UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

FEB 08 2018

REPLY TO THE ATTENTION OF:

WC-15J

Mr. Thomas Moe, Environmental Engineer
Minnesota Ore Operations
United States Steel Corporation - Minntac
P.O. Box 417
Mountain Iron, Minnesota 55768

Re: United States Steel Corporation – Minntac Tailings Basin Facility
NPDES Compliance Sampling Inspection Report

Dear Mr. Moe:

On September 12-13, 2017, the U.S. Environmental Protection Agency conducted an NPDES compliance sampling inspection at the U.S. Steel Corporation - Minntac Tailings Basin Facility, in Mountain Iron, Minnesota. The enclosed NPDES Compliance Sampling Inspection Report summarizes the inspection activities and includes the results of samples collected by EPA.

If you have questions or concerns regarding this report, or believe any part of the report is not accurate, please contact Dean Maraldo of my staff at (312) 353-2098 or maraldo.dean@epa.gov.

Sincerely,

Ryan Bahr
Chief, Water Enforcement and Compliance
Assurance Branch, Section 2

Enclosure

cc: Chrissy Bartovich, United States Steel Corporation
Eric Williams, United States Steel Corporation

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CWA COMPLIANCE SAMPLING INSPECTION REPORT
U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 5

Purpose: Compliance Sampling Inspection Report

Facility: U.S. Steel – Minntac Tailings Basin Area Facility
County Road 102
Mountain Iron, MN

NPDES Permit Number: MN0057207

Dates of Inspection: September 12-13, 2017

Facility Representatives:
Chrissy Bartovich, Environmental Director, (218) 749-7485
Tom Moe, Environmental Director, (218) 749-7364
Eric Williams, Water Manager, (412) 302-3624

Report Prepared by:
Dean Maraldo, EPA Region 5 Inspector

EPA Representatives:
Dean Maraldo, EPA Region 5 Inspector; (312) 353-2098
Jonathan Moody, EPA Region 5 Inspector, (312) 886-7570

EPA Inspector Signature: _____

Report Date: 2/7/18

Approver Name & Title:
Ryan Bahr, Chief, Compliance Section 2

Approver Signature _____

Approval Date: 2/7/18

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I. INTRODUCTION

On September 12-13, 2017, I conducted an announced compliance sampling inspection at the U.S. Steel – Minntac Tailings Basin Area facility (Facility) located near Mountain Iron, Minnesota. I was joined by inspector Jonathan Moody from EPA's Region 5 Office of Enforcement and Compliance Assurance. The purpose of the inspection was to collect samples and assess the Facility's compliance with the Clean Water Act (CWA) and the National Pollutant Discharge Elimination System (NPDES) permit for the Facility. The inspection consisted of the following major activities:

- Inspection opening conference;
- Brief interview and discussions with Facility representatives regarding past reported violations, and the status of the Sand River surface seepage collection and return system (SC&R), and the proposed Dark River surface seepage collection and return system;
- Compliance sampling; and
- Closing conference.

This report summarizes the results of the inspection. The following personnel were involved in the CWA inspection of the Facility:

U.S. Steel:	Chrissy Bartovich, Environmental Director Tom Moe, Environmental Director Eric Williams, Water Manager
EPA:	Dean Maraldo, EPA Region 5 Inspector Jonathan Moody, EPA Region 5 Inspector

II. BACKGROUND

The Minntac Tailings Basin is a part of a U.S. Steel (USS) mining operation in St. Louis County, Minnesota. See Figure 1 for an aerial image of the Facility. Figures are included in Appendix A. The Facility was constructed in the 1960's and was issued NPDES/State Disposal System Permit #MN0057207 (permit), authorizing discharges from the Tailings Basin to the Sand and Dark rivers in 1987. The permit expired in 1992. The Tailings Basin sits on a divide between two major watersheds. When the Tailings Basin was constructed, the headwaters of both watersheds were filled in with mine tailings. The principal activity at this Facility is taconite processing. According to USS's 2014 NPDES Permit Application, USS generates an average of 14 million long tons of taconite pellets per year at its Mountain Iron mining operation, and discharges a maximum of 100 gallons per minute (gpm) of wastewater through the Tailings Basin Seep Water Outfall SD001 to the Dark River. The process operations performed at USS are classified by the Standard Industrial Classification (SIC) codes 1011, Metal Mining (Ore Mining and Dressing) – Iron Ores.

III. COMPLIANCE SAMPLING INSPECTION ACTIVITY SUMMARY

III. A. Opening Conference

I arrived at the Facility security building at 7:45 am on September 12, 2017. I was joined by EPA inspector Jonathan Moody. After checking in with the security office we drove to the nearby Administration Building to meet with USS officials and begin the inspection. We arrived at 8:08 am, and were met by USS representatives Chrissy Bartovich, Tom Moe, and Eric Williams. The inspection opening conference began with introductions, and I presented my EPA Inspector Credentials to the USS

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representatives at 8:16 am. Ms. Bartovich mentioned that she copied all of the available documents I requested prior to the inspection to a flash drive. I confirmed that none of the documents were claimed as confidential business information (CBI).

I went over the inspection objectives, including a review of any recent reported violations, review of SC&R operations and efforts to address west side seeps, observing and sampling wastewater seep areas along the Tailings Basin perimeter dike, observing and sampling Timber Creek, and review of information requested. I asked for the name and title of the USS official responsible for NPDES compliance and discharge monitoring report (DMR) certification. Mr. Moe stated that he signs DMRs for the Facility.

III. B. Interview

Review of Recent Violations:

I asked the group to describe any permit compliance issues experienced over the last few years. Mr. Moe mentioned a "3-hour discharge" from outfall SD002 in 2015. He said the discharge, caused by a seep collection issue associated with the SC&R system, was reported in the Facility's June 2015 DMR, and addressed. Mr. Moe also mentioned having trouble meeting limits on a scrubber, and said that they are working with the state to resolve the issue.

Seep Collection Systems:

Mr. Moe described the SC&R system, which includes a series of seep catch basins. The basins capture flow from 13 wastewater seepage locations. The captured seep flow is piped to pump stations and returned to the Tailings Basin. Sheet pile cut-off walls were installed downgradient of each catch basin, down to a depth of approximately 15 feet, to minimize impacts to surface waters. Mr. Moe confirmed that the SC&R system went on line at the end of 2010. I asked how the SC&R system is performing. Mr. Moe stated that they had some clogging issues with catch basin inlets, but the use of tanks and cleanouts has helped to improved performance. Ms. Bartovich added that they've had some "growing pains" related to maintenance. Mr. Moe mentioned having some issues with pump stations and access to pumps.

I asked if they've had any issues with overtopping or bypasses with the SC&R system. Mr. Moe stated that they have not. Ms. Bartovich added that beehive inlets at the catch basins have been effective.

Mr. Moe provided an update on the planned Dark River surface seepage collection and return system (SCRS). He described the SCRS as being similar to the east side SC&R system design, including four seep catch basins with sheet pile cut-off walls, and a pump system to return seep flow to the Tailings basin. Mr. Moe provided an update on status of SCRS design and construction. He said the preliminary design was complete and they are waiting for Clean Water Act Section 404 permit approvals. I asked for a copy of the 404 permit application.

Sampling Overview:

I went over the plan for sampling of seeps along the Tailings Basin dike perimeter, with a focus on the seepage locations identified in USS's *Minntac Western Seepage Collection System – Phase 2 Report* (Hatch, 2014). I also mentioned an interest in observing and collecting samples from wastewater seeps on the east side of the Tailings Basin dike perimeter and Timber Creek. Mr. Moe mentioned that some of the flow discharging at Seep C (see Figure 2 for USS's western seepage locations), comes from the Dark River Pond (see Figure 3). Mr. Moe said a constructed subsurface French drain conveys flow from the Dark River Pond to a ditch along the southwest perimeter of the Tailings Basin Dike, eventually discharging at Seep C. He added that the water in the Dark River Pond is currently used for drill water.

I concluded the interview at 9:00 am on September 12, 2017.

III. C. Sampling Description

At 9:05 am, on September 12, 2017, EPA Inspector Jonathan Moody and I began preparing for sample collection from wastewater seep areas along the Tailings Basin dike and Timber Creek. The objective of the EPA sampling event was to characterize wastewater discharges from the Tailings Basin to the Sand River, Dark River, and Timber Creek watersheds. Photographs referenced in this report are included in the Photo Log (Appendix B). Samples were collected for laboratory analytical parameters, including total metals, total alkalinity, hardness, bromide, chloride, fluoride, total mercury, total suspended solids (TSS), total dissolved solids (TDS), and sulfate. Details regarding the sampling plan and quality assurance can be found in the Quality Assurance Project Plan/Sampling Plan (Appendix C). In addition to collecting samples for laboratory analysis, we used a YSI 556 MPS water quality meter to monitor pH, Specific Conductance, Temperature, and Dissolved Oxygen at each sample location, and additional areas where laboratory samples were not collected. Mr. Moody calibrated the YSI meter on September 12, 2017, at 9:13 am. All buffers and calibration standards were within expiration dates. See buffers and standards in photograph USSM0252.jpg.

Figure 1 provides an overview of sampling stations. Figures 4-11 include aerial images of sampling locations based on Township 59N, Range 18W, section numbers. Table 1 provides a description of the physical sampling locations. Tables 2-4 summarize the results of sample analysis for all fifteen sample stations locations, including locations where only field parameters were measured. At 9:31 am, on September 12, 2017, I began the sampling inspection and was joined by Mr. Moody, Mr. Moe, and Mr. Williams. Mr. Moe and Mr. Williams collected co-located samples and field measurements.

On September 12, 2017, we concluded sampling activities at 4:25 pm. We secured the samples in iced coolers in our locked vehicle overnight.

On September 13, 2017, we returned to the facility at 7:50 am. Mr. Moody re-calibrated the YSI meter at 8:08 am. Prior to sampling we met with Ms. Bartovich, Mr. Moe and Mr. William to discuss the plan for the day. I asked for additional details regarding the design of the seep collection systems. Ms. Bartovich added that the seep collection systems were "designed to collect upper seep flow, not designed to capture deeper flow." We resumed sampling activities at 9:16 am, again joined by Mr. Moe and Mr. Williams.

Table 1. Description of the physical sampling locations.

Sample Station No.	Sample Numbers - Type	Collection Date/Time	Sample Description and Location	Sampling Method and Photos
S01	S01-Water	9/12/2017 9:57 am	Township 59N, Range 18W, Sections 19 and 24; Location of Seep "A" (Hatch, 2014). Flowing seep water under access road. See Figure 5. 47.57985 N; 92.68961 W	Grab sample. Glass grab jar used to collect samples. USSM0253.jpg USSM0255.jpg
S02	S02-Water	9/12/2017 10:23 am	Township 59N, Range 18W, Section 30; Location of Seep "C" (Hatch, 2014). Referred to as "Dark River Pond Seep" by Mr. Moe. See Figures 3 and 4. 47.56834 N; 92.68606 W	Grab sample, collected directly into sample bottles. USSM0256.jpg USSM0257.jpg

Sample Station No.	Sample Numbers - Type	Collection Date/Time	Sample Description and Location	Sampling Method and Photos
S03	S03-Water	9/12/2017 10:50 am	Township 59N, Range 18W, Section 18; Location of Seep "3" (Hatch, 2014). Sampled trickle flow from base of berm. See Figure 6. 47.59813 N; 92.68136 W	Grab sample, collected directly into sample bottles. USSM0258.jpg USSM0259.jpg
S04 D05	S04-Water D05-S04 duplicate	9/12/2017 11:08 am	Township 59N, Range 18W, Section 18; Outfall SD001 and location of Seep "4" (Hatch, 2014). Sampled directly from outfall discharge. See Figure 6. 47.59878 N; 92.67989 W	Grab sample, collected directly into sample bottles. USSM0260.jpg USSM0261.jpg
S06	S06-Water	9/12/2017 11:30 am	Township 59N, Range 18W, Section 7; Location of Seep "7" (Hatch, 2014). Sampled seep pool at base of berm. See Figure 7. 47.61200 N; 92.68116 W	Grab sample, collected directly into sample bottles. USSM0262.jpg USSM0263.jpg
S07	S07-Water	9/12/2017 11:51 am	Township 59N, Range 18W, Section 6; Location of Seep "13" (Hatch, 2014). Sampled seep pool at base of berm. See Figure 8. 47.62307 N; 92.68063 W	Grab sample, collected directly into sample bottles. USSM0264.jpg USSM0265.jpg
S08	S08-Water	9/12/2017 2:03 pm	Township 59N, Range 18W, Section 23; Sampled wetland at foot of berm. Mr. Moe referred to wetland as "background area" and mentioned background monitoring well located in this wetland. See Figure 11. 47.58625 N; 92.60082 W	Grab sample, collected directly into sample bottles. USSM0266.jpg USSM0267.jpg
S09	S09-Water	9/12/2017 3:00 pm	Township 59N, Range 18W, Section 10; Sampled wetland immediately outside of sheet pile at SC&R Catch Basin-12. See Figure 9. Noticed sheet pile cut very low. 47.61222 N; 92.61440 W	Grab sample. Glass grab jar used to collect samples. USSM0268.jpg USSM0269.jpg USSM0270.jpg USSM0271.jpg
S10	S10-Water	9/12/2017 3:15 pm	Township 59N, Range 18W, Section 10; Sampled seepage collected in SC&R Catch Basin-12. See Figure 9. 47.61234 N; 92.61449 W	Grab sample. Glass grab jar used to collect samples. USSM0272.jpg USSM0273.jpg

Sample Station No.	Sample Numbers - Type	Collection Date/Time	Sample Description and Location	Sampling Method and Photos
S11	S11-Water	9/13/2017 9:44 am	Township 59N, Range 18W, Section 15; Sampled wetland immediately outside of sheet pile at SC&R Catch Basin-5. See Figure 10. 47.59992 N; 92.60999 W	Grab sample, collected directly into sample bottles. USSM0275.jpg USSM0278.jpg USSM0279.jpg
S12	S12-Water	9/13/2017 9:49 am	Township 59N, Range 18W, Section 15; Sampled seepage collected in SC&R Catch Basin-5, near inlet. See Figure 10. 47.59991 N; 92.60999 W	Grab sample. Glass grab jar used to collect samples. USSM0276.jpg USSM0277.jpg USSM0280.jpg USSM0281.jpg
S13	S13-Water	9/13/2017 9:55 am	Township 59N, Range 18W, Section 15; Sampled wetland immediately outside of north end of sheet pile at SC&R Catch Basin-5. See Figure 10. 47.59993 N; 92.61001 W	Grab sample. Glass grab jar used to collect samples. USSM0282.jpg USSM0283.jpg
S14	S14-Water	9/13/2017 10:23 am	Township 59N, Range 18W, Section 30; Dark River Pond. Sampled at base of coarse tailing berm at edge of pond. See Figures 3 and 4. 47.55915 N; 92.67916 W	Grab sample, collected directly into sample bottles. USSM0284.jpg USSM0285.jpg USSM0286.jpg
S15	S15-Water	9/13/2017 11:10 am	Township 59N, Range 18W, Section 24; Sampled flowing water in Timber Creek, at mid-stream. Stream width at sampling point was 6 feet. Stream depth at sampling point was 4-6 inches. See Figure 5. 47.56624 N; 92.67951 W	Grab sample. Glass grab jar used to collect samples. USSM0288.jpg USSM0289.jpg USSM0290.jpg
R16	R16-Water	9/13/2017 12:52 pm	Field blank.	Grab sample, collected directly into sample bottles. USSM0291.jpg

Sample Station No.	Sample Numbers - Type	Collection Date/Time	Sample Description and Location	Sampling Method and Photos
CB11	CB11out - Water field parameters	9/12/2017 3:36 pm	Township 59N, Range 18W, Section 10; Field measurements at wetland location immediately outside of sheet pile at SC&R Catch Basin-11. See Figure 9. Noted water staining at level of sheet pile handling holes. 47.61070 N; 92.61397 W	Field measurement, YSI 556 MPS. Glass grab jar used to collect sample. USSM0274.jpg
	CB11in - Water field parameters	9/12/2017 3:43 pm	Township 59N, Range 18W, Section 10; Field measurements of seepage collected in SC&R Catch Basin-11, immediately inside of sheet pile. See Figure 9. 47.61070 N; 92.61397 W	Field measurement, YSI 556 MPS. Glass grab jar used to collect sample. USSM0274.jpg
CB12	CB12in - Water field parameters	9/12/2017 2:50 pm	Township 59N, Range 18W, Section 10; Field measurements of seepage collected in SC&R Catch Basin-12, 20 feet southeast of inlet. See Figure 9.	Field measurement, YSI 556 MPS. Glass grab jar used to collect sample.
DRP Swale	DRPSwale-Water field parameters	9/13/2017 10:35 am	Township 59N, Range 18W, Section 30; Dark River Pond Swale, just downgradient of Dark River Pond. Sampled at south end of swale. See Figure 4. 47.559722 N; 92.680833 W	Field measurement, YSI 556 MPS. Glass grab jar used to collect sample. USSM0287.jpg

We completed sampling activities and returned to the Administration Building parking lot at 11:38 am on September 13, 2017. Mr. Moody collected a field blank sample, identified as sample "R16", at 12:52 pm. We secured the samples in iced coolers in our locked vehicle.

III. D. Closing Conference

The group returned to the Administration Building conference room and I initiated the closing conference at 1:05 pm. Ms. Bartovich provided a flash drive with copies of requested documents. I copied the files to my laptop, and again confirmed that the files did not contain CBI. I asked the USS representatives which NPDES permit is currently being implemented. Mr. Moe said "the 2010 Permit modification." Ms. Bartovich agreed. I asked if USS has a storm water pollution prevention plan (SWPPP). Mr. Moe said they do and are following requirements of MPCA's general storm water permit. I asked if any of USS' previous permits for the Tailings Basin included storm water requirements. Mr. Moe confirmed that previous permits did not contain storm water requirements. Mr. Moe added "with the exception of rain on outer perimeter dike, all runoff goes into the basin." I asked for a copy of the SWPPP covering the Tailings Basin. Ms. Bartovich provided the SWPPP documents on a flash drive and I copied the files to my laptop.

I provided Mr. Moe with a list of documents received and a sample receipt (Appendix D), and asked him to make copies. Ms. Bartovich signed the document receipt. I asked the USS representatives if they had any questions. Ms. Bartovich requested a copy of EPA's last inspection. I said that I was not involved

with the last inspection and would pass along the request to EPA management. I concluded the closing conference and Mr. Moody and I departed the facility at 2:35 pm.

III. E. Sample Handling Information

All samples were tagged, placed in a cooler and packed with ice. Custody was maintained during the inspection by locking the samples in an EPA vehicle. Mr. Moody retained the vehicle keys. After being sealed in a bag in the coolers, Mr. Moody and I delivered the samples to the Chicago Regional Lab on September 14, 2017. Copies of the sample chain-of-custody forms are included in Appendix E.

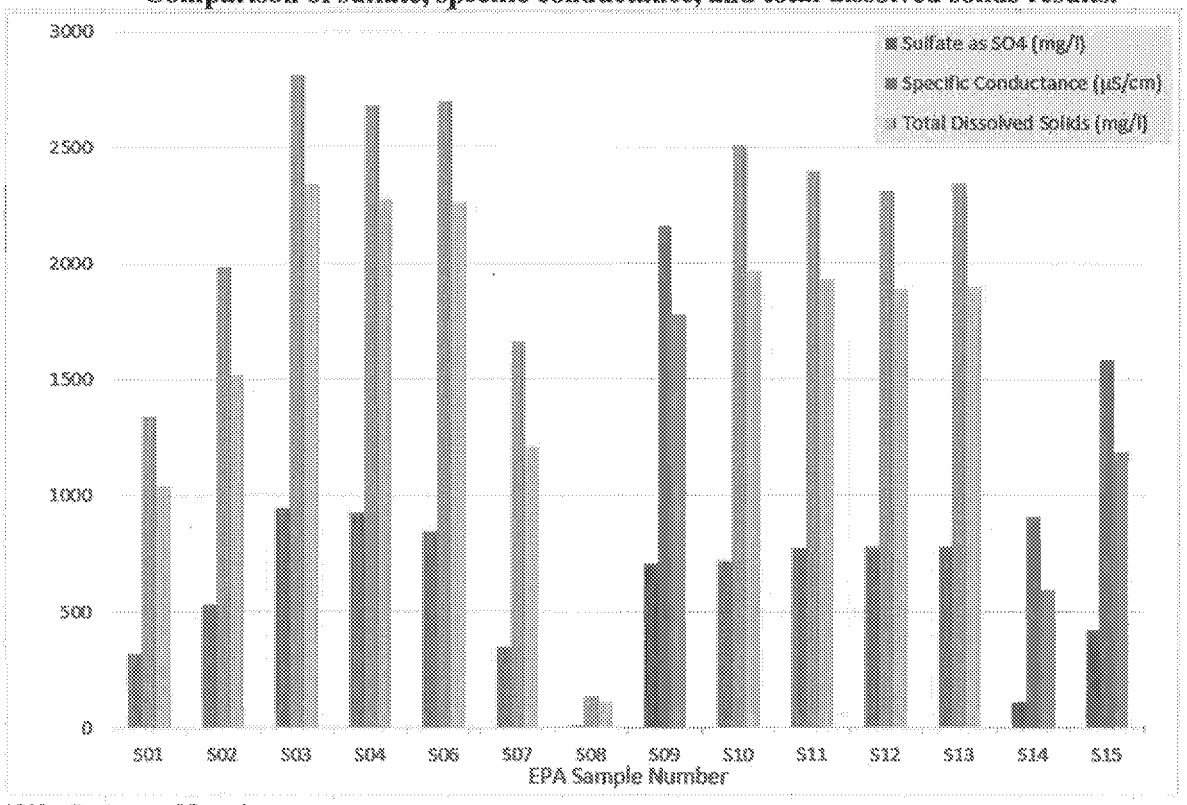
IV. DOCUMENTS RECEIVED

The list of documents received during the inspection is included in Appendix D "Inspection Documents Received by EPA."

V. COMPLIANCE SAMPLING INSPECTION RESULTS

The results of EPA's compliance sampling inspection are summarized in Tables 2-4, below, and in Figure 12, which includes results for a subset of parameters. The laboratory reports are provided in Appendix F. The graph below compares results of sulfate, specific conductance, and total dissolved solids.

Comparison of sulfate, specific conductance, and total dissolved solids results.



***S08 = Background Location**

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Table 2: Sample Results - Metals

		EPA Sample Number															
		S01	S02	S03	S04	D05*	S06	S07	S08	S09	S10	S11	S12	S13	S14	S15	R16
Parameter		09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/13/17	09/13/17	09/13/17	09/13/17	09/13/17	09/13/17
Total Metals EPA 200.8																	
Antimony	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Arsenic	ug/L	2.64	1.11	U	U	U	U	1.03	U	14.0	U	1.52	2.12	1.89	1.21	1.13	U
Barium	ug/L	31.8	19.7	18.2	40.0	40.4	22.3	U	22.9	125.0	10.8	27.2	80.6	37.9	10.7	33.7	U
Beryllium	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Cadmium	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Chromium	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Cobalt	ug/L	U	U	4.21	1.26	1.23	5.31	U	0.64	3.08	0.53	0.75	3.01	2.41	U	U	U
Copper	ug/L	U	U	U	U	U	U	U	2.05	U	U	U	U	U	U	U	U
Lead	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Manganese	ug/L	711	20.2	2340	1530	1540	1130	117	217	9980	330	522	1980	2160	76.2	47.4	U
Molybdenum	ug/L	U	1.17	2.05	3.87	3.93	U	U	U	U	U	7.52	16.1	9.45	U	U	U
Nickel	ug/L	U	1.15	3.95	2.51	2.47	3.45	U	2.32	2.59	2.37	2.16	2.18	2.24	U	U	U
Selenium	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Silver	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Thallium	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Zinc	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Total Metals EPA 200.7																	
Aluminum	mg/L	U	U	U	U	U	U	U	U	0.431	U	U	U	U	U	U	U
Iron	mg/L	0.917	U	0.188	0.141	0.101	0.212	0.382	1.76	35.1	U	0.759	6.25	1.7	0.425	0.187	U
Lithium	mg/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Magnesium	mg/L	156	233	289	277	279	279	228	6.95	188	216	230	232	225	110	201	U
Phosphorus	mg/L	U	U	U	U	U	U	U	U	0.277	U	U	U	U	U	U	U
Potassium	mg/L	4.97	11.8	16.6	16.6	16.6	12.4	4.38	1.58	5.79	7.07	20.1	19.3	18.9	5.93	9.33	U
Sodium	mg/L	19.3	27.3	70.2	68.7	69.2	59.1	10.8	3.02	72.1	60.4	70.8	71.6	68.8	9.7	21.6	U
Tin	mg/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Titanium	mg/L	U	U	U	U	U	U	U	U	0.006	U	U	U	U	U	U	U
Vanadium	mg/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U
Boron	mg/L	0.142	0.222	0.318	0.244	0.243	0.297	0.261	U	0.129	0.193	0.232	0.186	0.23	0.0938	0.163	U
Calcium	mg/L	63	86.5	161	146	147	163	47.1	12.8	174	175	136	122	130	30.9	61.1	U
Mercury (EPA 245.1)	ug/L	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U	U

Notes:
U - Not detected
* Duplicate sample (S04)

Table 3: Sample Results - Field measurements, water quality and conventional parameters

		EPA Sample Number															
		S01	S02	S03	S04	D05*	S06	S07	S08	S09	S10	S11	S12	S13	S14	S15	R16
Parameter		09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/12/17	09/13/17	09/13/17	09/13/17	09/13/17	09/13/17	09/13/17
Field Measurements																	
Sp. Conductance	uS/cm	1342	1985	2815	2683		2699	1661	134	2163	2509	2399	2316	2348	907	1583	NR
pH	S.U.	7.27	8.02	7.03	7.16		6.77	8.23	7.19	7.24	7.08	7.83	7.13	7.51	8.34	8.18	NR
Dissolved Oxygen	mg/l	0.73	10.58	1.14	4.19		1.19	8.2	7.11	NR	3.64	12.53	1.47	6.62	9.01	8.76	NR
Temperature	°C	17.6	13.07	7.55	8.4		9.71	18.01	19.11	17.47	19.17	17.91	11.19	17.54	18.03	18.96	NR
Water Quality																	
Hardness	mg CaCO3/L	799	1170	1590	1510	1510	1560	1060	60	1210	1330	1290	1260	1250	530	981	U
Alkalinity	mg CaCO3/L	480	650	610	540	550	700	710	55	380	580	470	390	470	430	600	U
Fluoride	mg/L	0.3	0.28	1.08	1.44 L	1.32	1	0.28	0.1	0.55	0.64	1.97 L	1.64	1.78	0.13	0.18	U
Chloride	mg/L	28.1	25.3	96	102	102	87.8	11.7	1.28	122	91.2	114	114	110	5.24	18.8	U
Sulfate as SO4	mg/L	314	532	945	925	950	847	343	3.61	702	720	773	780	781	107	418	0.12 J
Bromide	mg/L	U	U	1.18	0.67	0.63	0.48	U	U	0.73	0.57	0.68 J	0.63	0.51	U	U	U
Conventionals																	
TDS	mg/L	1040 J	1520 J	2350 J	2280 J	2300 J	2270 J	1210 J	104 J	1780 J	1970 J	1930	1890	1900	594	1190	U
TSS	mg/L	6 J	U	10	U	12 J	U	U	13 J	206 J	U	U	12	18	6	U	U

Notes:

J - The identification of the analyte is acceptable; the reported value is an estimate.

U - Not detected

NR - No reading

L - The identification of the analyte is acceptable; the reported value may be biased low. The actual value is expected to be greater than the reported value.

* Duplicate sample (S04)

Table 4: Sample Results - Field measurement stations

		DRP Swale	CB12 in	CB11 out	CB11 in
Parameter		09/13/17	09/12/17	09/12/17	09/12/17
Field Measurements					
Sp. Conductance	uS/cm	908	2387	877	2518
pH	S.U.	8.29	7.23	7.71	8.16
Dissolved Oxygen	mg/l	7.85	7.54	9.64	7.79
Temperature	°C	17.76	21.8	20.1	26.06

VI. REFERENCES

Hatch (2014). *Minntac Western Seepage Collection System – Phase 2 Report.* United States Steel Corporation, Mountain Iron, Minnesota. March 2014.

MPCA (1987). NPDES/SDS Permit MN0057207. U.S. Steel Minntac Tailings Basin. Minnesota. September 30, 1987.

MPCA (1989). NPDES/SDS Permit MN0057207, Permit Modification. U.S. Steel Minntac Tailings Basin. Minnesota. February 23, 1989.

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Appendix A: Figures

ED_004744_00033652-00018

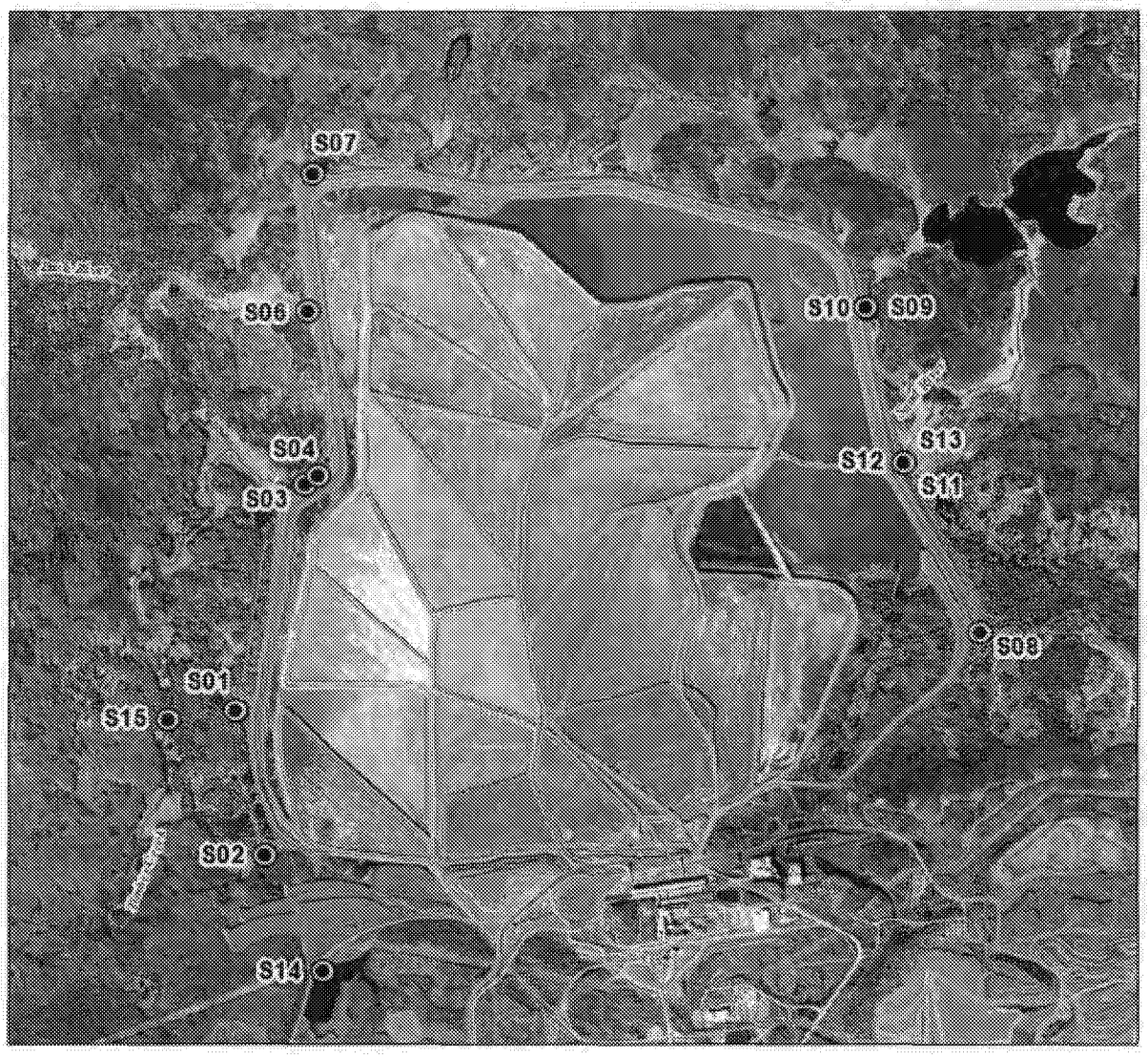
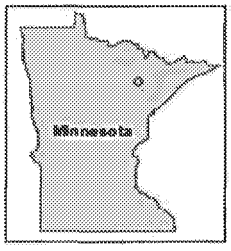


Figure 1. EPA Sampling Station Locations



EPA Sampling Location

N

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0 1 2 4 Miles

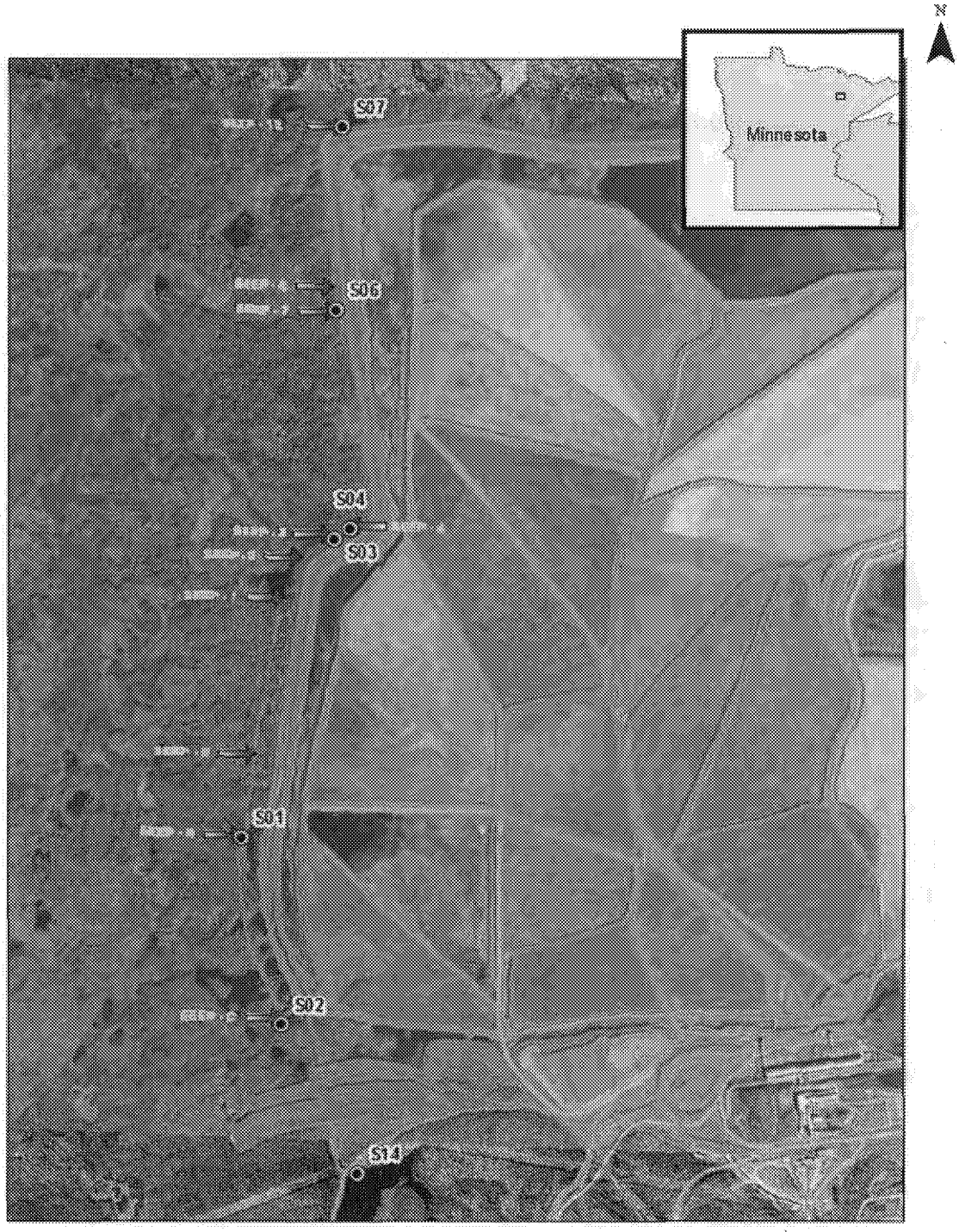


Figure 2. US Steel Western Seepage Locations (Hatch, 2014).
(annotated to show EPA samping locations)

◉ EPA Sampling Station

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

1 0.5 0 1 Miles

Figure 3. Dark River Pond

EPA Sampling Station

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

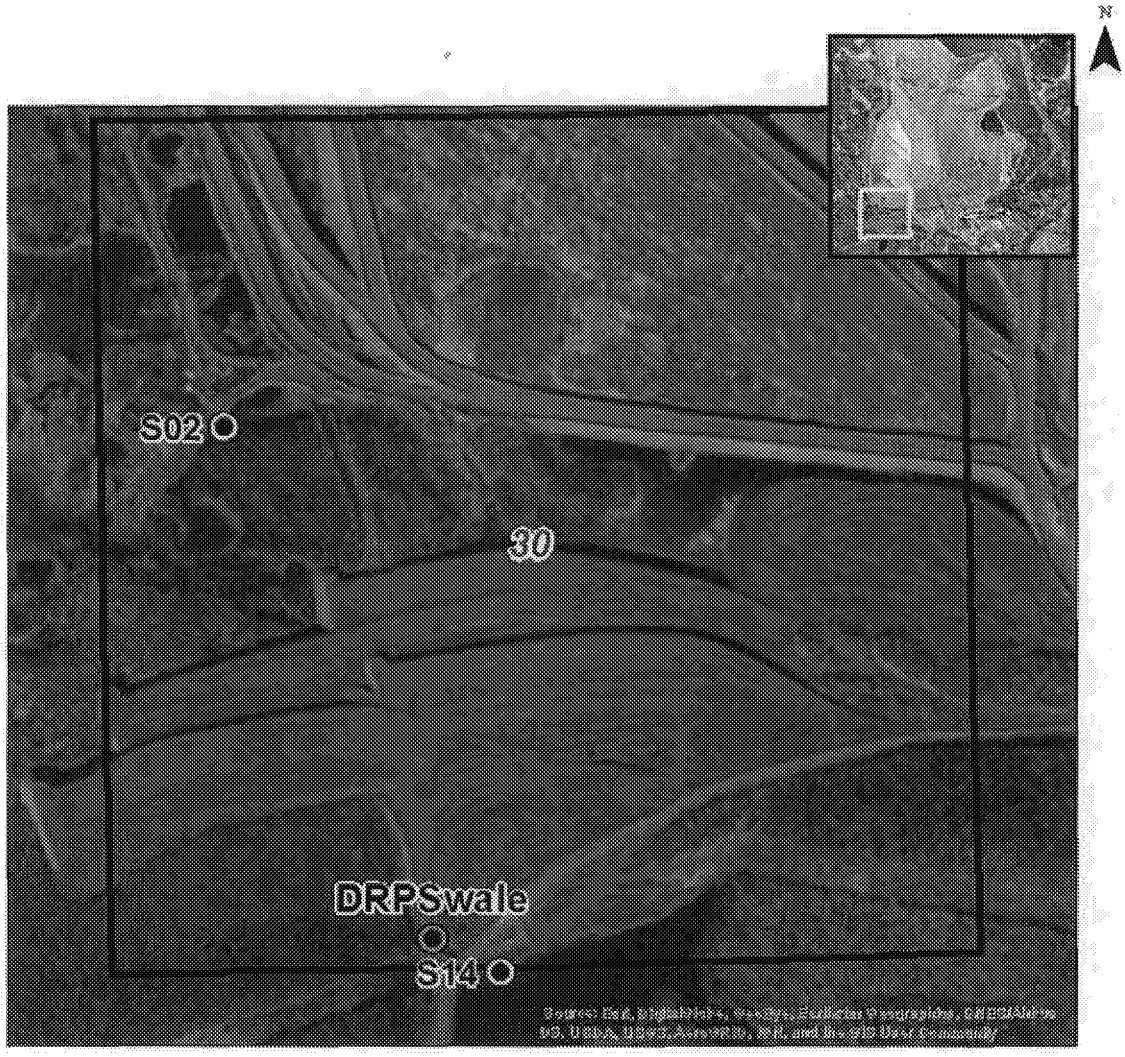


Figure 4. Sampling Locations in Township 59N, Range 18W, Section 30

Field Parameter Measurement Location

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

Figure 5. Sampling Locations in Township 59N, Range 18W, Section 19 and 24

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

Figure 6. Sampling Locations in Township 59N, Range 18W, Section 18

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

Figure 7. Sampling Locations in Township 59N, Range 18W, Section 7

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

Figure 8. Sampling Locations in Township 59N, Range 18W, Section 6

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.5 0.25 0 0.5 Miles

Figure 9. Sampling Locations in Township 59N, Range 18W, Section 10

Field Parameter Measurement Location

EPA Sampling Location

Sheetpile

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

0.1 0.05 0 0.1 Miles

Figure 10. Sampling Locations in Township 59N, Range 18W, Section 15

EPA Sampling Location

Sheetpile

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

250 125 0 250 Feet

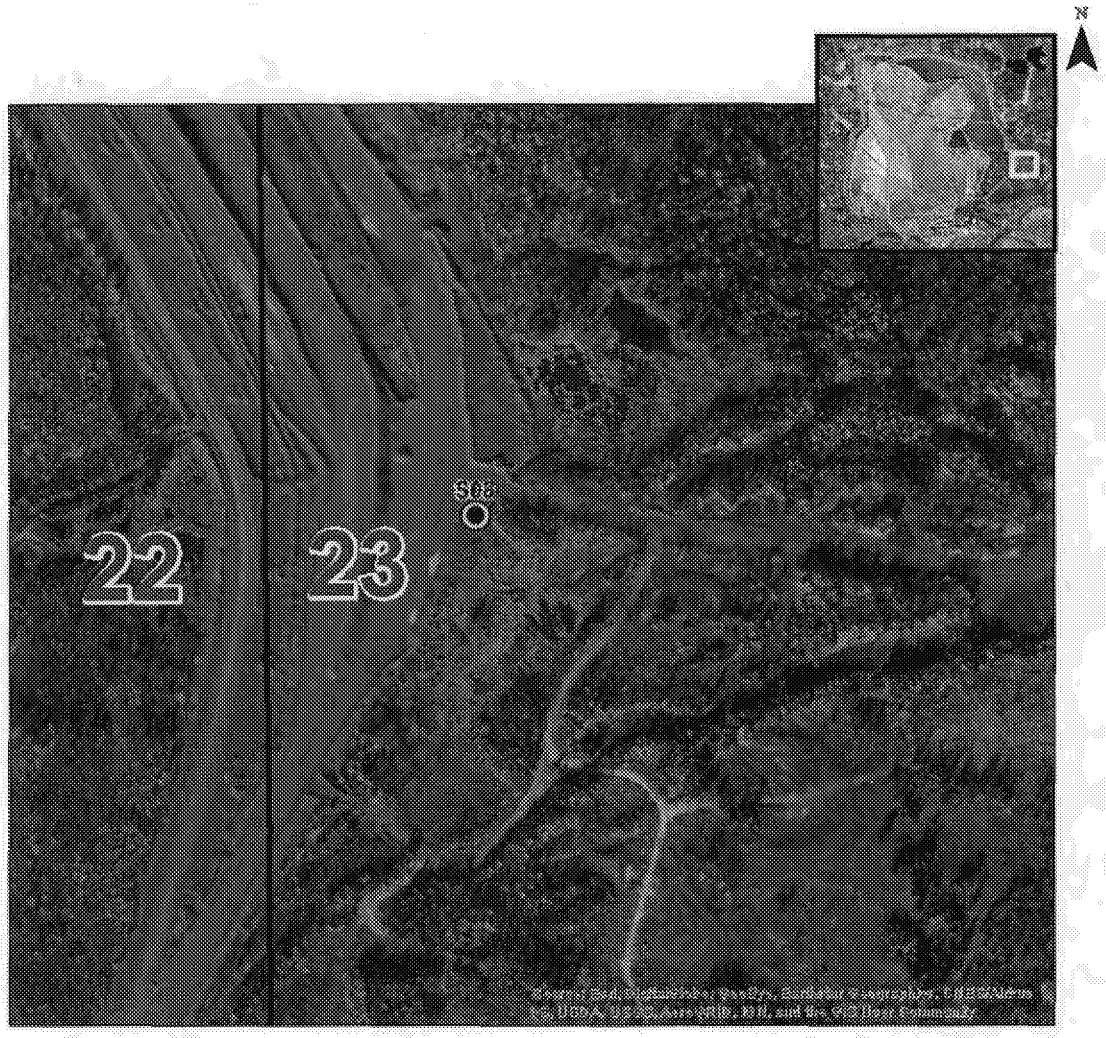


Figure 11. Sampling Locations in Township 59N, Range 18W, Section 23

EPA Sampling Location

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

1,000 500 0 1,000 Feet

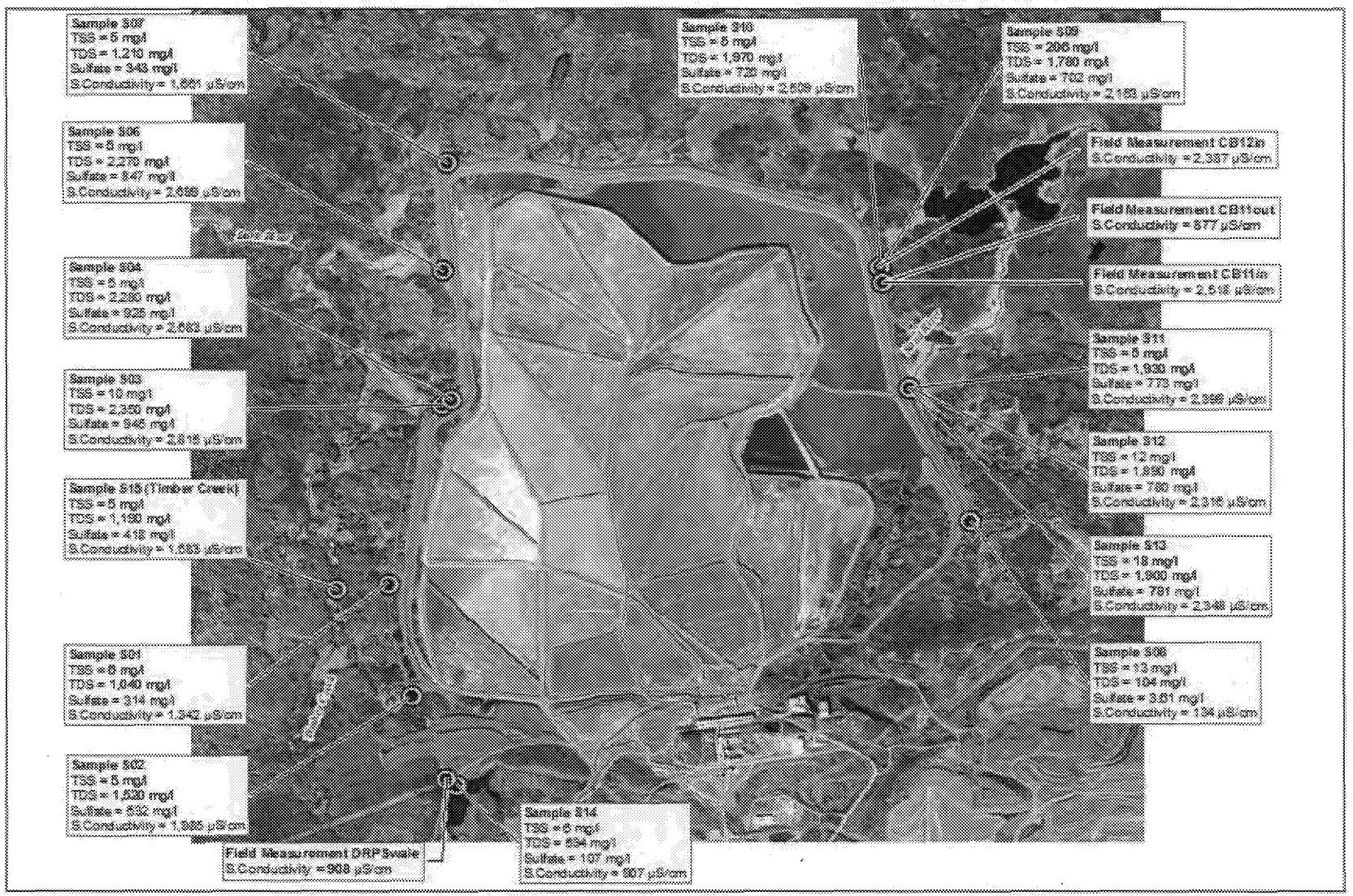


Figure 12. EPA Sample Results (TDS/TSS, Sulfate, Specific Conductivity)

EPA Sample Locations

N

0 0.5 1 2 3 4 Miles

Clean Water Act Inspection Report
U.S. Steel – Minntac Tailings Basin Area Facility

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Appendix B: Photo Log

ED_004744_00033652-00032

U.S. Steel - Minntac Tailings Basin Area Facility
EPA Inspection September 12-13, 2017
All photos taken by Dean Maraldo, Inspector, U.S. EPA
Camera: Ricoh WG-4

1: USSM0252
Description: Buffers and standards used to calibrate YSI 556 MPS meter.
Location: Administration Building parking lot.
Camera Direction: 126°
Date/Time: September 12, 2017/ 9:18 am

ED_004744_00033652-00033

2: USSM0253
Description: EPA Sample Location S01 (Seep "A", Hatch, 2014).
Location: Township 59N, Range 18W, Sections 19 and 24; 47.57985 N; 92.68961 W
Camera Direction: 345°
Date/Time: September 12, 2017/ 9:52 am

ED_004744_00033652-00034

3: USSM0255
Description: EPA Sample Location S01 sample bottles.
Location: Township 59N, Range 18W, Sections 19 and 24
Camera Direction: 116°
Date/Time: September 12, 2017/ 10:01 am

ED_004744_00033652-00035

4: USSM0256
Description: EPA Sample Location S02 (Seep "C", Hatch, 2014); also referred to by USS as Dark River Pond Seep.
Location: Township 59N, Range 18W, Section 30; 47.56834 N; 92.68606 W
Camera Direction: 141°
Date/Time: September 12, 2017/ 10:21 am

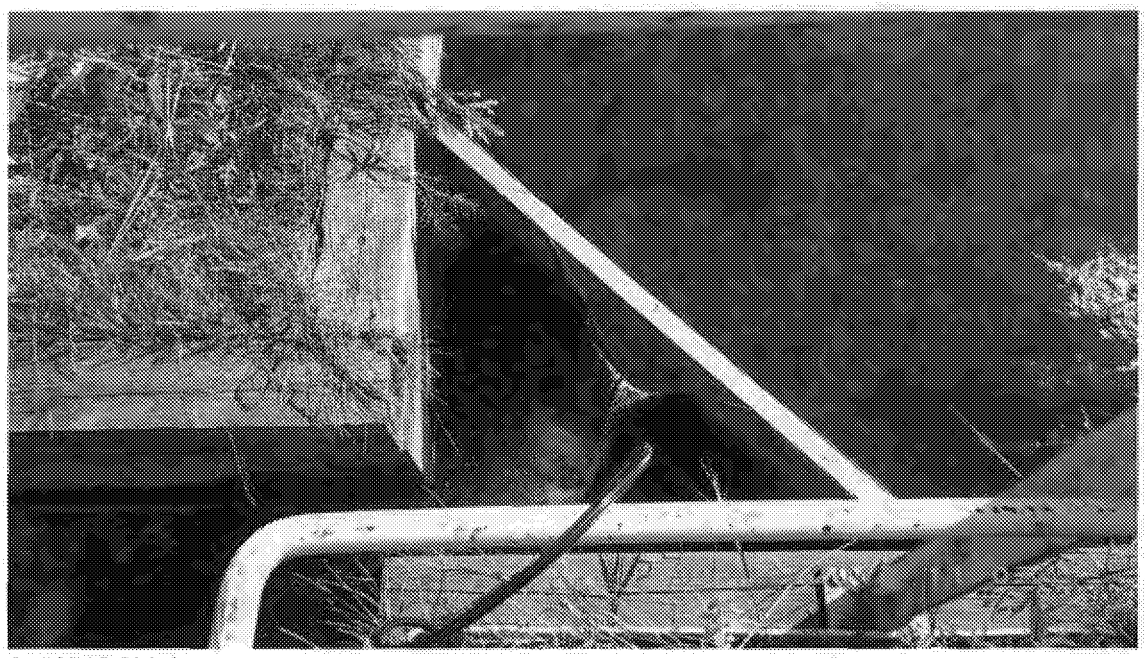
5: USSM0257
Description: EPA Sample Location S02 sample bottles.
Location: Township 59N, Range 18W, Section 30
Camera Direction: 165°
Date/Time: September 12, 2017/ 10:24 am

ED_004744_00033652-00036

6: USSM0258
Description: EPA Sample Location S03 (Seep "3", Hatch, 2014).
Location: Township 59N, Range 18W, Section 18; 47.59813 N; 92.68136 W
Camera Direction: 229°
Date/Time: September 12, 2017/ 10:46 am

7: USSM0259
Description: EPA Sample Location S03 sample bottles.
Location: Township 59N, Range 18W, Section 18
Camera Direction: 245°
Date/Time: September 12, 2017/ 10:53 am

ED_004744_00033652-00037



8: USSM0260
Description: EPA Sample Location S04 (Outfall SD001 and location of Seep "4", Hatch, 2014).
Location: Township 59N, Range 18W, Section 18; 47.59878 N; 92.67989 W
Camera Direction: 81°
Date/Time: September 12, 2017/ 11:07 am

9: USSM0261
Description: EPA Sample Location S04 sample bottles.
Location: Township 59N, Range 18W, Section 18
Camera Direction: 73°
Date/Time: September 12, 2017/ 11:11 am

ED_004744_00033652-00038

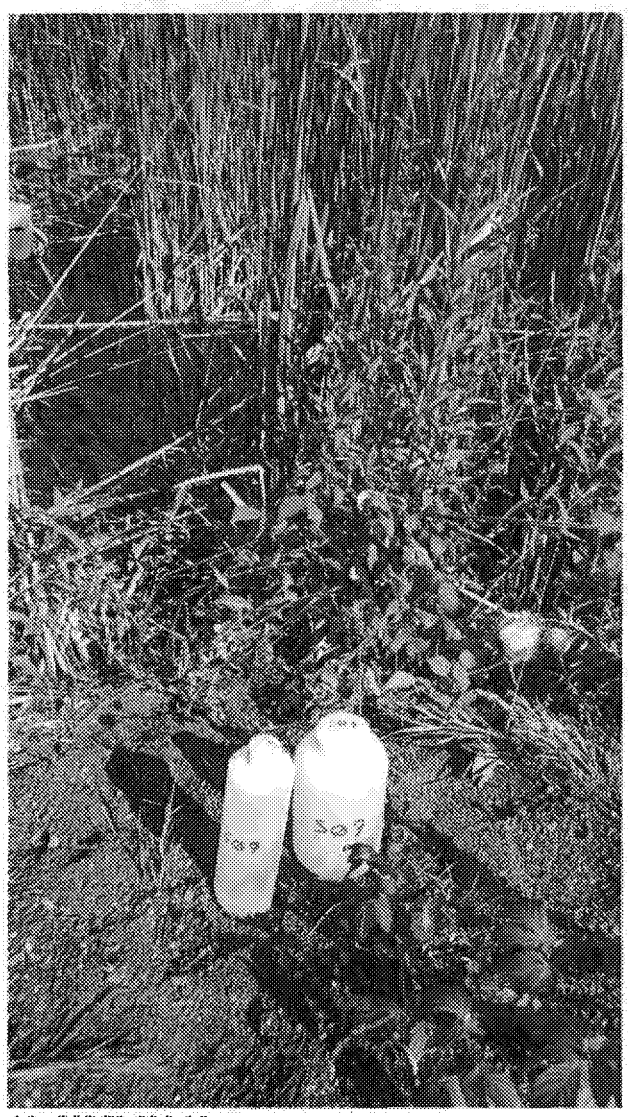
10: USSM0262
Description: EPA Sample Location S06 (Seep "7", Hatch, 2014).
Location: Township 59N, Range 18W, Section 7; 47.61200 N; 92.68116 W
Camera Direction: 287°
Date/Time: September 12, 2017/ 11:26 am

11: USSM0263
Description: EPA Sample Location S06 sample bottles.
Location: Township 59N, Range 18W, Section 7
Camera Direction: 271°
Date/Time: September 12, 2017/ 11:31 am

ED_004744_00033652-00039

12: USSM0264
Description: EPA Sample Location S07 (Seep "13", Hatch, 2014).
Location: Township 59N, Range 18W, Section 6; 47.62307 N; 92.68063 W
Camera Direction: 26°
Date/Time: September 12, 2017/ 11:46 am

ED_004744_00033652-00040



13: USSM0265
Description: EPA Sample Location S07 sample bottles.
Location: Township 59N, Range 18W, Section 6
Camera Direction: 12°
Date/Time: September 12, 2017/ 11:52 am

ED_004744_00033652-00041

14: USSM0266
Description: EPA Sample Location S08 (background area wetland).
Location: Township 59N, Range 18W, Section 23; 47.58625 N; 92.60082 W
Camera Direction: 43°
Date/Time: September 12, 2017/ 1:58 pm

15: USSM0267
Description: EPA Sample Location S08 sample bottles.
Location: Township 59N, Range 18W, Section 23
Camera Direction: 76°
Date/Time: September 12, 2017/ 2:04 pm

ED_004744_00033652-00042

16: USSM0268
Description: EPA Sample Location S09 sample bottles.
Location: Township 59N, Range 18W, Section 10
Camera Direction: 96°
Date/Time: September 12, 2017/ 3:03 pm

17: USSM0269
Description: EPA Sample Location S09. Wetland immediately outside of sheet pile at SC&R Catch Basin-12.
Location: Township 59N, Range 18W, Section 10; 47.61222 N; 92.61440 W
Camera Direction: 77°
Date/Time: September 12, 2017/ 3:04 pm

ED_004744_00033652-00043

18: USSM0270
Description: EPA Sample Location S09 – SC&R Catch Basin-12. Notice sheet pile cut low.
Location: Township 59N, Range 18W, Section 10
Camera Direction: 78°
Date/Time: September 12, 2017/ 3:05 pm

19: USSM0271
Description: EPA Sample Location S09. Another view of sheet pile cut low.
Location: Township 59N, Range 18W, Section 10
Camera Direction: 317°
Date/Time: September 12, 2017/ 3:05 pm

ED_004744_00033652-00044

20: USSM0272
Description: EPA Sample Location S10. Sampled seepage collected in SC&R Catch Basin-12.
Location: Township 59N, Range 18W, Section 10; 47.61234 N; 92.61449 W
Camera Direction: 20°
Date/Time: September 12, 2017/ 3:13 pm

21: USSM0273
Description: EPA Sample Location S10 sample bottles.
Location: Township 59N, Range 18W, Section 10
Camera Direction: 51°
Date/Time: September 12, 2017/ 3:21 pm

ED_004744_00033652-00045

22: USSM0274
Description: Sheet pile at Catch Basin -11. Noted water staining at level of sheet pile handling holes.
Location: Township 59N, Range 18W, Section 10
Camera Direction: 22°
Date/Time: September 12, 2017/ 3:43 pm

23: USSM0275
Description: Sheet pile at SC&R Catch Basin-5.
Location: Township 59N, Range 18W, Section 15; 47.59992 N; 92.60999 W
Camera Direction: 4°
Date/Time: September 12, 2017/ 4:03 pm

ED_004744_00033652-00046

24: USSM0276
Description: Seepage collected in SC&R Catch Basin-5.
Location: Township 59N, Range 18W, Section 15
Camera Direction: 198°
Date/Time: September 12, 2017/ 4:04 pm

25: USSM0277
Description: Sheet pile, SC&R Catch Basin-5
Location: Township 59N, Range 18W, Section 15
Camera Direction: 40°
Date/Time: September 12, 2017/ 4:05 pm

ED_004744_00033652-00047

26: USSM0278
Description: EPA Sample Location S11. Wetland immediately outside of sheet pile at SC&R Catch Basin-5.
Location: Township 59N, Range 18W, Section 15; 47.59992 N; 92.60999 W
Camera Direction: 11°
Date/Time: September 13, 2017/ 9:45 am

27: USSM0279
Description: EPA Sample Location S11 sample bottles.
Location: Township 59N, Range 18W, Section 15
Camera Direction: 14°
Date/Time: September 13, 2017/ 9:45 am

ED_004744_00033652-00048

28: USSM0280
Description: EPA Sample Location S12. Sampled seepage collected in SC&R Catch Basin-5, near inlet.
Location: Township 59N, Range 18W, Section 15; 47.59991 N; 92.60999 W
Camera Direction: 231°
Date/Time: September 13, 2017/ 9:47 am

29: USSM0281
Description: EPA Sample Location S12 sample bottles.
Location: Township 59N, Range 18W, Section 15
Camera Direction: 230°
Date/Time: September 13, 2017/ 9:51 am

ED_004744_00033652-00049

30: USSM0282
Description: EPA Sample Location S13. Wetland immediately outside of north end of sheet pile at SC&R Catch Basin-5.
Location: Township 59N, Range 18W, Section 15; 47.59993 N; 92.61001 W
Camera Direction: 14°
Date/Time: September 13, 2017/ 9:52 am

31: USSM0283
Description: EPA Sample Location S13 sample bottles.
Location: Township 59N, Range 18W, Section 15
Camera Direction: 22°
Date/Time: September 13, 2017/ 9:57 am

ED_004744_00033652-00050

32: USSM0284
Description: EPA Sample Location S14. Sampled at base of coarse tailing berm at edge of Dark River Pond.
Location: Dark River Pond;Township 59N, Range 18W, Section 30; 47.55915 N; 92.67916 W
Camera Direction: 211°
Date/Time: September 13, 2017/ 10:21 am

33: USSM0285
Description: EPA Sample Location S14 sample bottles.
Location: Dark River Pond;Township 59N, Range 18W, Section 30
Camera Direction: 196°
Date/Time: September 13, 2017/ 10:24 am

ED_004744_00033652-00051

34: USSM0286
Description: View of Dark River Pond.
Location: Dark River Pond;Township 59N, Range 18W, Section 30
Camera Direction: 186°
Date/Time: September 13, 2017/ 10:26 am

35: USSM0287
Description: View of Dark River Pond Swale, just downgradient of Dark River Pond.
Location: Dark River Pond Swale; Township 59N, Range 18W, Section 30; 47.559722 N; 92.680833 W
Camera Direction: 173°
Date/Time: September 13, 2017/ 10:32 am

ED_004744_00033652-00052

36: USSM0288
Description: EPA Sample Location S15. Sampled flowing water in Timber Creek, at mid-stream.
Location: Timber Creek; Township 59N, Range 18W, Section 24; 47.56624 N; 92.67951 W
Camera Direction: 280°
Date/Time: September 13, 2017/ 11:07 am

37: USSM0289
Description: EPA Sample Location S15 sample bottles.
Location: Timber Creek; Township 59N, Range 18W, Section 24
Camera Direction: 271°
Date/Time: September 13, 2017/ 11:11 am

ED_004744_00033652-00053

38: USSM0290
Description: EPA Sample Location S15 sample bottles, with sampling pole and glass collection jar.
Location: Timber Creek; Township 59N, Range 18W, Section 24
Camera Direction: 273°
Date/Time: September 13, 2017/ 11:16 am

39: USSM0291
Description: EPA Sample Location R16 sample bottles. Field blank.
Location: Administration Building parking lot.
Camera Direction: 251°
Date/Time: September 13, 2017/ 12:54 pm

ED_004744_00033652-00054

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ED_004744_00033652-00055

Appendix C: Quality Assurance Project Plan/Sampling Plan

ED_004744_00033652-00056

U.S. EPA, REGION 5
QUALITY ASSURANCE PROJECT PLAN/SAMPLING PLAN
FOR ENVIRONMENTAL COMPLIANCE ASSESSMENT

PREPARED BY: _____ 8/25/17
Dean Maraldo, Inspector — Date
WECAB, U.S. EPA, Region 5

APPROVED BY: _____ 8/31/17
Ken Gunter, QA/QC Sampling Manager — Date
WECAB, U.S. EPA, Region 5

APPROVED BY: _____ 8/31/17
Ryan Bahr, Section 2 Chief — Date
WECAB, U.S. EPA, Region 5

Site Name & Location: U.S. Steel – Minntac Tailings Basin Area Facility
County Road 102
Mountain Iron, MN 55768

Sampling Dates: September 12-14, 2017

Introduction

U.S. Environmental Protection Agency Region 5 plans to conduct an announced compliance sampling inspection (CSI) at the U.S. Steel – Minntac Tailings Basin Area Facility located near Mountain Iron, Minnesota (see Figure 1). EPA will collect samples to determine the presence of specific pollutants and to document compliance with the Clean Water Act (CWA) and applicable regulations.

Site Description
The Minntac Tailings Basin is a part of a U.S. Steel (USS) mining operation in St. Louis County, Minnesota. The facility was constructed in the 1960's and was issued an NPDES/State Disposal System permit authorizing discharges from the Tailings Basin to the Sand and Dark rivers in 1987. The permit expired in 1992. The Tailings Basin sits on a divide between two major watersheds. When the basin was constructed the headwaters of both watersheds were filled in with mine tailings. The principal activity at this facility is taconite processing. At the maximum operating rate, the facility will generate 15 million long tons of taconite pellets per year.

Project/Sampling Objectives
The objectives of the EPA sampling event are to 1) characterize wastewater discharges from the Tailings Basin through seeps along the basin berms and into surface waters in the Sand River, Dark River, and Timber Creek watersheds; and 2) to determine compliance with USS's NPDES permit. Sample results will be used to document and characterize the wastewater pollutant discharges to the watersheds. USS identified ten surface seepage locations in the report *Minntac Western Seepage Collection System Phase 2 Report* (Hatch, March 11, 2014; see Table 4 below). EPA will evaluate these seeps as potential sampling locations along with other areas along the basin perimeter. EPA will offer to split samples with facility personnel.

During the inspection, EPA will collect grab samples from USS's wastewater seep outfall #SD001, and other berm seeps along the western and northern sides of the Tailings Basin, as discussed above (see Figure 1 for location of outfall #SD001, and Figure 2 for location of known western Basin seeps). EPA may collect stormwater grab samples from permitted stormwater outfalls and/or any observed discharges to surface water through conveyances other than the permitted outfalls (e.g., pipes, ditches, culverts).

Table 1 contains the sampling location, the anticipated analytical parameters of concern, and the types of samples EPA personnel will collect.

Grab samples will correspond with the sampling techniques outlined in 40 C.F.R. §136. Samples will be collected from seepage pools or directly from flowing seeps when visible and above seepage pool water level. However, depending on the information gathered during the on-site evaluation, EPA personnel may modify the sampling approach to assure the collection of representative samples. For example, if a visible seep if flowing along the surface of the berm wall, EPA may dig out some soil below the seep discharge to allow for collection of free flowing seep water into the sample jar. EPA will collect field parameters including pH, temperature, specific conductance (conductivity), and dissolved oxygen at each sample location. EPA personnel will ship samples to the Region 5 Central Regional Laboratory (CRL) in Chicago, IL with a chain of custody (COC).

<u>Sample Equipment and Procedures:</u>
EPA will collect grab samples directly from outfalls, seeps, or seep collection areas. EPA will use best judgement in collecting any stormwater discharges to surface water through conveyances other than outfalls (e.g., pipes, ditches, culverts). EPA will collect samples for field analysis in a 1-L glass jar and analyze within 15 minutes of collection.

ED_004744_00033652-00058

EPA will collect one field blank using DI water obtained from the Central Regional Laboratory or from EPA's Cleveland Office, to be analyzed for the parameters listed in Table 2. EPA will pour DI water into an unused and clean grab sample collection bottle, (where used for that individual analyses), agitate it, and then pour into the field blank sample bottles. Blank bottles will be prepared and preserved for each analysis listed in Table 3. EPA will also collect one field duplicate sample from SD 001 or one of the seep discharge locations.

CRL will prepare sample bottles and coolers and ship to the OECA Cleveland Office, care of Jonathan Moody, for arrival by September 6.

Following sampling and preservation, EPA will immediately pack sample bottles in coolers and chill them to less than 6°C. EPA will make best efforts to collect samples on September 12-13, for delivery to CRL by September 14.

Table 3 contains a listing of the parameters to be analyzed, analytical methods, preservatives and containers to be used during this project. The actual number and types of samples collected will be determined by the EPA inspectors on-site during the inspection. EPA Inspectors will contact the laboratory immediately following the inspection and notify the lab of the actual number and types of samples being brought in for analysis.

Climatic Limits on Sampling
Adverse weather (high winds/ rain/snow) may delay sampling for health and safety concerns.

Decontamination Procedures
New or pre-cleaned dedicated sampling equipment will be used at the sampling site. No onsite decontamination procedures are anticipated for this sampling activity.

Sample Packaging and Transport
EPA will wrap samples in bubble wrap and/or Ziploc bags if needed, and pack them in a cooler with ice for transport. EPA will place a Chain of Custody form inside a Ziploc bag, taped to the top inside lid of the sample cooler. EPA will tape each sample cooler shut and place custody seals on all 4 corners of the cooler. Alternatively, EPA may place the samples in a plastic garbage bag inside the cooler, seal the garbage bag with tape, and place a custody seal around the tape. EPA inspectors will deliver the samples to CRL.

Safety Procedures
Safety procedures in the Region 5 Health and Safety Manual - R 1440 (May 2000 edition) will be followed during the inspection. Once on-site, USS's safety and health policies will also be discussed and followed where applicable. If there is a conflict between EPA's safety plan and the facility's safety plan, EPA inspectors will coordinate with EPA's Safety Office and decide which procedure provides greater protection for the inspection team members.

Sampling Plan Modification
This sampling plan is subject to modification depending upon conditions encountered in the field and other circumstances.

ED_004744_00033652-00059

Assessments
EPA inspectors will conduct an internal assessment of the sampling plan upon completion of initial on-site surveillance. If sampling and/or laboratory issues are identified, EPA inspectors will coordinate with CRL and/or Region 5 chemists to develop timely corrective actions. Any changes to the sampling plan will be recorded in the inspectors' field notebooks, and included in the inspection report submitted to EPA management.

Contact Information for Use during this Sampling Project

EPA Field Personnel:

Dean Maraldo	312-353-2098 (office)
Jonathan Moody	440-250-1708 (office)

CRL Lab Personnel:

Robert Thompson	312-353-9078 (office)
Robert Snyder	312-353-9083 (office)

Attachments
Table 1 - Sample Location and Sample Type
Table 2 - NPDES Analytical List
Table 3 - Water/ Wastewater Sampling
Table 4 - Western Tailings Basin seepage flow and location coordinates
Figure 1 - USS Minntac Tailings Basin Site with outfall locations
Figure 2 - Western Tailings Basin seepage locations

ED_004744_00033652-00060

Tables

ED_004744_00033652-00061

Table 1 – Sample Location and Sample Type			
Sample Location	Outfall Location	Sample Type (# of samples)	Samples Parameters
USS Minntac NPDES Permit Location SD 001	Seep discharge from western side of Tailings Basin	Grab Samples (1)	**Laboratory:** Total metals[1]; total alkalinity; hardness, bromide; chloride; fluoride; total mercury; TSS/TDS[2]; sulfate. **Field parameters**: pH, dissolved oxygen, specific conductance, and temperature.
Tailings Basin Berm Seep Discharges	Seep discharges from western and northern side of Tailings Basin	Grab Samples (9)	**Laboratory:** Total metals[1]; total alkalinity; hardness, bromide; chloride; fluoride; total mercury; TSS/TDS[2]; sulfate. **Field parameters**: pH, dissolved oxygen, specific conductance, and temperature.
Field Blank	----	Field Blank Sample	**Laboratory:** Total metals[1]; total alkalinity; hardness, bromide; chloride; fluoride; total mercury; TSS/TDS[2]; sulfate. **Field parameters**: pH, dissolved oxygen specific conductance, and temperature.
Field Duplicate	SD 001 or a seep discharge location	Field Duplicate Sample	**Laboratory:** Total metals[1]; total alkalinity; hardness, bromide; chloride; fluoride; total mercury; TSS/TDS[2]; sulfate. **Field parameters**: pH, dissolved oxygen, specific conductance, and temperature.

(1) Total Metals includes Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, P, Se, Ag, Na, Sr, Tl, Sn, Ti, V, Zn.

(2) TSS = Total Suspended Solids; TDS = Total Dissolved Solids

ED_004744_00033652-00062

Table 2 - Minntac NPDES Analytical List	
Method	**Parameters/Reporting Limit**
Total Metals / EPA 200.7	Total Metals reported to CRL RL Al 200 ug/l Ba 10 ug/l B 50 ug/l Ca 0.5 mg/l Fe 80 ug/l Li 25.0 ug/l Mg 0.5 mg/l Na 0.5 mg/l P 0.25 mg/l Sn 20 ug/l Ti 20 ug/l V 10 ug/l
Total Metals / EPA 200.8	Total Metals reported to CRL RL Sb 1.0 ug/l As 1.0 ug/l Be 1.0 ug/l Cd 1.0 ug/l Cr 2.0 ug/l Co 0.5 ug/l Cu 2.0 ug/l Pb 0.5 ug/l Mn, 10 ug/l Mo 12 ug/l Ni 1.0 ug/l Se 2.0 ug/l Ag 1.0 ug/l Tl 0.5 ug/l Zn 10 ug/l
Hardness / SM 2340B	Hardness 1 mg/l $CaCO_3$/L
Total Mercury / EPA 245.1	Mercury, 0.5 ug/l
Alkalinity, as $CaCO_3$ / SM 2320B	Alkalinity, as $CaCO_3$/L, 20 mg/l
Total Filterable Residue (TDS) / SM 2540 C	TDS, 20 mg/l
Total Suspended Solids / SM 2540 D	TSS, 5.0 mg/l
Anions: Bromide, Chloride, Fluoride, Sulfate / EPA 300.0	1.0 mg/l

ED_004744_00033652-00063

Table 3 - Water/ Wastewater Sampling						
Total Number of Samples (1)	**Matrix (2)**	**Container Size/Type (3)**	**Parameter / Analytical Method No.**	**Preservative**	**Holding Time**	**Sample Type**
12	AQ (water)	One 500 ml HDPE bottle for each sample.	Total Metals / EPA 200.7*/200.8	HNO_3 to pH <2	6 months	Grab
12	AQ (water)	Pulled from 500 ml Total metals bottle.	Total Mercury/ EPA 245.1	HNO_3 to pH <2	28 days	Grab
12	AQ (water)	Calculated from Ca and Mg results, no additional bottles needed.	Hardness SM 2340B	HNO_3 to pH <2	6 months	Grab
12	AQ (water)	One 1-L HDPE bottle for each sample.	Alkalinity, as $CaCO_3$ SM 2320B	Cool to ≤ 6°C	14 days	Grab
12	AQ (water)	Pulled from 1-L alkalinity bottle.	Total Filterable Residue (TDS)/ SM 2540 C	Cool to ≤ 6°C	7 days	Grab
12	AQ (water)	Pulled from 1-L alkalinity bottle.	Total Suspended Solids SM 2540 D	Cool to ≤ 6°C	7 days	Grab
12	AQ (water)	Pulled from 1-L alkalinity bottle.	Anions: Bromide, Chloride, Fluoride, Sulfate / EPA 300.0	Cool to ≤ 6°C	28 days	Grab
12	AQ (water)	Field Analysis	Temperature, pH, specific conductance, dissolved oxygen	None	Analyze within 15 minutes.	Grab

(1) This number includes EPA SD001 and seep samples, field blank, and duplicate sample.
(2) AQ=Aqueous
(3) HDPE=High Density Polyethylene
(4) This method has provisions for samples that are to known contain high concentrations of anions to include the use of the Method of Standard Additions (MSA). The NPDES permit fact sheet for this facility includes analyses with generally high levels of TDS, conductance and Hardness (Ca and Mg).

ED_004744_00033652-00064

Table 4 - Western Tailings Basin seepage flow and location coordinates.

Seep Point	Location Coordinates*		Measured Flow (gpm)
A	15,789.611	-16,793.702	57.7
B	17,587.810	-16,554.610	10.8
C	11,704.567	-15,738.228	603.2
1	21,153.456	-16,018.758	27.9
2	22,042.807	-15,679.247	204.1
3	22,570.900	-15,044.560	416.3
4	22,799.087	-14,619.613	98.7
7	27,481.470	-15,129.004	30.7
8	28,040.241	-15,210.393	43.1
13	31,582.326	-15,083.452	159.9

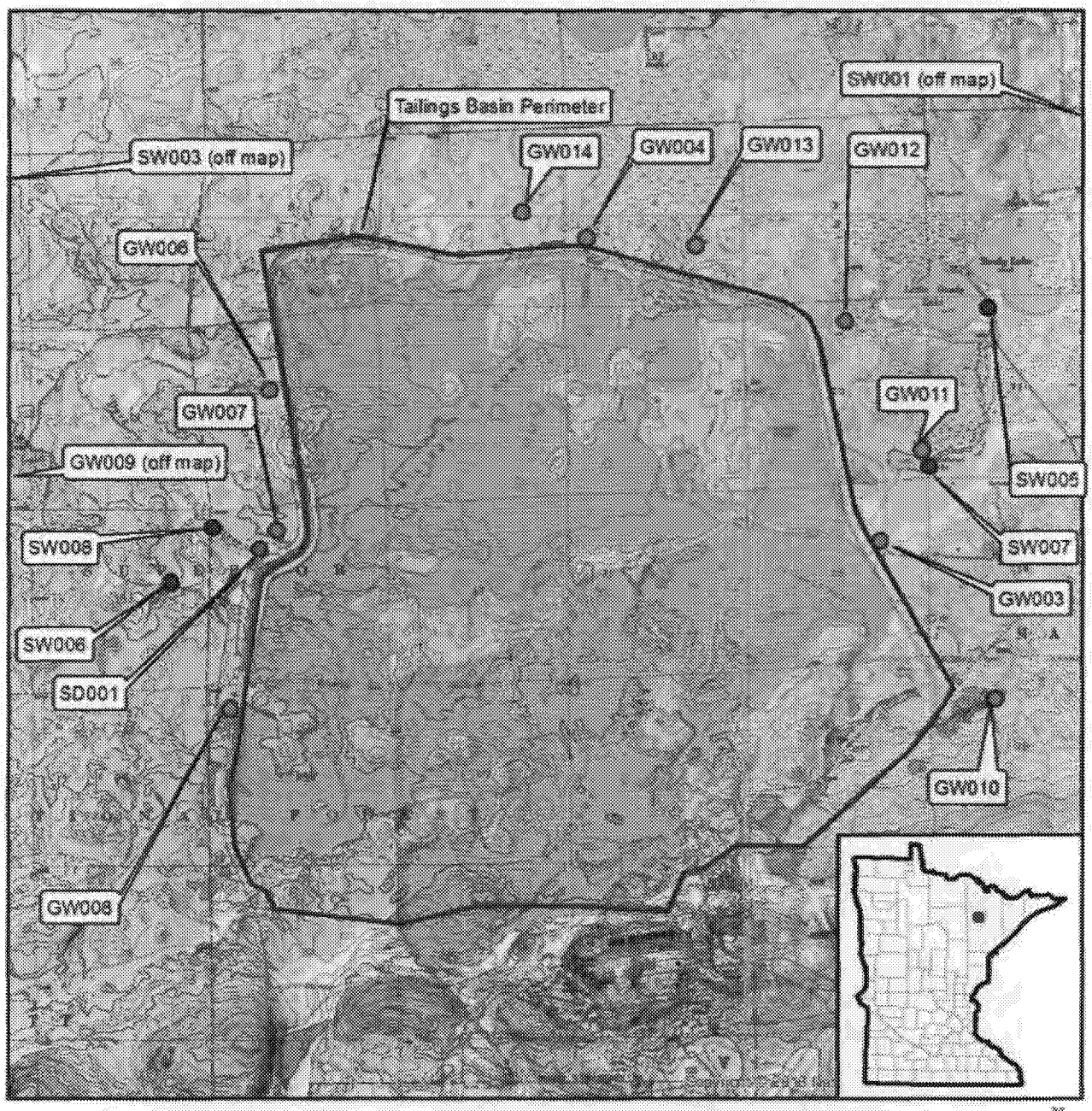
*Coordinates are in local Minntac coordinates system.

Figures

ED_004744_00033652-00066

Topographic Map of Permitted Facility

MN0057207: US Steel Minntac Tailings Basin
T59N, R18W, Sections 3-10, 14-23, 27-30
Mt. Iron, St. Louis County, Minnesota



Map produced by: MPCA Staff, 1/30/2015
Source: USGS Quad
Scale: 1:50,000

0 0.5 1 2 Miles

N

Figure 1: Topographic map of USS Minntac Tailings Basin Site with monitoring locations.

Figure 2: Western Tailings Basin seepage locations.

ED_004744_00033652-00068

U.S. EPA, REGION 5
QUALITY ASSURANCE PROJECT PLAN/SAMPLING PLAN ADDENDUM
FOR ENVIRONMENTAL COMPLIANCE ASSESSMENT

Wadeable Stream Sampling Addendum

PREPARED BY: _____ 9/8/17
Dean Maraldo, Inspector — Date
WECAB, U.S. EPA, Region 5

APPROVED BY: _____ 9/8/17
Ken Gunter, QA/QC Sampling Manager — Date
WECAB, U.S. EPA, Region 5

APPROVED BY: _____ 9/8/17
Ryan Bahr, Section 2 Chief — Date
WECAB, U.S. EPA, Region 5

Site Name & Location: U.S. Steel – Minntac Tailings Basin Area Facility
County Road 102
Mountain Iron, MN 55768

Sampling Dates: September 12-14, 2017

Introduction

U.S. Environmental Protection Agency Region 5 plans to conduct an announced compliance sampling inspection (CSI) at the U.S. Steel – Minntac Tailings Basin Area Facility located near Mountain Iron, Minnesota (see Figure 1). EPA will collect samples to determine the presence of specific pollutants and to document compliance with the Clean Water Act (CWA) and applicable regulations.

Project/Sampling Objectives
The objectives of the EPA sampling event include the characterization of wastewater discharges from the Tailings Basin through seeps along the basin berms and into surface waters in the Sand River, Dark River, and Timber Creek watersheds. EPA may collect surface water grab samples from wadeable receiving water streams. EPA will offer to split samples with facility personnel.

Grab samples will correspond with the sampling techniques outlined in 40 C.F.R. §136. Samples will be collected in accordance with the procedure outlined below.

Wadeable Stream Sampling Procedure:

1. Put on nitrile gloves. Make sure not to handle sunscreen or other chemical contaminants until after the sample is collected.

2. Collect the water samples from the stream sample site in a flowing portion near the middle of the stream. Be sure to collect the water samples prior to any disturbance of the stream upstream of the site.

3. Rinse the pre-cleaned 1-L glass sample jar three times with water, and discard the rinse downstream.

4. Fill the 1-L glass sample jar with water and slowly pour into the 500ml HDPE sample bottle (for metals and hardness analysis) until full. Cap the bottle tightly. Fill the 1-L glass sample jar with water and slowly pour into the 1-L HDPE sample bottle (for alkalinity, TSS/TDS, and anions) until full. Cap the bottle tightly.

5. Collect a stream sample for field analysis (temperature, pH, specific conductance, and DO) in a 1-L glass jar and analyze within 15 minutes of collection.

6. Record any pertinent stream information (stream name, ID, date, etc.). Note anything that could influence sample chemistry (heavy rain, potential contaminants).

7. Following sampling and preservation, EPA will immediately pack sample bottles in coolers and chill them to less than 6°C. EPA will make best efforts to collect samples on September 12-13, for delivery to CRL by September 14.

EPA will collect one field blank using DI water obtained from the Central Regional Laboratory or from EPA's Cleveland Office, to be analyzed for the parameters listed in Table 2 of the QAPP/SAMP. EPA will pour DI water into an unused and clean grab sample collection bottle, (where used for that individual analyses), agitate it, and then pour into the field blank sample bottles. Blank bottles will be prepared and preserved for each analysis listed in Table 3 of the QAPP/SAMP.

Table 3 of the QAPP/SAMP contains a listing of the parameters to be analyzed, analytical methods, preservatives and containers to be used during this project. The actual number and types of samples collected will be determined by the EPA inspectors on-site during the

inspection. EPA Inspectors will contact the laboratory immediately following the inspection and notify the lab of the actual number and types of samples being brought in for analysis.

Decontamination Procedures
New or pre-cleaned dedicated sampling equipment will be used at the sampling site. No onsite decontamination procedures are anticipated for this sampling activity.

Sample Packaging and Transport
See QAPP/SAMP.

Safety Procedures
See QAPP/SAMP.

Sampling Plan Modification
This sampling plan is subject to modification depending upon conditions encountered in the field and other circumstances.

Assessments
See QAPP/SAMP.

Appendix D: Receipt for Samples and Documents, Attendance Sheet, Certification of Analysis for Sample Containers

ED_004744_00033652-00072

INSPECTION DOCUMENTS RECEIVED BY EPA

Page 1 of 1

Facility Name: USS-MINNTAC TAILINGS BASIN Inspection Date: 9/12/17-9/13/17
US EPA Inspector: DEAN MARALDO
Facility Representative: TOM MOE Title: ENV ENGINEER

Doc Number	Document Name	Claimed Confidential Business Information (CBI) Y/N
1	ORG STRUCTURE DOCUMENTS (FLASH DRIVE)	N
2	SITE PLANS AND MAPS (FLASH DRIVE)	N
3	NPDES REPORTABLE INCIDENTS (FLASH DRIVE)	N
4	TB WATER BALANCE-FLOW DIAG-SEEP REPORTS (FLASH DRIVE)	N
5	NPDES PERMIT APP (FLASH DRIVE)	N
6	GROUNDWATER FLOW/SULFATE TRANSPORT MODEL DOCS+DATA (FLASH DRIVE)	N
7	DMR AND LAB DATA (FLASH DRIVE)	N
8	DARK RIVER SEEP COLLECTION DOCS (FLASH DRIVE)	N
9	2011 GEOTECHNICAL INVEST REPORT (HATCH 2011) (FLASH DRIVE)	N
10	MINNTAC SWPPP 6-29-09 (FLASH DRIVE)	N
11	WEST TAILINGS BASIN SURFACE SEEPAGE SURVEY 2012 (FLASH DRIVE)	N

Facility Representative Signature: [signature] Date: 9-13-17

US Steel Minntac Tailings Basin Inspection
September 2017

ATTENDANCE SHEET

FACILITY NAME: MINNTAC

DATE: 9/12/17

Name	Title	Phone #	Email
DEAN MARALDO	INSPECTOR	312-353-2098	MARALDO.DEAN@EPA.gov
JONATHAN MOODY	INSPECTOR	440-250-1708	moody.jonathan@gmail.com
ERIC WILLIAMS	MANAGER - WATER HQ	412-302-3624	ewilliams@uss.com
TOM MOE	ENV. ENGR	218 749 7485	tmoe@uss.com
Chrissy Bartovich	Director - Environmental	218-749-7364	clbartovich@uss.com

RECEIPT FOR SAMPLES

Project Number	Project Name	Name of Facility
	02DM17	USS - MINNTAC CSI

Samplers Name (Signature) DEAN MARALDO
JONATHAN MOODY

Facility Location
COUNTY ROAD 102
MOUNTAIN IRON, MN 55768

Split Samples Offered
( ) Accepted ( ) Declined

FACILITY TECH CO-LOCATED FIELD SAMPLES

Station Number	Date	Time (EDT) or (EST)	Comp.	Grab	~~Split~~ JM Samples CO-LOCATED	Tag Numbers	Station Description	Number of Containers	Remarks
S01	9/12/17	0957		X	X		SEEP A	2	
S02		1023		X	X		DARK RIVER POND SEEP	2	
S03		1050		X	X		SEEP 3	2	
S04		1108		X	X		SD001	4	MS/MSD
D05		1108		X	X		SD001 DUPLICATE	2	
S06		1130		X	X		SEEP 7	2	
S07		1151		X	X		SEEP 13	2	
S08		1403		X	X		EAST "BACKGROUND" WETLAND	2	
S09		1500		X	X		CB-12 OUTSIDE SHEET PILE	2	
S10	9/12/17	1515		X	X		CB-12 INSIDE SHEET PILE	2	
S11	9/13/17	0944		X	X		CB-5 OUTSIDE SHEET PILE MID	2	
S12		0949		X	X		CB-5 INSIDE SHEET PILE	2	
S13		0955		X			CB-5 OUTSIDE SHEET PILE NORTH	2	
S14	9/13/17	1023		X	X		DARK RIVER POND	2	

~~Transferred by (Signature)~~ N/A

~~Received by (Signature)~~ N/A

Telephone

Date | Time

Title | Date | Time

RECEIPT FOR SAMPLES

Project Number	Project Name	Name of Facility
	02DM17	USS - MINNTAC CSI

Samplers Name (Signature) DEAN MARALDO
JONATHAN MOODY

Facility Location
COUNTY ROAD 102
MOUNTAIN IRON, MN 55768

Split Samples Offered
( ) Accepted ( ) Declined FACILITY TOOK CO-LOCATED FIELD SAMPLES

Station Number	Date	Time (EDT) or (EST)	Comp.	Grab	~~Split~~ Samples Co-Located	Tag Numbers	Station Description	Number of Containers	Remarks
S15	9/13/17	1110		X	X		TIMBER CREEK	2	
F16	9/13/17	1252		X			FIELD BLANK	2	

~~Transferred by (Signature)~~	~~Received by (Signature)~~	Telephone
N/A	N/A	
Date / Time	Title / Date / Time	

Qorpak
A Division of Berlin Packaging

CERTIFICATE OF ANALYSIS

This "Certificate of Analysis" represents a precleaned product that has been prepared in accordance with Performance-Based specifications. This product meets or exceeds analyte specifications established in the U. S. EPA OSWER Directive 9240.0-05A "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other Hazardous waste programs.

Group 2 Containers for Inorganics

Analyte	RL ug/L	Analyte	RL ug/L	Analyte	RL ug/L
Aluminum	10 U	Copper	1 U	Potassium	50 U
Antimony	1 U	Iron	75 U	Selenium	0.5 U
Arsenic	0.5 U	Lead	0.1 U	Silicon	50 U
Barium	0.5 U	Lithium	0.5 U	Silver	0.5 U
Beryllium	0.5 U	Magnesium	10 U	Sodium	50 U
Bismuth	0.5 U	Manganese	0.5 U	Strontium	0.5 U
Boron	5.0 U	Mercury	0.2 U	Thallium	0.5 U
Cadmium	0.1 U	Molybdenum	0.5 U	Tin	0.5 U
Calcium	40 U	Nickel	0.5 U	Titanium	1 U
Chromium (Total)	0.5 U	Palladium	0.5 U	Vanadium	1 U
Cobalt	1 U	Platinum	0.5 U	Zinc	5 U

Analyte	RL mg/L	Analyte	RL mg/L	Analyte	RL mg/L
Alkalinity	5.0 U	Chloride	0.5 U	Cyanide	0.02 U
Fluoride	0.2 U	Sulfide	0.05 U	Sulfate	0.5 U

NOTES:
a. Reporting Limit (RL) = The lowest concentration standard analyzed which can be verified.
b. U = The analyte was analyzed for but not detected above the Reporting Limit.

This "Certificate of Analysis" is provided for your records and is used to facilitate any required correspondences as needed.

Each container contains:

Barcoded: Yes *Stirbars:* No *Tared Weight:* No

Part Number: LPW032200013 **Date Product Prepared:** 03/20/17

Item Description: 950cc Natural W/M w/ 53-400 F217 lined cap **Lot Number:** 031817-2APN

Chemical Lot No.: N/A

Protocol: A **Level:** 1

Chemical Expiry Date: N/A

Group: 2 (applies)

Corporate Technical Director

Product processed at:
2202 I-49 N. Service Rd.
Opelousas LA 70570 USA

Revision 030817-06 st

Qorpak
A Division of Berlin Packaging

CERTIFICATE OF ANALYSIS

This "Certificate of Analysis" represents a precleaned product that has been prepared in accordance with Performance-Based specifications. This product meets or exceeds analyte specifications established in the U. S. EPA OSWER Directive 9240.0-05A "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other Hazardous waste programs.

Group 2 Containers for Inorganics

Analyte	RL ug/L	Analyte	RL ug/L	Analyte	RL ug/L
Aluminum	10 U	Copper	1 U	Potassium	50 U
Antimony	1 U	Iron	75 U	Selenium	0.5 U
Arsenic	0.5 U	Lead	0.1 U	Silicon	50 U
Barium	0.5 U	Lithium	0.5 U	Silver	0.5 U
Beryllium	0.5 U	Magnesium	10 U	Sodium	50 U
Bismuth	0.5 U	Manganese	0.5 U	Strontium	0.5 U
Boron	5.0 U	Mercury	0.2 U	Thallium	0.5 U
Cadmium	0.1 U	Molybdenum	0.5 U	Tin	0.5 U
Calcium	40 U	Nickel	0.5 U	Titanium	1 U
Chromium (Total)	0.5 U	Palladium	0.5 U	Vanadium	1 U
Cobalt	1 U	Platinum	0.5 U	Zinc	5 U

Analyte	RL mg/L	Analyte	RL mg/L	Analyte	RL mg/L
Alkalinity	5.0 U	Chloride	0.5 U	Cyanide	0.02 U
Fluoride	0.2 U	Sulfide	0.05 U	Sulfate	0.5 U

NOTES:
a. Reporting Limit (RL) = The lowest concentration standard analyzed which can be verified.
b. U = The analyte was analyzed for but not detected above the Reporting Limit.

This "Certificate of Analysis" is provided for your records and is used to facilitate any required correspondences as needed.

Each container contains:

Barcoded: Yes *Stirbars:* No *Tared Weight:* No

Part Number: LPW032200013 **Date Product Prepared:** 03/20/17

Item Description: 950cc Natural W/M w/ 53-400 F217 lined cap **Lot Number:** 031817-2APN

Chemical Lot No.: N/A

Protocol: A **Level:** 1

Group: 2 (applies) **Chemical Expiry Date:** N/A

Corporate Technical Director

Product processed at:
2202 I-49 N. Service Rd.
Opelousas LA 70570 USA

Revision 030817-08 sf

Qorpak
A Division of Berlin Packaging

CERTIFICATE OF ANALYSIS

This "Certificate of Analysis" represents a precleaned product that has been prepared in accordance with Performance-Based specifications. This product meets or exceeds analyte specifications established in the U. S. EPA OSWER Directive 9240.0-05A "Specification and Guidance for Contaminant-free Sample Containers" for use in Superfund and other Hazardous waste programs.

Group 2 Containers for Inorganics

Analyte	RL ug/L	Analyte	RL ug/L	Analyte	RL ug/L
Aluminum	10 U	Copper	1 U	Potassium	50 U
Antimony	1 U	Iron	75 U	Selenium	0.5 U
Arsenic	0.5 U	Lead	0.1 U	Silicon	50 U
Barium	0.5 U	Lithium	0.5 U	Silver	0.5 U
Beryllium	0.5 U	Magnesium	10 U	Sodium	50 U
Bismuth	0.5 U	Manganese	0.5 U	Strontium	0.5 U
Boron	5.0 U	Mercury	0.2 U	Thallium	0.5 U
Cadmium	0.1 U	Molybdenum	0.5 U	Tin	0.5 U
Calcium	40 U	Nickel	0.5 U	Titanium	1 U
Chromium (Total)	0.5 U	Palladium	0.5 U	Vanadium	1 U
Cobalt	1 U	Platinum	0.5 U	Zinc	5 U

Analyte	RL mg/L	Analyte	RL mg/L	Analyte	RL mg/L
Alkalinity	5.0 U	Chloride	0.5 U	Cyanide	0.02 U
Fluoride	0.2 U	Sulfide	0.05 U	Sulfate	0.5 U

NOTES:
a. Reporting Limit (RL) = The lowest concentration standard analyzed which can be verified.
b. U = The analyte was analyzed for but not detected above the Reporting Limit.

This "Certificate of Analysis" is provided for your records and is used to facilitate any required correspondences as needed.

Each container contains:

Barcoded: Yes *Stirbars:* No *Tared Weight:* No

Part Number: LPW016200012 **Date Product Prepared:** 03/20/17

Item Description: 500cc Natural W/M w/ 53-400 F217 lined cap **Lot Number:** 031817-2ANV

Chemical Lot No.: N/A

Protocol: A **Level:** 1

Group: 2 (applies) **Chemical Expiry Date:** N/A

Corporate Technical Director

Product processed at:
2202 I-49 N. Service Rd.
Opelousas LA 70570 USA

Revision 030617-06 sf

MINNESOTA ORE OPERATIONS – USS

HAZARD TRAINING PROGRAM

This training is for those who spend less than five consecutive days on the property.

In accordance with 30 CFR part 48.31, the following instructions apply to your visit of Minnesota Ore Operations. For your personal safety **STRICT ADHERENCE** of these regulations must be observed at all times.

Personal Protective Equipment: Approved hard hats, safety glasses and metatarsal guard footwear must be worn in all areas of the property except the administration offices, training rooms and other designated office areas. Hearing and respiratory protective devices must be worn in designated areas. After your first visit to Minnesota Ore Operations you must provide your own hard hat, safety glasses, and footwear. Entry may be denied for subsequent visits if you do not have these items with you.

Hazard Recognition and Avoidance

- Firearms, alcoholic beverages, drugs, narcotics are not to be transported or used on company property.
- Company policy is to arrest and prosecute anyone who commits theft of Minnesota Ore Operations property.
- Observe all "NO SMOKING" areas.
- There is no smoking in any building on the property.
- Use caution when putting out cigarettes, etc.
- Do not step in front of or get close to working or moving machinery as the operator may not see you.
- Use extreme caution when entering and exiting buildings with equipment, etc.
- Never take short cuts, use only designated walkways, watch your step, walk, don't run.
- Never walk under suspended loads.
- Be alert for slippery floors and tripping hazards.
- Shield your eyes in the immediate area of cutting and welding operations.
- Keep clear of jib crane and overhead crane swing areas.
- Know the fire exits and fire alarm warning.
- Loads delivered or picked up must be secured. While working on a truck bed, consider tie-off when above four feet off the ground or when secure footing cannot be maintained.
- Don't cross under, over, or between railroad cars.
- All vehicles are subject to periodic, unannounced inspection at any time and at any location on the property.
- Obey all traffic control signs. Stop, Look, and Listen at railroad crossings.
- The maximum speed limits are posted and will be strictly enforced.
- Park only in designated areas and do not park on or within six feet of railroad tracks.
- All mine operating equipment has the right-of-way over passenger vehicles.
- Authorization is required to drive on roads in the Mining Division areas or visit areas other than those related to your assignment.
- If authorized to be in maintenance shop areas, agglomerator, concentrator, crushers, mine, dumping, or loading-out facilities, you must have an MSHA 5000-23 Form or be accompanied by a Minnesota Ore Operations employee.

ISO 14001 Environmental Policy – 3C's

Continuous Improvement to the Environment (CITE)

Commitment to Prevention of Pollution

Compliance with Laws and Regulations

Emergency procedures

- In case of emergency, follow the directions of the area supervisor or your U.S.S. contact.
- Any accident or injury occurring during your visit must be reported to the area supervisor immediately.
- If you have any questions concerning the emergency procedures or safety regulations ,contact the Safety Department or the Security Gate.
  Emergency Phone Numbers Minntac: 1911 or 218-749-7442 Keetac: Fire: 500 Medical: 400 or 218-778-8798

On behalf of myself; my parent and subsidiary corporations; in consideration for the granting of permission to enter upon the property owned by Minnesota Ore Operations – U.S.S.; I hereby release and agree to hold harmless and indemnify Minnesota Ore Operations, affiliates, officers, employees, and agents from and against all claims and liability for any and all injuries, losses and damages to any person or property arising out of entry onto Minnesota Ore Operation's property from the date of this release.

I certify that I have read, understand, and will comply with the above safety regulations.

Print Name Dan Marino Date 9/12/17 Company U.S.E.P.A.

Signature

Hazard Training form is good for one year, must be carried when on M.O.O. property, and presented when requested.

CAT ID: 1898317 Rev. 04/14/15

Appendix E: Sample Chain of Custody (COC) Records

ED_004744_00033652-00081

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 5
77 West Jackson Boulevard
Chicago, Illinois 60604

CHAIN OF CUSTODY RECORD

Activity Code:

PROJ. NO.: 02DM17
PROJECT NAME: USS - Minntac CSI

SAMPLERS: (Print Name and Sign)
Dean Mamilton
Jonathan Moody

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Analyte: Total Metals (Hg CVAF)*	Hardness	Anions, Total Solids, Alkalinity				TAG NUMBERS
S01	9/13/17	09:50		✓	Seep A	2	1	1					*HNO3 to pH<2; ICE
S02		10:23		✓	Dark River Pond Seep	2	1	1					" "
S03		10:50		✓	Seep 3	2	1	1					" "
S04		11:08		✓	SD001	4	2	2					" " ; Contains extra sample bottle
D05		11:08		✓	SD001 Duplicate	2	1	1					" "
S06		11:30		✓	Seep 7	2	1	1					" "
S07		11:51		✓	Seep 13	2	1	1					" "
S08		14:05		✓	East "Background" wetland	2	1	1					" "
S09	↓	15:00		✓	CB-12 outside Sheet Pile	2	1	1					" "
S10	9/14/17	15:15		✓	CB-12 inside Sheet Pile	2	1	1					" "
S11	9/14/17	09:44		✓	CB-5 [illegible]	2	1	1					" "
S12		09:49		✓	CB-5 [illegible]	2	1	1					" "
S13		09:55		✓	CB-5 [illegible]	2	1	1					" "
S14	↓	10:23		✓	Dark River Pond	2	1	1					" "
S15	9/13/17	11:10		✓	Timber Creek	2	1	1					*HNO3 to pH<2; ICE

Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Jonathan Moody	9/14/17 1326	
Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature) / Date / Time
		14 SEP 17 1326

Ship To: U.S. EPA R5 CRL
536 Clark St. (10th FL)
Chicago IL 60605
ATTN: Rob Snyder

Airbill Number:

Chain of Custody Seal Numbers: 109207 } 109208

Distribution: White - Accompanies Shipment; Pink - Coordinator Field Files; Yellow - Laboratory File

5-23960

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 5
77 West Jackson Boulevard
Chicago, Illinois 60604

CHAIN OF CUSTODY RECORD

Activity Code:

PROJ. NO.: 02DM17
PROJECT NAME: USS-Mimtac CSI

SAMPLERS: (Print Name and Sign)
Dean Mansfield
Jonathan Moody

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Analyte: Tag: Metals, Hg (CVM) * Hardness	Anions, TDS/TSS Alkalinity					TAG NUMBERS
R16	9/13/17	1252		✓	Field Blank	2	1	1					*HNO3 to pH<2; ICE

Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Jonathan Moody	9/14/17 1326	
Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature) / Date / Time
		14SEP17 1326

Ship To:
U.S. EPA RS CRL
536 S. Clark St (10th Fl)
Chicago, IL 60605
ATTN: Rob Snyder

Airbill Number

Chain of Custody Seal Numbers
109207 & 109208

Distribution: White - Accompanies Shipment; Pink - Coordinator Field Files; Yellow - Laboratory File

Printed on Recycled Paper/Printed with Soy Based Ink

5-23961

Appendix F: EPA Compliance Sampling Analytical Results

ED_004744_00033652-00084

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2280 Testing

Date: 10/2/2017

Subject: **Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility**

To: **Water Division, WECA Region 5**
77 West Jackson Boulevard
Chicago, IL 60604

From: **Amanda Wroble, Chemist**
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X AMANDA WROBLE Digitally signed by AMANDA WROBLE Date: 2017.10.02 16:25:33 -05'00'

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

/ /

Data Coordinator and Date Transmitted

Analyses included in this report:

Metals ICP-MS (w)

ED_004744_00033652-00085

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-02-17 16:21

ANALYSIS CASE NARRATIVE

GENERAL INFORMATION

CRL received water samples for trace metals analysis by inductively coupled plasma-mass spectrometry (ICP-MS) on September 14, 2017. The assigned work order(s) and sample numbers to which this narrative applies are listed below:

Work Order (number of samples)	Batch Number
1709003 (16)	B17I021

The designated analyst for these samples, Amanda Wroble, can be reached at 312.353.0375. All holding times were met.

The samples were digested using CRL SOP METALS025, Rev. 3 (EPA Method 200.2) and analyzed using CRL SOP METALS001, Rev. 7 (EPA Method 200.8/SW-846 Method 6020B).

SAMPLE ANALYSIS AND RESULTS

The data reported herein meet the requirements referenced in the SOP used for analysis and any laboratory specifications stated in the *Quality Assurance Project Plan/Sampling Plan for Environmental Compliance Assessment for U.S. Steel – Minntac Tailings Basin Area Facility* dated August 2017 with a few minor exceptions. Barium was reported from ICP-MS instead of ICP-AES; and chromium, manganese, and molybdenum were reported down to 1.0 µg/L, which was lower than reporting limits specified in the QAPP.

Manganese results for multiple samples exceeded the highest calibration point (500 µg/L). Linearity was demonstrated up to 5 mg/L within the run. Manganese results for these samples that were less than the linearity check were reported from the undiluted samples. The manganese result that exceeded the linearity check (1709003-09) was reported from a fivefold dilution.

All analytes were reported from Run 1 with the exception of arsenic, which was reported from Run 2 due to failure of low level quality control audits (laboratory control blank and reporting limit check) for arsenic.

Several analytes were present in the field blank above their respective method detection limits (MDL). No flags were applied to the data, as CRL has no audit limits for field quality control samples.

QUALITY CONTROL

All quality control audits were within CRL limits for the requested analytes or did not result in qualification of the data.

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ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

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Metals by ICP-MS using EPA 200.8 (modified)
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S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	2.64			1.00	"	"	"	"	Sep-21-17
Barium	31.8			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	711			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	U			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	1.11			1.00	"	"	"	"	Sep-21-17
Barium	19.7			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	28.2			1.00	"	"	"	"	"
Molybdenum	1.17			1.00	"	"	"	"	"
Nickel	1.18			1.00	"	"	"	"	"

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Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Selenium	U			2.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	18.2			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	4.21			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	2340			1.00	"	"	"	"	"
Molybdenum	2.05			1.00	"	"	"	"	"
Nickel	3.95			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	40.0			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	1.26			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"

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77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Lead	U			0.50	ug/L	1	B171021	Sep-20-17	Sep-21-17
Manganese	1530			1.00	"	"	"	"	"
Molybdenum	3.87			1.00	"	"	"	"	"
Nickel	2.51			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	40.4			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	1.23			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	1540			1.00	"	"	"	"	"
Molybdenum	3.93			1.00	"	"	"	"	"
Nickel	2.47			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

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77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	22.3			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	5.31			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	1130			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	3.45			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	1.03			1.00	"	"	"	"	Sep-21-17
Barium	U			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	117			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	U			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"

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S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Zinc	U			10.0	ug/L	1	B171021	Sep-20-17	Sep-21-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	22.9			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	0.64			0.50	"	"	"	"	"
Copper	2.05			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	217			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	2.32			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	14.0			1.00	"	"	"	"	Sep-21-17
Barium	125			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	3.08			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	9980			5.00	"	5	"	"	Sep-21-17
Molybdenum	U			1.00	"	1	"	"	Sep-21-17

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S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Nickel	**2.59**			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	**10.8**			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	**0.53**			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	**330**			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	**2.37**			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	**1.52**			1.00	"	"	"	"	Sep-21-17
Barium	**27.2**			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	**0.75**			0.50	"	"	"	"	"

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Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

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S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Copper	U			2.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Lead	U			0.50	"	"	"	"	"
Manganese	522			1.00	"	"	"	"	"
Molybdenum	7.52			1.00	"	"	"	"	"
Nickel	2.18			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	2.12			1.00	"	"	"	"	Sep-21-17
Barium	80.6			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	3.01			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	1980			1.00	"	"	"	"	"
Molybdenum	16.1			1.00	"	"	"	"	"
Nickel	2.18			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

Environmental Protection Agency Region 5
Chicago Regional Laboratory
536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	1.89			1.00	"	"	"	"	Sep-21-17
Barium	37.9			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	2.41			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	2160			1.00	"	"	"	"	"
Molybdenum	9.45			1.00	"	"	"	"	"
Nickel	2.24			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	1.21			1.00	"	"	"	"	Sep-21-17
Barium	10.7			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	76.2			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	U			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"

ED_004744_00033652-00096

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-02-17 16:21

Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Zinc	U			10.0	ug/L	1	B171021	Sep-20-17	Sep-21-17

S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	1.13			1.00	"	"	"	"	Sep-21-17
Barium	33.7			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	47.4			1.00	"	"	"	"	"
Molybdenum	U			1.00	"	"	"	"	"
Nickel	U			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Antimony	U			1.00	ug/L	1	B171021	Sep-20-17	Sep-21-17
Arsenic	U			1.00	"	"	"	"	Sep-21-17
Barium	U			10.0	"	"	"	"	Sep-21-17
Beryllium	U			1.00	"	"	"	"	"
Cadmium	U			1.00	"	"	"	"	"
Chromium	U			1.00	"	"	"	"	"
Cobalt	U			0.50	"	"	"	"	"
Copper	U			2.00	"	"	"	"	"
Lead	U			0.50	"	"	"	"	"
Manganese	U			1.00	"	"	"	"	"

Environmental Protection Agency Region 5
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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

Metals by ICP-MS using EPA 200.8 (modified)
US EPA Region 5 Chicago Regional Laboratory

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Molybdenum	U			1.00	ug/L	1	B170021	Sep-20-17	Sep-21-17
Nickel	U			1.00	"	"	"	"	"
Selenium	U			2.00	"	"	"	"	"
Silver	U			1.00	"	"	"	"	"
Thallium	U			0.50	"	"	"	"	"
Zinc	U			10.0	"	"	"	"	"

ED_004744_00033652-00098

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-02-17 16:21

Notes and Definitions

U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2280 Testing

Date: 10/13/2017

Subject: Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility

To: Water Division, WECA Region 5
77 West Jackson Boulevard
Chicago, IL 60604

From: Francis Awanya, Chemist
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X Francis A Awanya 10/13/2017

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

/ /

Data Coordinator and Date Transmitted

Analyses included in this report:

Solids, TDS Solids, TSS

ED_004744_00033652-00100

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Analysis Case Narrative

General Information

Sixteen (16) water samples collected for the project were received at the Chicago Regional Laboratory (CRL) on 09/14/2017. Total Suspended Solids (Solids, TSS) analysis was assigned to those samples. The samples were analyzed for TSS using Standard Operating Procedure (SOP) CRL Document # AIG018 Version # 3 (Based on SM 2540 D (APHA, AWWA, WEF)). The designated analyst is Francis Awanya. Francis can be reached at 312-886-3682. Other pertinent information is provided in the final analysis report.

The sample preparation and analysis began on 09/20/2017 and were completed on 09/22/2017. Samples 1709003-01 (Sample Name: S01), 1709003-02 (Sample Name: S02), 1709003-03 (Sample Name: S03), 1709003-04 (Sample Name: S04), 1709003-05 (Sample Name: D05), 1709003-06 (Sample Name: S06), 1709003-07 (Sample Name: S07), 1709003-08 (Sample Name: S08), 1709003-09 (Sample Name: S09), 1709003-10 (Sample Name: S10) were not analyzed within the holding time. The sample results are flagged "J" for estimated: The identification of the analyte is acceptable; the reported value is an estimate. The remaining samples were analyzed within the holding time.

Sample Analysis and Results

The data reported herein met the requirements of Quality Assurance Project Plan (QAPP)/Sampling Plan for Environmental Compliance Assessment for U.S. Steel – Minntac Tailings Basin Area Facility dated August 2017 and the CRL SOP used for analysis. The QAPP is archived as "MN0057207_Minntac_QAPPSAMP_20170831" in the CRL share drive.

Quality Control

All required quality control criteria for the laboratory, method, and system performance audits were evaluated and determined to be within the CRL's QC limits except analysis holding time for the samples listed above.

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Analysis Case Narrative

General Information

Sixteen (16) water samples collected for the project were received at the Chicago Regional Laboratory (CRL) on 09/14/2017. Total Dissolved Solids (Solids, TDS) analysis was assigned to those samples. The samples were analyzed for TDS using Standard Operating Procedure (SOP) CRL Document # AIG017 Version # 4 (Based on SM 2540 C (APHA, AWWA, WEF)). The designated analyst is Francis Awanya. Francis can be reached at 312-886-3682. Other pertinent information is provided in the final analysis report.

The sample preparation and analysis began on 09/20/2017 and were completed on 09/22/2017. Samples 1709003-01 (Sample Name: S01), 1709003-02 (Sample Name: S02), 1709003-03 (Sample Name: S03), 1709003-04 (Sample Name: S04), 1709003-05 (Sample Name: D05), 1709003-06 (Sample Name: S06), 1709003-07 (Sample Name: S07), 1709003-08 (Sample Name: S08), 1709003-09 (Sample Name: S09), 1709003-10 (Sample Name: S10) were not analyzed within the holding time. The sample results are flagged "J" for estimated: The identification of the analyte is acceptable; the reported value is an estimate. The remaining samples were analyzed within the holding time.

Sample Analysis and Results

The data reported herein met the requirements of Quality Assurance Project Plan (QAPP)/Sampling Plan for Environmental Compliance Assessment for U.S. Steel – Minntac Tailings Basin Area Facility dated August 2017 and the CRL SOP used for analysis. The QAPP is archived as "MN0057207_Minntac_QAPPSAMP_20170831" in the CRL share drive.

Quality Control

All required quality control criteria for the laboratory, method, and system performance audits were evaluated and determined to be within the CRL's QC limits except analysis holding time for the samples listed above.

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Chicago Regional Laboratory

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Dissolved Solids, SM 2540C (modified)
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1040	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1520	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	2350	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	2280	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	2300	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	2270	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1210	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	104	J		20.0	mg/L	1	B17I024	Sep-20-17	Sep-20-17

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Dissolved Solids, SM 2540C (modified)
US EPA Region 5 Chicago Regional Laboratory

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1780	J		20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1970	J		20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1930			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1890			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1900			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	594			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	1190			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Dissolved Solids	U			20.0	mg/L	1	B170l024	Sep-20-17	Sep-20-17

Environmental Protection Agency Region 5
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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Total Suspended Solids, SM 2540 D (modified)
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	6	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	10	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	12	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	13	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Total Suspended Solids, SM 2540 D (modified)
US EPA Region 5 Chicago Regional Laboratory

S09 (1709003-09) Matrix: Water Sampled: Sep-12-17 15:00 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	206	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S10 (1709003-10) Matrix: Water Sampled: Sep-12-17 15:15 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U	J		5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S11 (1709003-11) Matrix: Water Sampled: Sep-13-17 09:44 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S12 (1709003-12) Matrix: Water Sampled: Sep-13-17 09:49 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	12			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S13 (1709003-13) Matrix: Water Sampled: Sep-13-17 09:55 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	18			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S14 (1709003-14) Matrix: Water Sampled: Sep-13-17 10:23 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	6			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

S15 (1709003-15) Matrix: Water Sampled: Sep-13-17 11:10 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

R16 (1709003-16) Matrix: Water Sampled: Sep-13-17 12:52 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Suspended Solids	U			5	mg/L	1	B170025	Sep-20-17	Sep-20-17

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-13-17 13:15

Notes and Definitions

J	The identification of the analyte is acceptable; the reported value is an estimate.
U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

ED_004744_00033652-00108

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2280 Testing

Date: 10/24/2017

Subject: Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility

To: Water Division, WECA Region 5
77 West Jackson Boulevard
Chicago, IL 60604

From: Colin Breslin, Chemist
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

x COLIN BRESLIN Digitally signed by COLIN BRESLIN Date: 2017.10.24 13:29:57 -05'00'

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

/ /

Data Coordinator and Date Transmitted

Analyses included in this report:

Hg Total CVAA

ED_004744_00033652-00109

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-24-17 13:25

Analysis Case Narrative

General Information

Water samples for the analysis of total mercury by cold vapor atomic absorption (Hg Total CVAA) were received at the Chicago Regional Laboratory (CRL). Dates of receipt are included in the body of this report. The samples met the pH preservation requirement upon receipt. The samples were analyzed within the 28 day hold time. The designated analyst, Colin Breslin, can be reached at 312-886-2912.

The samples were prepared and analyzed according to CRL SOP AIG044E Version #4 (based on EPA 245.1/SW-846: 7470A).

Note: supporting data are archived with work order 1709001.

Sample Analysis and Results

The data reported herein meets the requirements referenced in the SOP used for analysis and any laboratory specifications stated in the U.S. Steel – Minntac Basin Area Facility County Road 102 Mountain Iron, MN 55768 Quality Assurance Project Plan (QAPP), August 2017. The QAPP is saved on the CRL share drive with file name: MN0057207_Minntac_QAPPSAMP_20170831.pdf.

Quality Control

All Quality Control (QC) audits were within CRL limits for the requested analytes or did not result in qualification of the data.

ED_004744_00033652-00110

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-24-17 13:25

ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-24-17 13:25

Mercury by Cold Vapor, EPA Method 245.1/7470A (modified)
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-24-17 13:25

Mercury by Cold Vapor, EPA Method 245.1/7470A (modified)
US EPA Region 5 Chicago Regional Laboratory

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Environmental Protection Agency Region 5
Chicago Regional Laboratory
536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Mirntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-24-17 13:25

Mercury by Cold Vapor, EPA Method 245.1/7470A (modified)
US EPA Region 5 Chicago Regional Laboratory

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Mercury	U			0.5	ug/L	1	B171038	Sep-27-17	Sep-27-17

ED_004744_00033652-00114

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-24-17 13:25

Notes and Definitions

U-	Not Detected
NR	Not Reported
Q	QC limit Exceeded

ED_004744_00033652-00115

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2388 Testing

Date: 10/25/2017

Subject: **Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility**

To: **Water Division, WECA Region 5**
77 West Jackson Boulevard
Chicago, IL 60604

From: **Kathleen Swan, Analyst**
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X KATHLEEN SWAN Digitally signed by KATHLEEN SWAN Date: 2017.10.25 14:47:33 -05'00'

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

/ /

Data Coordinator and Date Transmitted

Analyses included in this report:

Ca ICP (W)	Metals ICP-AES (w)	Mg ICP (W)
Hardness by calc		

Environmental Protection Agency Region 5
Chicago Regional Laboratory
536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

ANALYSIS CASE NOTES
General Information

Work Order	Number of Samples	Date Received
1709003	16	09 14 17

All holding times were met. Analyst K Swan 312 353 9071.

Sample preparation was performed using CRL SOP Metals 025, Version No. 3, based on EPA sample preparation procedure 200.2 and analysis was performed using CRL SOP Metals003, Version 6, based on EPA ICP Method 200.7/SW-846 6010D. Supporting documents filed with 1708008.

Sample Analysis and Results

The data reported herein meet the requirements referenced in the SOP used for analysis and any laboratory specifications stated in the *Quality Assurance Project Plan/Sampling Plan for Environmental Compliance Assessment for U.S. Steel – Minntac Tailings Basin Area Facility* dated August 2017, with the exception that laboratory reporting limit for hardness is 1.32 mg CaCO3/L, which is greater than the 1.00 mg CaCO3/L mentioned in above requirements.

Iron is reported from 273 line and aluminum is reported from 308 line due to QC recoveries.

Quality Control

All quality control (QC) audits were within CRL limits or did not result in qualification of the data, with the following exceptions:

Calcium (and therefore hardness) is flagged J due to various QC recovery issues
Phosphorous is flagged L for 1709003-09 due to possible spectral interference

ED_004744_00033652-00117

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-25-17 14:39

ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.142			0.0500	"	"	"	"	Oct-17-17
Calcium	63.0	J		0.200	"	"	"	"	Oct-17-17
Iron	0.917			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	156			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	4.97			0.800	"	"	"	"	Oct-17-17
Sodium	19.3			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.222			0.0500	"	"	"	"	"
Calcium	86.5	J		0.200	"	"	"	"	Oct-17-17
Iron	U			0.0800	"	"	"	"	Oct-17-17
Lithium	U			0.0250	"	"	"	"	Oct-17-17
Magnesium	233			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	11.8			0.800	"	"	"	"	Oct-17-17
Sodium	27.3			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.318			0.0500	"	"	"	"	Oct-17-17
Calcium	161	J		0.200	"	"	"	"	Oct-17-17
Iron	0.188			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	289			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	16.6			0.800	"	"	"	"	Oct-17-17
Sodium	79.2			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.244			0.0500	"	"	"	"	Oct-17-17
Calcium	146	J		0.200	"	"	"	"	Oct-17-17
Iron	0.141			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	277			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	16.6			0.800	"	"	"	"	Oct-17-17
Sodium	68.7			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

Environmental Protection Agency Region 5
Chicago Regional Laboratory
536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B170021	Sep-20-17	Oct-17-17
Boron	0.243			0.0500	"	"	"	"	"
Calcium	147	J		0.200	"	"	"	"	"
Iron	0.101			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	279			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	16.6			0.800	"	"	"	"	Oct-17-17
Sodium	69.2			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	Oct-17-17
Vanadium	U			0.005	"	"	"	"	Oct-17-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B170021	Sep-20-17	Oct-17-17
Boron	0.297			0.0500	"	"	"	"	"
Calcium	163	J		0.200	"	"	"	"	Oct-17-17
Iron	0.212			0.0800	"	"	"	"	Oct-17-17
Lithium	U			0.0250	"	"	"	"	Oct-17-17
Magnesium	279			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	12.4			0.800	"	"	"	"	Oct-17-17
Sodium	59.1			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.261			0.0500	"	"	"	"	"
Calcium	47.1	J		0.200	"	"	"	"	Oct-17-17
Iron	0.382			0.0800	"	"	"	"	Oct-17-17
Lithium	U			0.0250	"	"	"	"	Oct-17-17
Magnesium	228			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	4.38			0.800	"	"	"	"	Oct-17-17
Sodium	10.8			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	U			0.0500	"	"	"	"	Oct-17-17
Calcium	12.6	J		0.200	"	"	"	"	Oct-17-17
Iron	1.76			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	6.95			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	1.58			0.800	"	"	"	"	Oct-17-17
Sodium	3.02			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	0.431			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.129			0.0500	"	"	"	"	"
Calcium	174	J		0.200	"	"	"	"	Oct-17-17
Iron	35.1			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	188			0.200	"	"	"	"	"
Phosphorus	0.277	L		0.200	"	"	"	"	Oct-17-17
Potassium	5.79			0.800	"	"	"	"	Oct-17-17
Sodium	72.1			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	0.006			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.193			0.0500	"	"	"	"	Oct-17-17
Calcium	175	J		0.200	"	"	"	"	Oct-17-17
Iron	U			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	216			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	7.07			0.800	"	"	"	"	Oct-17-17
Sodium	60.4			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B170021	Sep-20-17	Oct-17-17
Boron	0.232			0.0500	"	"	"	"	Oct-17-17
Calcium	136	J		0.200	"	"	"	"	Oct-17-17
Iron	0.759			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	230			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	20.1			0.800	"	"	"	"	Oct-17-17
Sodium	70.8			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B170021	Sep-20-17	Oct-17-17
Boron	0.186			0.0500	"	"	"	"	Oct-17-17
Calcium	122	J		0.200	"	"	"	"	Oct-17-17
Iron	6.25			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	232			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	19.3			0.800	"	"	"	"	Oct-17-17
Sodium	71.6			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified)
US EPA Region 5 Chicago Regional Laboratory

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.230			0.0500	"	"	"	"	Oct-17-17
Calcium	130	J		0.200	"	"	"	"	Oct-17-17
Iron	1.70			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	225			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	18.9			0.800	"	"	"	"	Oct-17-17
Sodium	68.6			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.0938			0.0500	"	"	"	"	Oct-17-17
Calcium	30.9	J		0.200	"	"	"	"	Oct-17-17
Iron	0.425			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	110			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	5.93			0.800	"	"	"	"	Oct-17-17
Sodium	9.70			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Metals by ICP-AES using EPA 200.7 (modified)
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S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	0.163			0.0500	"	"	"	"	Oct-17-17
Calcium	61.1	J		0.200	"	"	"	"	Oct-17-17
Iron	0.187			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	201			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	9.33			0.800	"	"	"	"	Oct-17-17
Sodium	21.6			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Aluminum	U			0.200	mg/L	1	B171021	Sep-20-17	Oct-17-17
Boron	U			0.0500	"	"	"	"	Oct-17-17
Calcium	U			0.200	"	"	"	"	Oct-17-17
Iron	U			0.0800	"	"	"	"	"
Lithium	U			0.0250	"	"	"	"	"
Magnesium	U			0.200	"	"	"	"	"
Phosphorus	U			0.200	"	"	"	"	Oct-17-17
Potassium	U			0.800	"	"	"	"	Oct-17-17
Sodium	U			0.400	"	"	"	"	"
Tin	U			0.0200	"	"	"	"	Oct-17-17
Titanium	U			0.006	"	"	"	"	"
Vanadium	U			0.005	"	"	"	"	"

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Hardness by Calculation
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	799	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1170	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1590	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1510	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1510	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1560	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1060	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	60.0	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

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Hardness by Calculation
US EPA Region 5 Chicago Regional Laboratory

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1210	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1330	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1290	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1260	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	1250	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	530	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	981	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Hardness	U	J	0.114	1.32	mg CaCO3/L	1	[CALC]	Sep-20-17	Oct-17-17

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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-25-17 14:39

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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified) - Quality Control
US EPA Region 5 Chicago Regional Laboratory

Batch B17I021 - Hot Block Digestion

Blank (B17I021-BLK1) Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	U			0.200	mg/L						
Boron	U			0.0500	"						
Calcium	U			0.200	"						
Iron	U			0.0800	"						
Lithium	U			0.0250	"						
Magnesium	U			0.200	"						
Phosphorus	U			0.200	"						
Potassium	U			0.800	"						
Sodium	U			0.400	"						
Tin	U			0.0200	"						
Titanium	U			0.006	"						
Vanadium	U			0.005	"						

LCS (B17I021-BS1) Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	0.968			0.200	mg/L	1.000		96.8%	85-115		
Boron	1.01			0.0500	"	1.000		101%	85-115		
Calcium	47.8			0.200	"	50.00		95.7%	85-115		
Iron	0.992			0.0800	"	1.000		99.2%	85-115		
Lithium	0.0950			0.0250	"	0.1000		95.0%	85-115		
Magnesium	18.7			0.200	"	20.00		93.5%	85-115		
Phosphorus	1.02			0.200	"	1.000		102%	85-115		
Potassium	23.2			0.800	"	25.00		92.8%	85-115		
Sodium	46.5			0.400	"	50.00		93.1%	85-115		

Environmental Protection Agency Region 5
Chicago Regional Laboratory
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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified) - Quality Control
US EPA Region 5 Chicago Regional Laboratory

Batch B171021 - Hot Block Digestion

LCS (B171021-BS1) Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Tin	0.488			0.0200	mg/L	0.5000		97.5%	85-115		
Titanium	0.0960			0.006	"	0.1000		96.0%	85-115		
Vanadium	0.105			0.005	"	0.1000		105%	85-115		

Duplicate (B171021-DUP1) Source: 1709003-04 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	U			0.200	mg/L		U				20
Boron	0.238			0.0500	"		0.244			2.22	20
Calcium	146			0.200	"		146			0.0530	20
Iron	0.141			0.0800	"		0.141			0.309	20
Lithium	0.0046			0.0250	"		0.0043			6.77	20
Magnesium	277			0.200	"		277			0.118	20
Phosphorus	U			0.200	"		U				20
Potassium	16.4			0.800	"		16.6			0.929	20
Sodium	68.7			0.400	"		68.7			0.0342	20
Tin	0.0051	Q		0.0200	"		0.0039			25.0	20
Titanium	U			0.006	"		U				20
Vanadium	0.0012			0.005	"		0.0011			11.9	20

Duplicate (B171021-DUP2) Source: 1709003-11 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	U			0.200	mg/L		U				20
Boron	0.229			0.0500	"		0.232			1.46	20
Calcium	132			0.200	"		136			3.51	20
Iron	0.736			0.0800	"		0.759			3.17	20
Lithium	0.0043			0.0250	"		0.0049			13.4	20
Magnesium	241			0.200	"		230			4.97	20
Phosphorus	U			0.200	"		U				20
Potassium	19.6			0.800	"		20.1			2.75	20

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified) - Quality Control
US EPA Region 5 Chicago Regional Laboratory

Batch B17I021 - Hot Block Digestion

Duplicate (B17I021-DUP2) Source: 1709003-11 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Sodium	68.4			0.400	mg/L		70.8			3.53	20
Tin	0.0043	Q		0.0200	"		0.0034			24.9	20
Titanium	U			0.006	"		U				20
Vanadium	0.0016	Q		0.005	"		0.0012			28.1	20

Matrix Spike (B17I021-MS1) Source: 1709003-04 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	1.00			0.200	mg/L	1.000	U	100%	75-125		
Boron	1.29			0.0500	"	1.000	0.244	105%	75-125		
Calcium	192			0.200	"	50.00	146	91.6%	75-125		
Iron	1.13			0.0800	"	1.000	0.141	98.5%	75-125		
Lithium	0.102			0.0250	"	0.1000	0.0043	97.7%	75-125		
Magnesium	297			0.200	"	20.00	277	101%	75-125		
Phosphorus	1.10			0.200	"	1.000	U	110%	75-125		
Potassium	40.5			0.800	"	25.00	16.6	95.6%	75-125		
Sodium	117			0.400	"	50.00	68.7	97.0%	75-125		
Tin	0.497			0.0200	"	0.5000	0.0039	98.5%	75-125		
Titanium	0.0841			0.006	"	0.1000	U	84.1%	75-125		
Vanadium	0.106			0.005	"	0.1000	0.0011	105%	75-125		

Matrix Spike (B17I021-MS2) Source: 1709003-11 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Aluminum	0.981			0.200	mg/L	1.000	U	98.1%	75-125		
Boron	1.28			0.0500	"	1.000	0.232	105%	75-125		
Calcium	178			0.200	"	50.00	136	82.7%	75-125		
Iron	1.74			0.0800	"	1.000	0.759	97.8%	75-125		
Lithium	0.101			0.0250	"	0.1000	0.0049	96.0%	75-125		
Magnesium	242	Q		0.200	"	20.00	230	60.6%	75-125		
Phosphorus	1.07			0.200	"	1.000	U	107%	75-125		

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-25-17 14:39

Metals by ICP-AES using EPA 200.7 (modified) - Quality Control
US EPA Region 5 Chicago Regional Laboratory

Batch B171021 - Hot Block Digestion

Matrix Spike (B171021-MS2) Source: 1709003-11 Prepared: Sep-20-17 Analyzed: Oct-17-17

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
Potassium	43.9			0.800	mg/L	25.00	20.1	95.3%	75-125		
Sodium	116			0.400	"	50.00	70.8	91.3%	75-125		
Tin	0.496			0.0200	"	0.5000	0.0034	98.6%	75-125		
Titanium	0.0864			0.006	"	0.1000	U	86.4%	75-125		
Vanadium	0.105			0.005	"	0.1000	0.0012	104%	75-125		

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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	Reported:
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-25-17 14:39

Notes and Definitions

L	The identification of the analyte is acceptable; the reported value may be biased low. The actual value is expected to be greater than the reported value.
J	The identification of the analyte is acceptable; the reported value is an estimate.
U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2280 Testing

Date: 10/26/2017

Subject: Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility

To: Water Division, WECA Region 5
77 West Jackson Boulevard
Chicago, IL 60604

From: Anna Knoebel, Chemist
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X _____

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

_____ / /

Data Coordinator and Date Transmitted

Analyses included in this report:

Anions by IC

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Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-26-17 09:36

ANALYSIS CASE NARRATIVE – Anions by IC

Work order #: 1709003
Analyst: Anna Knoebel
Phone #: (312) 353-9467

General Information

Sixteen samples for Anions by IC (fluoride, chloride, sulfate, and bromide) were received on September 14, 2017. All holding times were met.

Sample Analysis and Results

The samples were analyzed September 26 – 28, 2017 for Anions by IC in water using CRL SOP AIG045A, Version #6 (SOP based on EPA 300.0). The data reported herein meets any laboratory specifications referenced in the sampling QAPP titled "*MN0057207_Minntac_QAPPSAMP_20170831*" and the requirements referenced in CRL SOP AIG045A, Version #6 (SOP based on EPA 300.0) except for those listed in the QC section below.

Quality Control

Matrix Spike (MS)

The chloride and sulfate matrix spike recoveries for samples 1709003-04 (S04) and 1709003-11 (S11) were negative and below the acceptance criteria (80 – 120 %). These samples were diluted, as a result, the spikes were diluted out causing the spike result to be invalid. The sample results were not qualified on this basis

The fluoride matrix spike recoveries for sample 1709003-04 (S04) and 1709003-11 (S11) were also below the acceptance criteria (80 – 120 %). There may be some matrix interferences and the reported values may be biased low. The fluoride sample results were qualified "L" (The identification of the analyte is acceptable; the reported value may be biased low. The actual value is expected to be greater than the reported value).

Duplicate (DUP)

The bromide %RPD for sample 1709003-11 (S11) and its duplicate was above the acceptance criteria (≤ 20%). The sample result was qualified "J" (The identification of the analyte is acceptable; the reported value is an estimate).

Continuing Calibration Blank (CCB)

Six out of the ten CCB results for sulfate were above the MDL (0.02 mg/L). Sulfate was below the MDL in the other blanks. This blank contamination is most likely due to carryover from the high levels of sulfate in the samples. Sample 1709003-16 (R16) was qualified "J" (The identification of the analyte is acceptable; the reported value is an estimate). The remaining sample results were not affected by the blank contamination

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because they were more than ten times the CCB result, no qualifiers were necessary.

All other quality control audits were within CRL limits or did not result in qualification of the data.

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Oct-26-17 09:36

ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-26-17 09:36

Anions by Ion Chromatography, EPA 300.0 (modified)
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.30**			0.02	mg/L	1	B17I037	Sep-26-17	Sep-26-17
Chloride	**26.1**			0.12	"	"	"	"	"
Sulfate as SO4	**314**			0.62	"	5	"	"	Sep-27-17
Bromide	**U**			0.12	"	1	"	"	Sep-26-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.28**			0.02	mg/L	1	B17I037	Sep-26-17	Sep-26-17
Chloride	**25.3**			0.12	"	"	"	"	"
Sulfate as SO4	**532**			1.25	"	10	"	"	Sep-27-17
Bromide	**U**			0.12	"	1	"	"	Sep-26-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**1.08**			0.02	mg/L	1	B17I037	Sep-26-17	Sep-26-17
Chloride	**96.0**			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	**945**			2.50	"	20	"	"	Sep-27-17
Bromide	**1.18**			0.12	"	1	"	"	Sep-26-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**1.44**	L		0.02	mg/L	1	B17I037	Sep-26-17	Sep-26-17
Chloride	**102**			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	**925**			2.50	"	20	"	"	Sep-28-17
Bromide	**0.67**			0.12	"	1	"	"	Sep-26-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**1.32**			0.02	mg/L	1	B17I037	Sep-26-17	Sep-26-17
Chloride	**102**			0.25	"	2	"	"	Sep-27-17

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-26-17 09:36

Anions by Ion Chromatography, EPA 300.0 (modified)
US EPA Region 5 Chicago Regional Laboratory

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Sulfate as SO4	**950**			2.50	mg/L	20	B171037	Sep-26-17	Sep-27-17
Bromide	**0.63**			0.12	"	1	"	"	Sep-26-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**1.00**			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	**87.8**			0.12	"	"	"	"	"
Sulfate as SO4	**847**			2.50	"	20	"	"	Sep-27-17
Bromide	**0.48**			0.12	"	1	"	"	Sep-26-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.28**			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	**11.7**			0.12	"	"	"	"	"
Sulfate as SO4	**343**			0.62	"	5	"	"	Sep-27-17
Bromide	U			0.12	"	1	"	"	Sep-26-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.10**			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	**1.28**			0.12	"	"	"	"	"
Sulfate as SO4	**3.61**			0.12	"	"	"	"	"
Bromide	U			0.12	"	"	"	"	"

S09 (1709003-09) **Matrix: Water** **Sampled: Sep-12-17 15:00** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.55**			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	**122**			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	**702**			1.25	"	10	"	"	Sep-27-17
Bromide	**0.73**			0.12	"	1	"	"	Sep-26-17

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Anions by Ion Chromatography, EPA 300.0 (modified)
US EPA Region 5 Chicago Regional Laboratory

S10 (1709003-10) **Matrix: Water** **Sampled: Sep-12-17 15:15** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	0.64			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	91.2			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	720			1.25	"	10	"	"	Sep-27-17
Bromide	0.57			0.12	"	1	"	"	Sep-26-17

S11 (1709003-11) **Matrix: Water** **Sampled: Sep-13-17 09:44** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	1.97	L		0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	114			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	773			1.25	"	10	"	"	Sep-27-17
Bromide	0.68	J		0.12	"	1	"	"	Sep-26-17

S12 (1709003-12) **Matrix: Water** **Sampled: Sep-13-17 09:49** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	1.64			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	114			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	780			1.25	"	10	"	"	Sep-27-17
Bromide	0.63			0.12	"	1	"	"	Sep-26-17

S13 (1709003-13) **Matrix: Water** **Sampled: Sep-13-17 09:55** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	1.78			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	110			0.25	"	2	"	"	Sep-27-17
Sulfate as SO4	781			1.25	"	10	"	"	Sep-27-17
Bromide	0.51			0.12	"	1	"	"	Sep-26-17

S14 (1709003-14) **Matrix: Water** **Sampled: Sep-13-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	0.13			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	5.24			0.12	"	"	"	"	"
Sulfate as SO4	107			0.25	"	2	"	"	Sep-27-17
Bromide	U			0.12	"	1	"	"	Sep-26-17

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Oct-26-17 09:36

Anions by Ion Chromatography, EPA 300.0 (modified)
US EPA Region 5 Chicago Regional Laboratory

S15 (1709003-15) **Matrix: Water** **Sampled: Sep-13-17 11:10** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	**0.18**			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	**18.8**			0.12	"	"	"	"	"
Sulfate as SO4	**418**			1.25	"	10	"	"	Sep-27-17
Bromide	U			0.12	"	1	"	"	Sep-26-17

R16 (1709003-16) **Matrix: Water** **Sampled: Sep-13-17 12:52** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Fluoride	U			0.02	mg/L	1	B171037	Sep-26-17	Sep-26-17
Chloride	U			0.12	"	"	"	"	"
Sulfate as SO4	**0.12**	J		0.12	"	"	"	"	"
Bromide	U			0.12	"	"	"	"	"

ED_004744_00033652-00142

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Oct-26-17 09:36

Notes and Definitions

L	The identification of the analyte is acceptable; the reported value may be biased low. The actual value is expected to be greater than the reported value.
J	The identification of the analyte is acceptable; the reported value is an estimate.
U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

LABORATORY ACCREDITATION BUREAU
ACCREDITED ISO/IEC 17025
Certificate # L2280 Testing

Date: 1/30/2018

Subject: Review of Region 5 Data for U.S. Steel - Minntac Tailings Basin Area Facility

To: Water Division, WECA Region 5
77 West Jackson Boulevard
Chicago, IL 60604

From: Francis Awanya, Chemist
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.

X Francis A Awanya 1/31/2018

Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: U.S. Steel - Minntac Tailings Basin Area Facility

/ /

Data Coordinator and Date Transmitted

Analyses included in this report:

Alkalinity

ED_004744_00033652-00144

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Jan-30-18 17:16

Analysis Case Narrative

General Information

Sixteen (16) water samples collected for the project were received at the Chicago Regional Laboratory (CRL) on 09/14/2017. The samples were logged in the CRL LIMS for Total Alkalinity analysis on 09/15/2017 under Work Order Number 1709003. The samples were analyzed for Total Alkalinity using Standard Operating Procedure (SOP) CRL Document # AIG005 Version # 3 (Based on SM 2320B). The designated analyst for the samples is Francis Awanya. Francis can be reached at 312-886-3682. Other pertinent information is provided in the final analysis report. Analysis was completed within the holding time.

Sample Analysis and Results

The data reported herein meets the requirements of the CRL analytical SOP used for Total Alkalinity assay and the U.S. Steel – Minntac Basin Area Facility County Road 102 Mountain Iron, MN 55768 Quality Assurance Project Plan (QAPP), August 2017. The QAPP is archived in the CRL share drive with as "MN0057207_Minntac_QAPPSAMP_20170831.pdf".

Quality Control

All required quality control criteria for the laboratory, method, and system performance audits were evaluated and determined to be within the CRL's QC limits or did not result in qualification of the data.

ED_004744_00033652-00145

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Jan-30-18 17:16

ANALYTICAL REPORT FOR SAMPLES

Sample ID	Laboratory ID	Matrix	Date Sampled	Date Received
S01	1709003-01	Water	Sep-12-17 09:57	Sep-14-17 13:26
S02	1709003-02	Water	Sep-12-17 10:23	Sep-14-17 13:26
S03	1709003-03	Water	Sep-12-17 10:50	Sep-14-17 13:26
S04	1709003-04	Water	Sep-12-17 11:08	Sep-14-17 13:26
D05	1709003-05	Water	Sep-12-17 11:08	Sep-14-17 13:26
S06	1709003-06	Water	Sep-12-17 11:30	Sep-14-17 13:26
S07	1709003-07	Water	Sep-12-17 11:51	Sep-14-17 13:26
S08	1709003-08	Water	Sep-12-17 14:03	Sep-14-17 13:26
S09	1709003-09	Water	Sep-12-17 15:00	Sep-14-17 13:26
S10	1709003-10	Water	Sep-12-17 15:15	Sep-14-17 13:26
S11	1709003-11	Water	Sep-13-17 09:44	Sep-14-17 13:26
S12	1709003-12	Water	Sep-13-17 09:49	Sep-14-17 13:26
S13	1709003-13	Water	Sep-13-17 09:55	Sep-14-17 13:26
S14	1709003-14	Water	Sep-13-17 10:23	Sep-14-17 13:26
S15	1709003-15	Water	Sep-13-17 11:10	Sep-14-17 13:26
R16	1709003-16	Water	Sep-13-17 12:52	Sep-14-17 13:26

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Jan-30-18 17:16

Alkalinity by SM 2320B
US EPA Region 5 Chicago Regional Laboratory

S01 (1709003-01) **Matrix: Water** **Sampled: Sep-12-17 09:57** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	480			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S02 (1709003-02) **Matrix: Water** **Sampled: Sep-12-17 10:23** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	650			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S03 (1709003-03) **Matrix: Water** **Sampled: Sep-12-17 10:50** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	610			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S04 (1709003-04) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	540			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

D05 (1709003-05) **Matrix: Water** **Sampled: Sep-12-17 11:08** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	550			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S06 (1709003-06) **Matrix: Water** **Sampled: Sep-12-17 11:30** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	700			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S07 (1709003-07) **Matrix: Water** **Sampled: Sep-12-17 11:51** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	710			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

S08 (1709003-08) **Matrix: Water** **Sampled: Sep-12-17 14:03** **Received: Sep-14-17 13:26**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	55			20	mg CaCO3/L	1	B171022	Sep-19-17	Sep-19-17

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	Reported: Jan-30-18 17:16

Alkalinity by SM 2320B
US EPA Region 5 Chicago Regional Laboratory

S09 (1709003-09) Matrix: Water Sampled: Sep-12-17 15:00 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	380			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S10 (1709003-10) Matrix: Water Sampled: Sep-12-17 15:15 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	580			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S11 (1709003-11) Matrix: Water Sampled: Sep-13-17 09:44 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	470			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S12 (1709003-12) Matrix: Water Sampled: Sep-13-17 09:49 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	390			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S13 (1709003-13) Matrix: Water Sampled: Sep-13-17 09:55 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	470			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S14 (1709003-14) Matrix: Water Sampled: Sep-13-17 10:23 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	430			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

S15 (1709003-15) Matrix: Water Sampled: Sep-13-17 11:10 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	600			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

R16 (1709003-16) Matrix: Water Sampled: Sep-13-17 12:52 Received: Sep-14-17 13:26

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Total Alkalinity	0			20	mg CaCO3/L	1	B170022	Sep-19-17	Sep-19-17

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Water Division, WECA Region 5	Project: U.S. Steel - Minntac Tailings Basin Area Facility	
77 West Jackson Boulevard	Project Number: 02DM17	**Reported:**
Chicago IL, 60604	Project Manager: Dean Maraldo	Jan-30-18 17:16

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Phone:(312)353-8370 Fax:(312)886-2591

Water Division, WECA Region 5 77 West Jackson Boulevard Chicago IL, 60604	Project: U.S. Steel - Minntac Tailings Basin Area Facility Project Number: 02DM17 Project Manager: Dean Maraldo	**Reported:** Jan-30-18 17:16

Notes and Definitions

U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

ED_004744_00033652-00150

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

WC-15J

MEMORANDUM

SUBJECT: Close-out ACME Galvanizing Inspection Report

FROM: Donald R. Schwer III, Agricultural Engineer
Water Enforcement and Compliance Assurance Branch

THROUGH: Ryan Bahr, Section Chief
Section 2, Water Enforcement & Compliance Assurance Branch

TO: Patrick F. Kuefler, Branch Chief
Water Enforcement Compliance Assurance Branch

The purpose of this memo is to close the ACME Galvanizing Inspection Report. Significant Non-Compliance was not found at the ACME Galvanizing facility. I recommend sending this letter to inform the Milwaukee Metropolitan Sewerage District (MMSD) that ACME Galvanizing may be categorized incorrectly.

Recycled/Recyclable ● Printed with Vegetable Oil Based Inks on 100% Recycled Paper (100% Post Consumer)

ED_004744_00033652-00151