 1: Dark Lake – Wild Rice Stand (Density = 1)  
(Wild Rice Point: MT\_BRB\_WR-1\_20130819)



Photo 2: Dark Lake – Wild Rice Stand (Density = 1)  
(Wild Rice Point: MT\_BRB\_WR-3\_20130819)



Photo 3: Dark Lake – Macrophytes located west of Wild Rice  
(Wild Rice Point: MT\_BRB\_WR-3\_20130819)



Photo 4: Dark River – Macrophytes located ½ mile NW of Sherwood Anderson Road  
(Wild Rice Point: MT\_BRB\_NWR-2\_20130822)

## **Appendix D**

### **Macrophyte Table**

### Macrophyte Species Observed in 2013 Survey

Species	Common Name	Dark Lake		Dark River	
		WR <sup>1</sup>	NWR <sup>1</sup>	WR <sup>1</sup>	NWR <sup>1</sup>
		n=2 <sup>2</sup>	n=9 <sup>2</sup>	n=0 <sup>2</sup>	n=12 <sup>2</sup>
<i>Elodea canadensis</i>	Canadian waterweed				1
<i>Glyceria borealis</i>	Northern mannagrass		1		
<i>Lemna trisulca</i>	Star duckweed	1			
<i>Nuphar lutea</i>	Yellow pond-lily	1	3		1
<i>Polygonum amphibium</i>	Water knotweed				1
<i>Potamogeton natans</i>	Floating pondweed	1			1
<i>Sagittaria latifolia</i>	Broadleaf arrowhead				1
<i>Schoenoplectus acutus</i>	Hardstem bulrush	1	5		
<i>Sparganium</i> spp.	Bur-reed				2
<i>Stuckenia pectinata</i>	Sago pondweed		2		
<i>Typha</i> spp.	Cattails		1		
<i>Utricularia macrorhiza</i>	Common bladderwort		1		

<sup>1</sup>WR = sample points at which wild rice was present; NWR = sample points at which no wild rice was present.

<sup>2</sup> n = total number of sample points at which macrophyte presence or absence was documented, categorized by whether wild rice was also present or not.