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LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING – COMPLETION REPORT

RAMBOLL Bright ideas. Sustainable change.

STATE-ONLY ENVIRONMENTALLY BENEFICIAL PROJECT LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING – COMPLETION REPORT

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1. EXECUTIVE SUMMARY

On August 30, 2021, the U.S. District Court for the Northern District of Indiana entered the Revised Consent Decree[1] between the United States of America, on behalf of the United States Environmental Protection Agency (EPA), the National Park Service of the United States Department of the Interior and the National Oceanic and Atmospheric Administration of the United States Department of Commerce; and the State of Indiana, on behalf of the Indiana Department of Environmental Management (IDEM) and the Indiana Department of Natural Resources and United States Steel Corporation (U. S. Steel) for issues related to U. S. Steel's Midwest Plant Facility in Portage, Indiana (Midwest Plant).

The Revised Consent Decree included a state-only environmentally beneficial project (EBP) that requires water quality testing and reporting from seven Indiana shore locations along Lake Michigan. The goal of the state EBP is to "contribute to significant public health benefits to the communities near the plant and to those who use the popular Indiana Dunes National Park for recreation" (USEPA, 2019[2]). Specifically, the objective of generating the Lake Michigan water quality data is to determine, at the locations sampled, whether the water quality is safe for recreational use.

The EBP began sampling in November 2021. This Final Report summarizes the EBP field collection program and analytical results for the entire program through November 2024, as required by the Consent Decree under Section VII. Paragraph 19. Completion Report. It is being submitted to IDEM with an information-only copy sent to EPA.

All seven Consent Decree-identified locations were sampled and all required parameters were measured or analyzed. Laboratory methods that detect low levels of total chromium and hexavalent chromium, at or below the detection limits required by the Consent Decree, were used. 2024 analytical results are summarized in Table 1 and previous years' results are provided in Appendix 1 and include non-detect and "J-qualified" results. Non-detect results, shown with a "less than" sign (<) followed by a number, indicate that the constituent was not detected at the displayed limit of detection (LOD) capable by the laboratory method. J-qualified values indicate that the constituent was detected above the LOD, but below the limit of quantitation (LOQ) for which the laboratory can reliably quantify the concentration. Therefore, the concentrations of total chromium and hexavalent chromium in some samples are estimated.

This Completion Report with the attached summary of expenditures and evidence of completion (Appendix 2) demonstrates that U. S. Steel Midwest has completed the State-Only EBP required under the Revised Consent Decree.

[1] USDC IN/ND case 2:18-cv-00127-TLS-JEM document 46-1 filed 11/20/19

[2] United States EPA (United States Environmental Protection Agency). 2019. U. S. Steel Corporation Consent Decree. Available on-line at: https://www.epa.gov/in/u-s-steel-corporation-consent-decree. Site last updated on March 18, 2024; site last accessed March 25, 2024.

2. FIELD SAMPLING

2.1 Locations

Water quality measurements and analytical laboratory samples were collected from seven locations along southern Lake Michigan in Indiana (Figure 1), as outlined in the Paragraph 14 of the Revised Consent Decree. The location codes are defined as:

a) Burns Ditch (BDXX)
b) Burns Ditch / Lake Michigan Mixing Zone (BDMZ)
c) Kemil Beach (KMXX)
d) Indiana Dunes Beach – Western Area (IDBW)
e) Michigan City (MCXX)
f) Vicinity of American Water Intake – Gary[3]
    - Lakeside Pump House (Winter Sampling) (AWGL)
    - Breakwall (Summer Sampling) (AWGB)
g) Vicinity of American Water Intake – Ogden (AMOG)

2.2 Methodology and Equipment

Surface water samples and water quality measurements were collected via grab sampling. Beach locations (KMXX, IDBW, MCXX, and AMOG) were sampled by dipping a jar into the water by hand, when accessible and safe. Samples were collected from the American Water Intake Breakwall (AWGB) using a rope and bottle. Surface water was collected from the Lakeside Pump House (AWGL) by lowering a sampling container into the intake forebay. The same approach was used to collect samples from the pier at Burns Ditch/Lake Michigan Mixing Zone (BDMZ) and the platform at the Burns Ditch location (BDXX) during the first event. To better collect well-mixed water from mid-depth, a sub-surface water sampler (e.g., Van Dorn bottle) was used to collect samples from these locations for the subsequent events.

2.3 Schedule

Per Section VII. Paragraph 15 of the Consent Decree, from May 1 through September 30, water quality sampling occurred weekly at MCXX, AWGL or AWGB, and AMOG, and twice weekly at BDXX, BDMZ, KMXX, and IDBW. Generally, all seven locations were sampled during the first event of the week, and BDXX, BDMZ, KMXX, and IDBW were resampled during the second event.

The EBP included events on:

- November 16, 2021
- December 2, 2021
- January 4, 2022
- February 23, 2022(1)
- March 21, 2022
- April 4, 2022
- May 2, 2022
- May 4, 2022
- May 9, 2022
- May 11, 2022
- May 13, 2022
- May 16, 2022
- May 18, 2022
- May 23, 2022(2)
- May 25, 2022
- May 31, 2022
- June 1, 2022
- June 6, 2022
- June 9, 2022
- June 14, 2022
- June 15, 2022
- June 20, 2022
- June 22, 2022
- June 27, 2022
- June 29, 2022
- July 5, 2022
- July 6, 2022
- July 11, 2022
- July 13, 2022
- July 18, 2022

[3] Samples are collected from the Lakeside Pump House intake forebay when the intake is not being chlorinated (typically the months of October/November through April/May). To avoid interference with the analyses of *E. coli* and cyanobacteria during chlorination, samples are collected from the west side of the slip breakwall when safe, during periods of intake chlorination.

- July 20, 2022
- July 25, 2022
- July 27, 2022
- August 1, 2022
- August 3, 2022
- August 8, 2022
- August 10, 2022
- August 15, 2022
- August 17, 2022
- August 22, 2022
- August 24, 2022
- August 29, 2022
- August 31, 2022
- September 6, 2022
- September 7, 2022
- September 14, 2022
- September 15, 2022
- September 19, 2022
- September 21, 2022
- September 29, 2022
- September 30, 2022
- October 3, 2022
- November 9, 2022[3]
- December 12, 2022
- January 9, 2023
- February 20, 2023
- March 15, 2023
- April 11, 2023
- May 2, 2023
- May 4, 2023
- May 8, 2023
- May 10, 2023
- May 15, 2023
- May 17, 2023
- May 22, 2023
- May 24, 2023
- May 30, 2023
- May 31, 2023
- June 5, 2023
- June 7, 2023
- June 13, 2023[2]
- June 14, 2023
- June 19, 2023
- June 21, 2023
- June 26, 2023
- June 28, 2023
- July 3, 2023
- July 4, 2023
- July 10, 2023
- July 12, 2023
- July 17, 2023
- July 19, 2023
- July 24, 2023
- July 26, 2023
- July 31, 2023
- August 2, 2023
- August 8, 2023
- August 9, 2023
- August 14, 2023
- August 16, 2023
- August 21, 2023
- August 23, 2023
- August 28, 2023
- August 29, 2023
- September 5, 2023
- September 6, 2023
- September 11, 2023
- September 13, 2023
- September 19, 2023
- September 20, 2023
- September 25, 2023
- September 27, 2023
- October 18, 2023
- November 14, 2023[3]
- December 7, 2023
- January 9, 2024
- February 9, 2024
- March 21 , 2024[4]
- April 9, 2024
- May 1, 2024[5]
- May 2, 2024
- May 6, 2024
- May 8, 2024
- May 13, 2024
- May 14, 2024
- May 20, 2024
- May 22, 2024
- May 28, 2024
- May 30, 2024
- June 3, 2024[2]
- June 5, 2024
- June 11, 2024
- June 12, 2024
- June 18, 2024
- June 19, 2024
- June 24, 2024
- June 26, 2024
- July 2, 2024
- July 3, 2024
- July 8, 2024
- July 9, 2024
- July 15, 2024
- July 17, 2024
- July 22, 2024
- July 24, 2024
- July 29, 2024
- July 31, 2024
- August 5, 2024
- August 8, 2024
- August 12, 2024
- August 14, 2024
- August 21, 2024
- August 22, 2024
- August 26, 2024
- August 28, 2024
- September 3, 2024
- September 4, 2024
- September 9, 2024
- September 11, 2024
- September 16, 2024
- September 18, 2024
- September 24, 2024
- September 25, 2024
- September 30, 2024
- October 2, 2024
- November 7, 2024[3][6]

Schedule Notes:

(1) Partial event. Beaches were checked weekly in February, but ice precluded safe sampling. During the last week of the month, samples were collected from Burns Ditch (BDXX), Burns Ditch / Lake Michigan Mixing Zone (BDMZ), and Lakeside Pump House (AWGL).

(2) U. S. Steel Gary Works began seasonal chlorination at the Lakeside Pump House; therefore, the Vicinity of American Water Intake – Gary sample location changed to the breakwall (AWGB).
(3) U. S. Steel Gary Works discontinued seasonal chlorination at the Lakeside Pump House; therefore, the Vicinity of American Water Intake – Gary sample location was changed back to the Lakeside Pump House (AWGL).
(4) Attempts were made to sample on March 6, March 11, March 18, and March 19, 2024, but safe access to the sampling sites was not possible until March 21, 2024.
(5) USGS changed the monitoring setup on the platform used to sample the Burns Waterway Upstream (BDXX) site. Therefore, a slight adjustment was made to the sampling location (previously from the end of the platform) for safety to avoid the open railing and inaccessibility of the end of the platform.
(6) This sampling event was performed to supplement the partial sampling event on February 23, 2022 that was limited due to ice conditions on the lake, as established in the November 2021 Sampling and Analysis Plan and Quality Assurance Project Plan (See Appendix 3). All locations were sampled and analyzed for completeness.

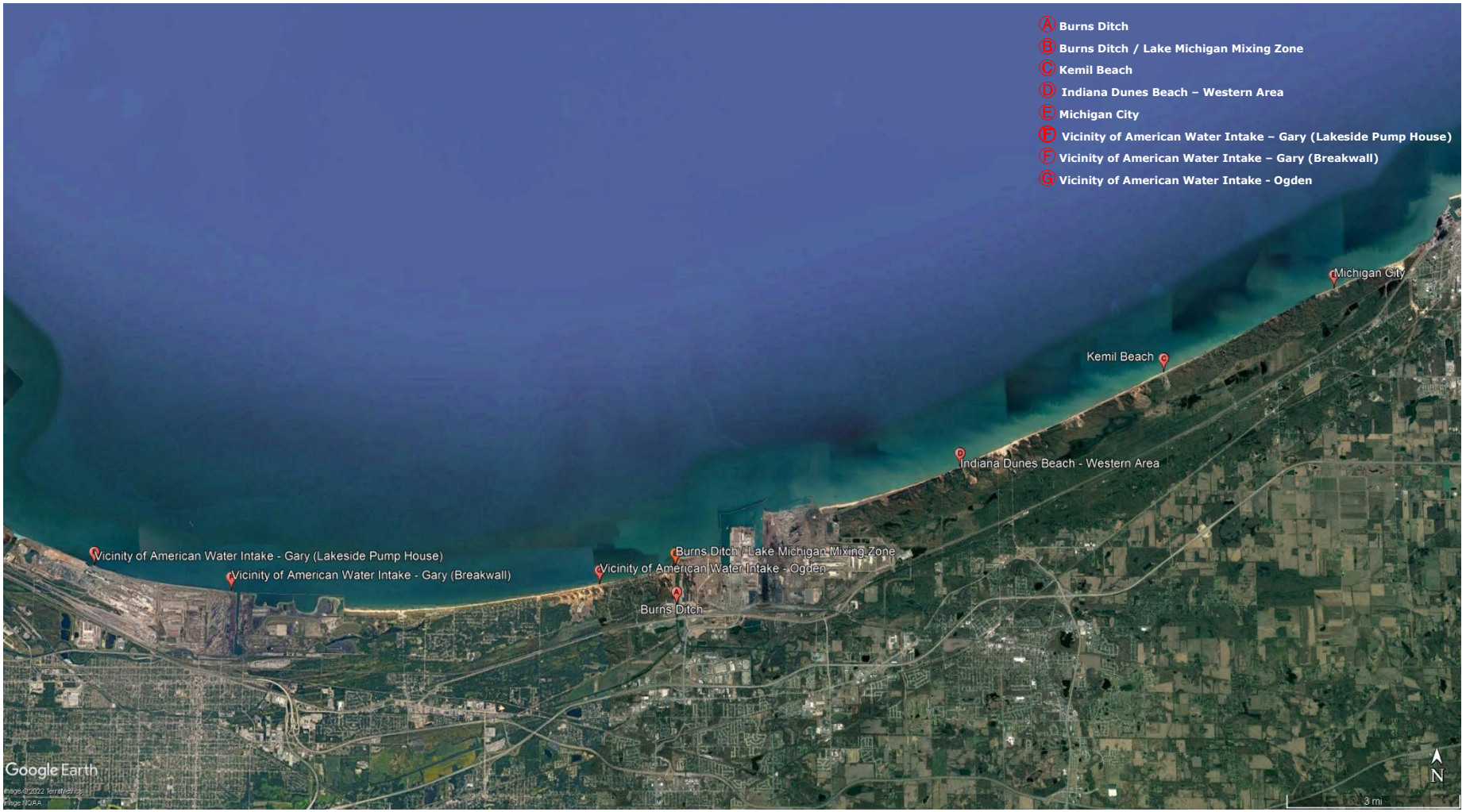

Figure 1. Sampling Locations along Southern Lake Michigan

3. WATER QUALITY PARAMETERS

Surface water collected from the sampling locations identified in Section 2.1 were measured or analyzed for the following parameters using the equipment and methodology described in the Revised Consent Decree:

i. hexavalent chromium
ii. total chromium
iii. cyanobacteria
iv. *E. coli*
v. pH
vi. total suspended solids (TSS)
vii. temperature
viii. turbidity (as an indication of transparency)

pH, temperature, and turbidity were measured and recorded in the field. Hexavalent chromium, total chromium, cyanobacteria, and TSS were analyzed by ALS Valparaiso, and *E. coli* was analyzed by Utility Services until May 13, 2022 when ALS Valparaiso began analyzing *E. coli*.

Only minor deviations from the Sampling and Analysis Plan and Quality Assurance Project Plan occurred in relation to laboratory analyses and reporting, including:

- A supply shortage of cyanobacteria testing equipment delayed reporting of the July 27, 2022 results until August 1, 2022 and the August 3, 2022 results until August 8, 2022. No samples were out of hold times.
- The field blank collected on September 21, 2022 had a detection of total chromium that was disproportionately high compared to the field sample and field duplicate. No issues were found in the prep batch or data entry, but the lab ran the field blank neat on the instrument (straight out of bottle with no digestion). The result was non-detect at 0.43 µg/L. The field blank was then re-digested and re-analyzed, resulting in a non-detect at 0.43 µg/L.
- A supply shortage of cyanobacteria testing equipment delayed reporting of the July 3 and July 4, 2023 results until July 10, 2023. No samples were out of hold times.
- Method detection limits (LODs) and reporting limits (LOQs) for some total chromium results from the July 8 and July 9, 2024 events were 0.61 µg/L and 5.0 µg/L, respectively because those samples were analyzed at the ALS Holland laboratory due to equipment issues at the ALS Valparaiso laboratory.
- September 9 and September 11, 2024 sampling results were reported on September 17, 2024 due to the hexavalent chromium analyzer being serviced.

Table 1 provides the results analytical results from 2024. Historical results from 2021 through 2023 are included in Appendix 1.

Table 1. 2024 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
MCXX	1/9/2024	13:21	25.8	25.4	37.8	7.80	<1.0	0.875 J	0.146	3.60
KMXX	1/9/2024	12:55	23.9	19.8	37.5	7.70	<1.0	0.986 J	0.139	4.60
IDBW	1/9/2024	12:30	19.7	16.0	36.8	7.60	<1.0	0.791 J	0.139	4.60
BDMZ	1/9/2024	11:58	9.69	8.40	41.7	7.70	<1.0	0.573 J	0.066	279
BDXX	1/9/2024	11:45	11.6	8.60	41.9	7.60	<1.0	0.662 J	0.0651	320
AMOG	1/9/2024	11:20	8.16	7.50	39.3	7.50	<1.0	0.669 J	0.118	10.8
AWGL	1/9/2024	10:22	5.30	5.70	43.5	7.40	<1.0	0.708 J	0.146	<1.0
AMOG_DP	1/9/2024	11:23	8.19	9.00	39.4	7.50	<1.0	0.600 J	0.127	39.8
AMOG_FB	1/9/2024	11:25	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0231 J	<1.0
MCXX	2/9/2024	13:45	5.50	3.70	41.3	7.70	<1.0	0.553 J	0.136	<1.0
KMXX	2/9/2024	13:20	6.10	4.10	42.6	7.60	<1.0	0.735 J	0.146	<1.0
IDBW	2/9/2024	13:00	5.70	5.60	42.0	7.50	<1.0	0.527 J	0.146	<1.0
BDMZ	2/9/2024	12:47	15.9	14.2	51.0	7.60	<1.0	0.885 J	0.0559	116
BDXX	2/9/2024	12:24	14.0	14.3	51.6	7.60	<1.0	0.962 J	0.0517	158
AMOG	2/9/2024	11:45	7.01	7.60	50.0	7.80	<1.0	0.532 J	0.117	<1.0
AWGL	2/9/2024	10:50	2.31	2.20	42.1	7.50	<1.0	0.559 J	0.148	<1.0
BDXX_DUP	2/9/2024	12:26	14.1	12.2	51.5	7.60	<1.0	0.868 J	0.0569	158
BDXX_FB	2/9/2024	12:27	N/A	<0.302	N/A	N/A	<1.0	<0.433	0.0157 J	<1.0
MCXX	3/21/2024	13:40	22.7	20.7	44.2	7.76	<1.0	0.915 J	0.177	2.00
KMXX	3/21/2024	13:05	24.5	21.3	41.9	7.82	<1.0	0.911 J	0.146	3.60
IDBW	3/21/2024	12:33	19.7	21.8	42.6	7.65	<1.0	0.914 J	0.145	<1.0
BDMZ	3/21/2024	11:55	13.7	9.90	47.6	7.60	<1.0	0.724 J	0.0991	72.9
BDXX	3/21/2024	11:33	13.8	11.9	47.6	7.60	<1.0	0.717 J	0.111	61.1
AMOG	3/21/2024	11:08	12.6	11.3	43.3	7.60	<1.0	0.935 J	0.147	14.0
AWGL	3/21/2024	10:13	7.90	8.80	42.0	7.30	<1.0	0.683 J	0.145	1.00
BDMZ_DUP	3/21/2024	12:00	13.0	9.60	46.9	7.70	<1.0	0.809 J	0.0956	75.8
BDMZ_FB	3/21/2024	12:05	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0378	<1.0

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
MCXX	4/9/2024	13:00	8.40	5.48	58.6	7.92	<1.0	0.504 J	0.192	<1.0
KMXX	4/9/2024	12:25	10.2	6.50	60.0	7.60	<1.0	0.885 J	0.166	<1.0
IDBW	4/9/2024	11:50	11.8	6.50	59.1	7.91	<1.0	0.704 J	0.196	<1.0
BDMZ	4/9/2024	11:10	27.0	19.3	58.8	7.60	<1.0	1.99 J	0.0765	163
BDXX	4/9/2024	10:56	26.9	19.9	55.5	7.60	<1.0	1.92 J	0.0729	126
AMOG	4/9/2024	10:32	10.3	9.30	57.8	7.90	<1.0	0.975 J	0.143	9.60
AWGL	4/9/2024	9:55	5.72	4.80	46.9	7.71	<1.0	0.452 J	0.149	<1.0
IDBW_DUP	4/9/2024	12:00	11.8	5.70	59.2	7.94	<1.0	0.730 J	0.170	1.00
IDBW_FB	4/9/2024	12:05	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0275 J	<1.0
MCXX	5/1/2024	6:58	5.03	5.40	56.3	7.54	<1.0	0.783 J	0.206	3.00
KMXX	5/1/2024	7:20	4.44	2.70	55.6	7.49	<1.0	0.478 J	0.175	4.05
IDBW	5/1/2024	7:54	4.81	4.70	55.9	7.33	<1.0	0.583 J	0.157	2.00
BDMZ	5/1/2024	9:15	23.8	20.1	66.7	7.95	<1.0	1.50 J	0.0928	411
BDXX	5/1/2024	9:45	26.9	23.3	67.1	8.21	<1.0	1.58 J	0.0812	451
AMOG	5/1/2024	8:42	2.87	2.30	56.4	7.87	<1.0	0.494 J	0.174	1.00
AWGL	5/1/2024	8:25	2.66	1.12 J	51.1	7.90	<1.0	0.533 J	0.191	<1.0
KMXX_DUP	5/1/2024	7:20	4.39	3.50	55.7	7.51	<1.0	0.523 J	0.180	3.05
KMXX_FB	5/1/2024	7:20	N/A	<0.300	N/A	N/A	<1.0	0.446 J	<0.0130	<1.0
KMXX	5/2/2024	7:02	3.13	2.40	53.1	7.71	<1.0	0.583 J	0.153	1.00
IDBW	5/2/2024	7:33	2.51	2.30	52.5	7.84	<1.0	0.559 J	0.155	2.50
BDMZ	5/2/2024	8:05	19.8	16.1	67.6	8.34	<1.0	1.12 J	0.0558	188
BDXX	5/2/2024	8:23	16.7	17.7	66.3	8.27	<1.0	1.19 J	0.0372	214
BDXX_DUP	5/2/2024	8:23	17.1	17.6	66.5	8.23	<1.0	1.09 J	0.0472	184
BDXX_FB	5/2/2024	8:23	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	5/6/2024	13:30	16.1	13.7	61.7	8.10	<1.0	0.828 J	0.0918	15.8
KMXX	5/6/2024	12:54	11.0	9.00	58.2	8.00	<1.0	0.831 J	0.111	16.4
IDBW	5/6/2024	12:25	5.63	5.30	59.0	8.10	<1.0	0.591 J	0.119	13.9
BDMZ	5/6/2024	11:38	20.9	20.4	64.5	7.75	<1.0	1.31 J	0.0239 J	170

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDXX	5/6/2024	11:20	20.9	18.9	65.3	7.80	<1.0	1.41 J	0.0512	204
AMOG	5/6/2024	11:00	14.6	14.8	61.3	8.10	<1.0	0.870 J	0.109	44.2
AWGL	5/6/2024	10:00	8.20	3.00	57.2	8.10	<1.0	0.485 J	0.127	<1.0
MCXX_DUP	5/6/2024	13:34	16.1	13.0	61.8	8.10	<1.0	0.754 J	0.102	23.8
MCXX_FB	5/6/2024	13:37	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	5/8/2024	6:55	3.47	5.60	53.2	8.13	<1.0	0.560 J	0.111	4.60
IDBW	5/8/2024	7:18	2.80	5.20	53.4	8.26	<1.0	0.711 J	0.122	10.2
BDMZ	5/8/2024	7:58	21.8	26.5	64.7	7.87	<1.0	1.47 J	0.0598	662
BDXX	5/8/2024	8:15	23.5	24.8	65.3	7.79	<1.0	1.02 J	0.0263 J	645
BDMZ_DUP	5/8/2024	7:58	21.8	26.2	64.4	7.91	<1.0	1.37 J	0.0442	642
BDMZ_FB	5/8/2024	7:58	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	5/13/2024	6:55	5.31	5.20	57.6	8.17	<1.0	0.579 J	0.150	13.4
KMXX	5/13/2024	7:20	6.04	3.40	57.9	7.97	<1.0	0.632 J	0.149	21.6
IDBW	5/13/2024	7:45	5.87	4.30	58.3	8.23	<1.0	0.537 J	0.167	11.2
BDMZ	5/13/2024	9:02	14.6	20.7	68.1	7.76	<1.0	0.948 J	0.0456	158
BDXX	5/13/2024	9:20	25.2	17.6	69.0	7.80	<1.0	1.08 J	0.0552	247
AMOG	5/13/2024	8:35	2.34	2.20	59.3	8.07	<1.0	0.484 J	0.143	<1.0
AWGL	5/13/2024	10:15	3.50	1.60 J	59.4	8.10	<1.0	0.507 J	0.144	<1.0
AWGL_DUP	5/13/2024	10:20	3.50	2.80	59.3	8.10	<1.0	0.463 J	0.155	<1.0
AWGL_FB	5/13/2024	10:30	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	5/14/2024	12:31	6.40	14.3	61.0	8.10	<1.0	0.600 J	0.144	261
IDBW	5/14/2024	11:55	4.40	3.30	59.9	8.10	<1.0	0.487 J	0.140	2.00
BDMZ	5/14/2024	11:13	15.2	15.9	67.1	7.60	<1.0	0.923 J	0.0177 J	140
BDXX	5/14/2024	10:48	19.4	21.1	69.1	7.70	<1.0	1.05 J	0.0275 J	168
IDBW_DUP	5/14/2024	12:01	4.40	4.10	60.0	8.10	<1.0	0.660 J	0.143	1.50
IDBW_FB	5/14/2024	12:06	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	5/20/2024	7:30	5.31	4.20	62.2	7.78	<1.0	0.553 J	0.136	4.15
KMXX	5/20/2024	7:55	4.05	2.80	61.7	7.65	<1.0	0.506 J	0.134	4.05

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
IDBW	5/20/2024	8:23	6.58	4.90	62.4	7.72	<1.0	0.576 J	0.118	4.55
BDMZ	5/20/2024	9:35	11.4	15.6	73.4	7.94	<1.0	0.937 J	0.0456	49.7
BDXX	5/20/2024	9:50	12.1	16.1	73.4	7.96	<1.0	0.727 J	0.0301 J	71.0
AMOG	5/20/2024	9:05	3.05	5.40	63.6	8.21	<1.0	0.543 J	0.132	4.10
AWGL	5/20/2024	10:00	1.81	1.70 J	61.0	7.92	<1.0	0.560 J	0.142	<1.0
AMOG_DUP	5/20/2024	9:05	2.99	4.70	63.5	8.20	<1.0	0.576 J	0.130	4.60
AMOG_FB	5/20/2024	9:05	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	5/22/2024	7:18	7.19	14.3	63.8	7.67	<1.0	0.582 J	0.152	21.5
IDBW	5/22/2024	7:55	7.76	13.9	64.2	8.02	<1.0	0.602 J	0.139	15.6
BDMZ	5/22/2024	8:35	21.0	23.3	71.6	7.82	<1.0	1.03 J	0.0450	197
BDXX	5/22/2024	8:52	13.0	15.6	72.8	7.73	<1.0	0.758 J	0.0403	162
KMXX_DUP	5/22/2024	7:18	7.13	11.0	63.8	7.73	<1.0	0.658 J	0.152	16.4
KMXX_FB	5/22/2024	7:18	N/A	0.600 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	5/28/2024	13:24	7.92	8.60	66.5	8.10	<1.0	0.441 J	0.143	5.15
KMXX	5/28/2024	12:22	5.52	7.10	64.0	8.10	<1.0	0.453 J	0.143	3.60
IDBW	5/28/2024	11:52	3.69	3.80	64.5	8.20	<1.0	0.503 J	0.136	3.60
BDMZ	5/28/2024	10:20	9.90	9.80	70.7	7.94	<1.0	0.569 J	0.0286 J	46.7
BDXX	5/28/2024	10:40	12.5	13.8	71.9	7.88	<1.0	0.778 J	0.0150 J	71.5
AMOG	5/28/2024	11:05	5.53	6.89	62.6	8.14	<1.0	0.503 J	0.135	6.70
AWGL	5/28/2024	9:20	2.53	2.10	61.3	8.08	<1.0	0.525 J	0.131	<1.0
BDXX_DUP	5/28/2024	10:40	13.0	13.9	72.8	7.89	<1.0	0.888 J	0.0167 J	80.0
BDXX_FB	5/28/2024	10:41	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	5/30/2024	11:36	18.7	15.3	62.2	8.23	<1.0	0.658 J	0.129	5.20
IDBW	5/30/2024	11:09	14.3	12.0	65.6	8.30	<1.0	0.754 J	0.126	9.00
BDMZ	5/30/2024	10:12	12.6	14.1	67.6	7.94	<1.0	1.24 J	0.0350	251
BDXX	5/30/2024	10:40	20.6	25.8	68.5	7.93	<1.0	1.38 J	0.0191 J	191
BDXX_DUP	5/30/2024	10:40	20.4	23.4	68.7	7.96	<1.0	1.57 J	0.0195 J	237
BDXX_FB	5/30/2024	10:40	N/A	0.600 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
MCXX	6/3/2024	12:12	4.10	1.30 J	66.9	8.19	<1.0	0.456 J	0.145	<1.0
KMXX	6/3/2024	11:45	2.37	1.50 J	66.0	8.12	<1.0	0.470 J	0.145	<1.0
IDBW	6/3/2024	11:25	3.23	3.50	66.4	8.24	<1.0	0.597 J	0.146	1.50
BDMZ	6/3/2024	10:15	11.7	13.9	72.6	7.77	<1.0	1.12 J	0.107	54.0
BDXX	6/3/2024	10:35	13.4	16.3	72.9	7.85	<1.0	0.695 J	0.0318 J	84.1
AMOG	6/3/2024	11:00	2.30	2.00	65.6	8.29	<1.0	<0.433	0.143	<1.0
AWGB	6/3/2024	9:20	1.09	1.30 J	65.8	8.55	<1.0	0.490 J	0.148	1.00
BDMZ_DUP	6/3/2024	10:15	11.5	14.4	72.5	7.74	<1.0	1.10 J	0.104	49.6
BDMZ_FB	6/3/2024	10:15	N/A	0.30 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	6/5/2024	6:48	2.70	2.30	65.4	7.85	<1.0	0.486 J	0.145	2.00
IDBW	6/5/2024	7:23	2.98	4.20	66.5	7.92	<1.0	0.534 J	0.142	1.00
BDMZ	6/5/2024	8:05	10.5	14.2	73.9	8.02	<1.0	0.917 J	0.0896	246
BDXX	6/5/2024	8:28	11.6	12.9	74.3	8.09	<1.0	0.944 J	0.0322 J	138
BDMZ_DUP	6/5/2024	8:05	10.3	13.1	73.8	7.97	<1.0	0.770 J	0.0885	359
BDMZ_FB	6/5/2024	8:05	N/A	<0.522	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	6/11/2024	13:16	26.6	18.5	72.3	8.20	<1.0	0.814 J	0.134	5.15
KMXX	6/11/2024	12:44	10.2	8.20	69.0	8.10	<1.0	0.839 J	0.128	2.55
IDBW	6/11/2024	12:06	12.8	10.4	70.1	8.00	<1.0	0.624 J	0.133	1.00
BDMZ	6/11/2024	11:35	7.39	9.50	74.1	8.10	<1.0	1.09 J	0.0444	33.3
BDXX	6/11/2024	11:22	9.32	12.8	75.0	8.05	<1.0	0.575 J	0.0291 J	42.6
AMOG	6/11/2024	11:03	7.51	12.1	68.0	8.30	<1.0	0.590 J	0.107	10.1
AWGB	6/11/2024	10:05	2.89	2.40	68.1	8.40	<1.0	0.694 J	0.148	<1.0
IDBW_DUP	6/11/2024	12:10	12.9	10.3	70.1	8.00	<1.0	0.541 J	0.127	1.00
IDBW_FB	6/11/2024	12:10	N/A	0.400 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	6/12/2024	7:17	5.43	4.60	64.5	7.91	<1.0	0.729 J	0.131	1.50
IDBW	6/12/2024	7:40	4.84	4.40	65.1	8.02	<1.0	0.584 J	0.137	<1.0
BDMZ	6/12/2024	8:25	10.3	12.5	71.9	7.89	<1.0	0.847 J	0.0430	27.0
BDXX	6/12/2024	8:50	10.3	10.6	72.1	8.04	<1.0	0.878 J	0.0392	66.9

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
IDBW_DUP	6/12/2024	7:40	5.03	3.60	64.9	7.99	<1.0	0.601 J	0.132	3.10
IDBW_FB	6/12/2024	7:40	N/A	0.400 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	6/18/2024	6:50	2.02	0.900 J	70.1	7.68	<1.0	0.483 J	0.140	2.00
KMXX	6/18/2024	7:22	1.41	1.30 J	69.6	7.75	<1.0	0.537 J	0.140	2.00
IDBW	6/18/2024	7:50	1.88	1.30 J	69.1	7.76	<1.0	0.594 J	0.140	4.10
BDMZ	6/18/2024	9:00	9.79	11.1	77.5	7.52	<1.0	0.817 J	0.0790	32.2
BDXX	6/18/2024	9:17	7.55	9.00	78.8	7.56	<1.0	0.899 J	0.0540	81.0
AMOG	6/18/2024	8:35	3.46	0.800 J	68.7	7.83	<1.0	0.593 J	0.160	<1.0
AWGB	6/18/2024	10:30	1.97	0.800 J	69.2	8.32	<1.0	0.789 J	0.210	1.00
KMXX_DUP	6/18/2024	7:22	1.54	1.50 J	69.5	7.79	<1.0	0.869 J	0.140	3.10
KMXX_FB	6/18/2024	7:22	N/A	0.300 J	N/A	N/A	<1.0	0.456 J	<0.0130	<1.0
KMXX	6/19/2024	7:18	2.74	2.20	70.9	7.92	<1.0	0.494 J	0.160	<1.0
IDBW	6/19/2024	7:45	2.11	1.10 J	71.9	7.82	<1.0	<0.433	0.140	3.10
BDMZ	6/19/2024	8:25	10.6	13.1	78.2	7.64	<1.0	0.803 J	0.190	46.8
BDXX	6/19/2024	8:40	7.82	9.80	78.8	7.79	<1.0	0.628 J	0.0660	84.2
KMXX_DUP	6/19/2024	7:18	2.68	2.10	71.0	7.90	<1.0	0.553 J	0.160	1.00
KMXX_FB	6/19/2024	7:18	N/A	0.400 J	N/A	N/A	<1.0	0.726 J	0.0220	<1.0
MCXX	6/24/2024	6:28	12.7	18.2	68.7	7.74	<1.0	0.845 J	0.126	74.8
KMXX	6/24/2024	6:55	5.97	5.70	69.2	7.78	<1.0	0.519 J	0.132	8.60
IDBW	6/24/2024	7:40	2.34	4.50	69.0	7.84	<1.0	0.667 J	0.130	1.00
BDMZ	6/24/2024	8:55	15.7	15.0	76.8	7.37	<1.0	1.40 J	0.0268 J	2,040
BDXX	6/24/2024	9:15	14.3	15.0	79.2	7.56	<1.0	0.862 J	0.0268 J	4,880
AMOG	6/24/2024	8:30	2.47	2.30	68.8	7.89	<1.0	0.493 J	0.145	6.45
AWGB	6/24/2024	10:30	1.68	1.50 J	73.4	8.03	<1.0	0.622 J	0.149	5.75
MCXX_DUP	6/24/2024	6:28	12.4	16.9	68.8	7.71	<1.0	0.762 J	0.128	43.1
MCXX_FB	6/24/2024	6:28	N/A	<0.300	N/A	N/A	<1.0	<0.430	<0.0130	<1.0
KMXX	6/26/2024	11:40	2.61	3.90	71.6	8.28	<1.0	0.763 J	0.137	<1.0
IDBW	6/26/2024	11:16	2.48	3.70	71.9	8.25	<1.0	0.688 J	0.134	6.20

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	6/26/2024	10:35	7.13	10.4	76.4	7.90	<1.0	0.917 J	0.0299 J	274
BDXX	6/26/2024	10:10	8.43	11.8	76.1	7.90	<1.0	0.956 J	0.0225 J	388
BDXX_DUP	6/26/2024	10:15	7.97	11.0	75.7	7.90	<1.0	0.874 J	0.0247 J	303
BDXX_FB	6/26/2024	10:15	N/A	<0.300	N/A	N/A	<1.0	<0.430	<0.0130	<1.0
MCXX	7/2/2024	6:05	3.61	3.80	67.9	7.75	<1.0	0.467 J	0.151	3.05
KMXX	7/2/2024	6:30	2.94	3.70	67.1	7.84	<1.0	0.584 J	0.142	3.10
IDBW	7/2/2024	6:55	2.48	3.50	67.5	7.80	<1.0	0.566 J	0.140	2.00
BDMZ	7/2/2024	7:45	4.30	8.20	73.4	7.65	<1.0	1.06 J	0.325	88.8
BDXX	7/2/2024	8:00	6.51	10.6	74.4	7.73	<1.0	0.599 J	0.0727	67.2
AMOG	7/2/2024	7:20	4.12	5.40	66.5	7.77	<1.0	0.581 J	0.141	1.50
AWGB	7/2/2024	8:38	1.19	1.70 J	68.1	8.07	<1.0	0.507 J	0.164	<1.0
AWGB_DUP	7/2/2024	8:38	1.23	1.80 J	68.2	8.05	<1.0	0.614 J	0.161	1.50
AWGB_FB	7/2/2024	8:38	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	7/3/2024	6:40	2.88	2.40	70.1	7.83	<1.0	0.473 J	0.144	1.00
IDBW	7/3/2024	7:10	2.11	2.60	69.6	7.98	<1.0	0.451 J	0.148	10.2
BDMZ	7/3/2024	7:29	6.64	9.30	74.3	7.67	<1.0	0.895 J	0.137	50.1
BDXX	7/3/2024	7:55	5.46	8.70	74.4	7.58	<1.0	0.628 J	0.0527	107
BDMZ_DUP	7/3/2024	7:29	6.71	9.00	74.3	7.65	<1.0	0.687 J	0.130	72.5
BDMZ_FB	7/3/2024	7:29	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	7/8/2024	6:45	2.87	2.30	70.4	7.81	<1.0	0.564 J	0.142	1.00
KMXX	7/8/2024	7:08	1.97	1.80 J	69.9	7.62	<1.0	0.568 J	0.146	2.50
IDBW	7/8/2024	7:37	3.04	3.10	69.4	7.77	<1.0	0.619 J	0.146	1.55
BDMZ	7/8/2024	8:32	4.69	7.50	76.6	7.89	<1.0	0.816 J	0.0693	78.6
BDXX	7/8/2024	8:44	5.41	9.10	77.7	7.97	<1.0	0.714 J	0.0564	100
AMOG	7/8/2024	8:09	1.87	2.35	70.3	7.78	<1.0	<0.610	0.139	3.05
AWGB	7/8/2024	9:14	3.01	5.90	73.9	7.94	<1.0	2.03 J	0.150	1.00
AMOG_DUP	7/8/2024	8:09	1.92	2.63	70.2	7.80	<1.0	0.592 J	0.137	<1.0
AMOG_FB	7/8/2024	8:09	N/A	0.300 J	N/A	N/A	<1.0	<0.610	<0.0130	<1.0

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
KMXX	7/9/2024	11:26	1.50	1.00 J	74.5	7.82	<1.0	<0.610	0.146	1.00
IDBW	7/9/2024	11:55	2.60	0.900 J	73.9	7.73	<1.0	0.673 J	0.141	<1.0
BDMZ	7/9/2024	12:40	5.51	7.10	78.2	7.76	<1.0	0.876 J	0.0595	60.5
BDXX	7/9/2024	12:52	7.78	8.90	78.0	7.79	<1.0	0.953 J	0.0482	156
IDBW_DUP	7/9/2024	11:55	2.40	1.20 J	74.0	7.75	<1.0	0.650 J	0.140	1.00
IDBW_FB	7/9/2024	11:55	N/A	<0.300	N/A	N/A	<1.0	<0.610	<0.0130	<1.0
MCXX	7/15/2024	7:20	2.41	3.50	72.5	7.90	<1.0	<0.433	0.136	1.00
KMXX	7/15/2024	7:47	1.91	3.00	72.1	7.90	<1.0	0.533 J	0.138	4.6-
IDBW	7/15/2024	8:30	1.35	3.20	74.0	7.90	<1.0	0.551 J	0.134	<1.0
BDMZ	7/15/2024	9:10	21.3	24.6	78.2	7.40	<1.0	1.33 J	0.0417	1,210
BDXX	7/15/2024	9:43	34.4	31.7	79.8	7.50	<1.0	1.09 J	0.0457	1,860
AMOG	7/15/2024	10:15	1.71	1.90 J	75.9	8.00	<1.0	0.456 J	0.135	5.05
AWGB	7/15/2024	11:14	<1.0	1.10 J	78.9	8.20	<1.0	0.651 J	0.139	<1.0
BDXX_DUP	7/15/2024	9:50	31.6	30.2	80.4	7.60	<1.0	1.18 J	0.0491	1,210
BDXX_FB	7/15/2024	9:55	N/A	<0.300	N/A	N/A	<1.0	0.439 J	<0.0130	<1.0
KMXX	7/17/2024	6:48	4.06	8.22	73.9	7.91	<1.0	0.643 J	0.136	15.8
IDBW	7/17/2024	7:20	3.35	3.60	74.1	7.85	<1.0	0.491 J	0.132	12.2
BDMZ	7/17/2024	7:55	10.5	11.8	77.3	7.31	<1.0	0.908 J	0.0325 J	388
BDXX	7/17/2024	8:25	10.9	13.6	78.0	7.21	<1.0	1.08 J	0.0344 J	835
KMXX_DUP	7/17/2024	6:48	4.01	8.44	74.1	7.88	<1.0	0.553 J	0.127	13.0
KMXX_FB	7/17/2024	6:48	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	7/22/2024	7:08	8.40	9.10	70.9	7.83	<1.0	0.618 J	0.140	21.0
KMXX	7/22/2024	7:33	11.9	1.60 J	71.4	7.77	<1.0	0.477 J	0.138	3.05
IDBW	7/22/2024	7:55	10.0	8.40	71.8	7.91	<1.0	0.565 J	0.135	22.4
BDMZ	7/22/2024	9:05	8.94	8.60	78.7	7.67	<1.0	1.26 J	0.0673	71.5
BDXX	7/22/2024	8:44	11.1	12.9	76.1	7.51	<1.0	0.725 J	0.0466	170
AMOG	7/22/2024	8:27	10.0	5.80	72.1	7.90	<1.0	0.644 J	0.130	15.5
AWGB	7/22/2024	8:20	4.63	1.30 J	74.0	8.31	<1.0	0.485 J	0.141	2.00

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ_DUP	7/22/2024	9:05	9.12	8.00	78.6	7.71	<1.0	1.04 J	0.0655	112
BDMZ_FB	7/22/2024	9:05	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	7/24/2024	6:50	9.17	8.80	72.7	7.58	<1.0	0.691 J	0.147	18.9
IDBW	7/24/2024	7:18	8.08	9.50	73.0	7.73	<1.0	0.581 J	0.145	19.0
BDMZ	7/24/2024	7:45	7.62	7.40	78.0	7.66	<1.0	0.845 J	0.0555	97.0
BDXX	7/24/2024	8:05	8.96	9.70	76.3	7.52	<1.0	0.559 J	0.0465	139
BDXX_DUP	7/24/2024	8:05	9.01	9.50	76.2	7.54	<1.0	0.503 J	0.0459	127
BDXX_FB	7/24/2024	8:05	N/A	0.600 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	7/29/2024	6:00	1.82	1.30 J	73.1	7.87	<1.0	0.621 J	0.151	7.30
KMXX	7/29/2024	6:25	1.50	1.30 J	73.4	7.93	<1.0	0.441 J	0.148	5.90
IDBW	7/29/2024	6:55	2.03	1.40 J	74.0	7.81	<1.0	<0.433	0.145	2.00
BDMZ	7/29/2024	8:00	7.82	7.30	79.3	7.67	<1.0	1.14 J	0.0892	34.6
BDXX	7/29/2024	8:15	6.29	6.20	78.2	7.67	<1.0	0.911 J	0.0821	87.3
AMOG	7/29/2024	7:30	1.17	1.00 J	73.2	8.11	<1.0	0.509 J	0.152	5.60
AWGB	7/29/2024	8:54	1.93	1.40 J	74.1	8.10	<1.0	0.629 J	0.184	2.00
IDBW_DUP	7/29/2024	6:55	2.05	1.30 J	74.1	7.77	<1.0	<0.433	0.155	5.15
IDBW_FB	7/29/2024	6:55	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	7/31/2024	6:10	1.83	2.90	73.2	7.80	<1.0	0.672 J	0.149	5.65
IDBW	7/31/2024	6:48	2.10	5.00	73.9	8.00	<1.0	0.729 J	0.147	2.00
BDMZ	7/31/2024	8:16	6.20	5.90	80.4	7.80	<1.0	0.818 J	0.0713	50.1
BDXX	7/31/2024	8:30	7.50	9.60	79.1	7.70	<1.0	0.541 J	0.0583	36.0
BDMZ_DUP	7/31/2024	8:16	6.30	6.50	80.5	7.80	<1.0	1.15 J	0.0705	33.7
BDMZ_FB	7/31/2024	8:20	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	8/5/2024	6:05	1.40	1.40 J	75.5	8.10	<1.0	<0.433	0.154	2.00
KMXX	8/5/2024	6:47	1.32	1.30 J	75.0	8.20	<1.0	0.587 J	0.146	3.00
IDBW	8/5/2024	7:21	2.00	2.80	75.0	7.85	<1.0	0.527 J	0.149	4.70
BDMZ	8/5/2024	11:10	6.00	6.70	82.0	8.00	<1.0	0.886 J	0.207	812
BDXX	8/5/2024	11:30	9.20	10.3	82.9	8.00	<1.0	0.739 J	0.0677	302

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	8/5/2024	10:40	2.10	5.50	76.8	8.20	<1.0	0.505 J	0.150	8.95
AWGB	8/5/2024	10:30	1.75	0.900 J	78.9	8.30	<1.0	0.661 J	0.172	122
KMXX_DUP	8/5/2024	6:49	1.33	1.00 J	75.1	8.20	<1.0	0.804 J	0.145	<1.0
KMXX_FB	8/5/2024	6:49	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	8/8/2024	6:25	15.3	17.0	70.5	7.80	<1.0	0.821 J	0.132	14.6
IDBW	8/8/2024	7:00	15.6	12.4	71.2	8.10	<1.0	0.666 J	0.128	21.8
BDMZ	8/8/2024	8:00	11.3	6.40	80.1	7.80	<1.0	0.840 J	0.0504	38.2
BDXX	8/8/2024	8:25	13.3	8.20	78.0	7.90	<1.0	0.563 J	0.0444	95.7
IDBW_DUP	8/8/2024	7:10	15.5	10.4	71.1	8.10	<1.0	0.719 J	0.132	21.4
IDBW_FB	8/8/2024	7:10	N/A	0.400 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	8/12/2024	13:40	8.17	11.8	78.0	8.03	<1.0	0.719 J	<0.0470	10.5
KMXX	8/12/2024	13:10	10.3	12.1	77.3	8.14	<1.0	0.708 J	<0.0470	9.40
IDBW	8/12/2024	12:40	9.76	12.2	77.7	7.94	<1.0	0.827 J	<0.0470	10.6
BDMZ	8/12/2024	11:30	6.82	7.10	80.0	8.06	<1.0	0.852 J	<0.0072	80.5
BDXX	8/12/2024	11:50	7.27	6.00	79.7	7.97	<1.0	0.685 J	<0.0072	128
AMOG	8/12/2024	12:15	7.31	36.3	76.6	8.11	<1.0	1.16 J	<0.0470	197
AWGB	8/12/2024	10:20	1.81	1.80 J	75.9	8.06	<1.0	0.761 J	<0.0470	753
MCXX_DUP	8/12/2024	13:40	8.41	11.2	77.9	8.05	<1.0	0.697 J	<0.0470	9.45
MCXX4_FB	8/12/2024	13:40	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0072	<1.0
KMXX	8/14/2024	7:20	2.10	1.80 J	70.8	8.10	<1.0	<0.433	<0.0470	<1.0
IDBW	8/14/2024	8:00	1.89	1.30 J	72.8	7.90	<1.0	<0.433	<0.0470	2.00
BDMZ	8/14/2024	8:31	8.10	7.20	76.4	7.80	<1.0	0.523 J	<0.0072	75.0
BDXX	8/14/2024	6:25	8.71	7.00	75.5	7.70	<1.0	0.572 J	<0.0072	54.2
KMXX_DUP	8/14/2024	7:30	2.20	1.50 J	70.8	8.10	<1.0	<0.433	<0.0470	3.05
KMXX_FB	8/14/2024	7:30	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0072	<1.0
MCXX	8/21/2024	7:10	31.4	34.1	66.3	7.61	<1.0	0.928 J	0.106	23.9
KMXX	8/21/2024	7:40	35.6	32.2	66.7	7.48	<1.0	7.74	0.124	20.8
IDBW	8/21/2024	8:05	27.7	36.1	65.8	7.54	<1.0	1.02 J	0.131	39.5

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	8/21/2024	11:30	8.01	7.30	78.6	7.66	<1.0	0.882 J	0.0588	16.9
BDXX	8/21/2024	11:11	9.03	7.40	76.1	7.39	<1.0	0.724 J	0.0628	88.4
AMOG	8/21/2024	10:50	6.87	9.00	72.5	7.81	<1.0	0.576 J	0.133	41.6
AWGB	8/21/2024	8:35	3.82	3.20	72.1	8.00	<1.0	0.558 J	0.129	<1.0
AWGB_DUP	8/21/2024	8:35	3.82	2.40	72.1	8.00	<1.0	0.622 J	0.129	<1.0
AWGB_FB	8/21/2024	8:35	N/A	<0.300	N/A	N/A	<1.0	0.623 J	<0.0130	<1.0
KMXX	8/22/2024	7:10	5.46	5.70	69.1	7.74	<1.0	0.652 J	0.128	10.6
IDBW	8/22/2024	7:40	8.00	4.50	68.3	7.62	<1.0	0.497 J	0.130	4.05
BDMZ	8/22/2024	8:30	7.15	7.40	74.4	7.68	<1.0	0.855 J	0.0549	58.4
BDXX	8/22/2024	8:50	10.3	7.80	73.2	7.56	<1.0	0.695 J	0.0551	57.5
BDXX_DUP	8/22/2024	8:50	10.1	8.60	73.2	7.58	<1.0	0.955 J	0.0510	67.8
BDXX_FB	8/22/2024	8:50	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	8/26/2024	6:50	2.41	1.70 J	73.8	7.67	<1.0	0.554 J	0.141	23.1
KMXX	8/26/2024	7:26	1.97	1.50 J	73.2	7.59	<1.0	0.465 J	0.138	<1.0
IDBW	8/26/2024	8:00	3.12	1.40 J	74.1	7.91	<1.0	0.562 J	0.140	3.05
BDMZ	8/26/2024	8:50	6.60	6.80	79.7	7.53	<1.0	0.807 J	0.0593	57.2
BDXX	8/26/2024	9:03	7.93	6.80	79.3	7.64	<1.0	0.622 J	0.0583	63.0
AMOG	8/26/2024	8:27	2.68	1.60 J	73.4	7.80	<1.0	0.477 J	0.127	22.3
AWGB	8/26/2024	8:31	2.16	1.20 J	74.1	7.70	<1.0	0.742 J	0.229	<1.0
AMOG_DUP	8/26/2024	8:27	2.49	1.50 J	73.3	7.78	<1.0	0.435 J	0.125	36.3
AMOG_FB	8/26/2024	8:30	N/A	0.600 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	8/28/2024	7:37	9.95	6.00	75.5	7.69	<1.0	0.621 J	0.134	45.4
IDBW	8/28/2024	8:12	5.70	6.70	76.2	7.86	<1.0	0.563 J	0.133	24.0
BDMZ	8/28/2024	9:10	5.96	6.80	80.9	7.54	<1.0	0.809 J	0.0857	53.3
BDXX	8/28/2024	8:50	8.96	10.3	80.7	7.63	<1.0	2.35	0.0696	172
BDMZ_DUP	8/28/2024	9:10	6.69	5.70	81.3	7.72	<1.0	1.02 J	0.0800	38.1
BDMZ_FB	8/28/2024	9:10	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	9/3/2024	7:00	5.12	5.30	69.4	7.96	<1.0	0.616 J	0.224	4.05

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
KMXX	9/3/2024	7:25	6.07	5.90	68.9	7.89	<1.0	0.476 J	0.135	3.10
IDBW	9/3/2024	7:50	4.85	6.30	69.1	7.74	<1.0	0.549 J	0.134	2.55
BDMZ	9/3/2024	9:10	7.48	8.60	76.4	7.59	<1.0	0.733 J	0.074	60.8
BDXX	9/3/2024	9:35	9.34	8.90	75.3	7.79	<1.0	0.595 J	0.0559	43.4
AMOG	9/3/2024	8:30	3.04	4.30	69.0	7.75	<1.0	0.517 J	0.111	11.1
AWGB	9/3/2024	7:55	3.93	3.50	72.1	7.40	<1.0	0.522 J	0.134	<1.0
BDXX_DUP	9/3/2024	9:35	9.17	8.00	75.5	7.80	<1.0	0.582 J	0.0650	39.1
BDXX_FB	9/3/2024	9:35	N/A	<2.00	N/A	N/A	<1.0	<2.00	<0.0350	<1.0
KMXX	9/4/2024	6:25	2.52	2.40	68.0	7.40	<1.0	0.530 J	0.144	2.50
IDBW	9/4/2024	7:00	2.97	2.30	65.1	7.50	<1.0	0.462 J	0.129	2.00
BDMZ	9/4/2024	7:54	8.01	6.80	72.3	7.76	<1.0	0.564 J	0.0802	50.1
BDXX	9/4/2024	8:13	10.1	8.70	74.6	7.64	<1.0	0.660 J	0.0708	46.8
IDBW_DUP	9/4/2024	7:10	3.03	1.80 J	65.1	7.50	<1.0	0.562 J	0.132	3.50
IDBW_FB	9/4/2024	7:12	N/A	0.400 J	N/A	N/A	<1.0	<2.00	<0.0350	<1.0
MCXX	9/9/2024	7:10	17.8	20.9	66.7	7.71	<1.0	0.714 J	0.131	3.00
KMXX	9/9/2024	7:40	19.4	11.8	65.9	7.55	<1.0	0.778 J	0.132	2.50
IDBW	9/9/2024	8:10	16.9	20.0	66.3	7.64	<1.0	0.680 J	0.131	3.10
BDMZ	9/9/2024	6:20	7.46	6.40	70.7	6.90	<1.0	1.19 J	0.0521	54.0
BDXX	9/9/2024	6:40	7.69	6.50	70.8	7.70	<1.0	0.623 J	0.0457	97.8
AMOG	9/9/2024	8:44	4.10	3.20	66.0	7.39	<1.0	0.541 J	0.123	<1.0
AWGB	9/9/2024	8:25	3.24	3.00	69.8	7.20	<1.0	0.533 J	0.120	2.00
BDMZ_DUP	9/9/2024	6:25	7.41	7.30	70.6	6.90	<1.0	0.857 J	0.0507	65.8
BDMZ_FB	9/9/2024	6:27	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	9/11/2024	6:15	1.72	1.70 J	66.5	7.84	<1.0	0.0534 J	<0.00720	<1.0
IDBW	9/11/2024	6:48	1.78	1.70 J	65.1	7.81	<1.0	0.470 J	<0.00720	<1.0
BDMZ	9/11/2024	7:38	6.73	6.60	74.8	7.80	<1.0	1.12 J	<0.00720	63.9
BDXX	9/11/2024	7:53	8.83	8.00	72.6	7.80	<1.0	0.693 J	<0.00720	64.0
KMXX_DUP	9/11/2024	6:18	1.73	1.20 J	66.5	7.82	<1.0	0.578 J	<0.00720	<1.0

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
KMXX_FB	9/11/2024	6:20	N/A	0.40 J	N/A	N/A	<1.0	<0.433	<0.00720	<1.0
MCXX	9/16/2024	7:00	3.44	2.30	69.0	7.76	<1.0	0.592 J	0.0460	3.60
KMXX	9/16/2024	7:30	2.16	1.80 J	68.5	7.89	<1.0	0.506 J	0.0550	6.20
IDBW	9/16/2024	7:55	2.27	1.00 J	68.3	7.82	<1.0	0.519 J	0.0520	35.3
BDMZ	9/16/2024	6:20	8.28	5.90	73.3	7.00	<1.0	0.763 J	0.0250 J	48.6
BDXX	9/16/2024	6:48	7.50	4.80	74.5	7.00	<1.0	0.667 J	0.0270 J	59.0
AMOG	9/16/2024	8:20	1.68	1.60 J	67.6	7.74	<1.0	0.511 J	0.0230	27.2
AWGB	9/16/2024	8:40	1.96	1.30 J	69.3	8.30	<1.0	0.610 J	0.0410	<1.0
IDBW_DUP	9/16/2024	7:55	2.19	0.900 J	68.3	7.80	<1.0	0.582 J	0.0220	23.8
IDBW_FB	9/16/2024	7:55	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.00720	<1.0
KMXX	9/18/2024	7:23	<1.0	2.90	67.4	7.80	<1.0	0.467 J	0.135	5.70
IDBW	9/18/2024	7:57	<1.0	1.80 J	67.8	8.00	<1.0	<0.433	0.137	5.85
BDMZ	9/18/2024	8:43	2.20	7.50	76.8	7.80	<1.0	0.864 J	0.0721	50.8
BDXX	9/18/2024	9:00	2.04	5.60	76.1	7.80	<1.0	0.683 J	0.0545	64.2
BDXX_DUP	9/18/2024	9:05	2.72	5.50	76.1	7.80	<1.0	0.538 J	0.0558	58.8
BDXX_FB	9/18/2024	9:05	N/A	0.500 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	9/24/2024	12:50	5.84	6.90	70.1	7.91	<1.0	<0.433	0.135	137
KMXX	9/24/2024	12:25	8.90	5.30	69.9	7.76	<1.0	<0.433	0.132	35.8
IDBW	9/24/2024	11:55	5.91	8.80	70.3	7.83	<1.0	<0.433	0.148	34.8
BDMZ	9/24/2024	11:00	3.15	7.30	74.4	7.50	<1.0	1.09 J	0.840	262
BDXX	9/24/2024	10:40	4.92	9.30	72.6	7.55	<1.0	<0.433	0.0325 J	291
AMOG	9/24/2024	11:30	1.49	7.40	69.8	7.98	<1.0	0.449 J	0.124	157
AWGB	9/24/2024	10:00	<1.0	1.40 J	70.1	8.08	<1.0	<0.433	0.130	<1.0
KMXX_DUP	9/24/2024	12:25	8.74	6.70	69.9	7.79	<1.0	<0.433	0.136	33.4
KMXX_FB	9/24/2024	12:25	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
KMXX	9/25/2024	6:50	7.18	8.10	69.3	8.14	<1.0	0.751 J	0.126	26.8
IDBW	9/25/2024	7:15	4.92	7.40	68.9	7.99	<1.0	0.605 J	0.127	28.2
BDMZ	9/25/2024	7:40	4.22	8.20	72.5	7.65	<1.0	0.979 J	0.0616	435

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDXX	9/25/2024	8:00	6.71	9.50	69.9	7.55	<1.0	0.895 J	0.0498	442
BDMZ_DUP	9/25/2024	7:40	4.20	7.60	72.4	7.68	<1.0	1.51 J	0.0615	723
BDMZ_FB	9/25/2024	7:40	N/A	0.546 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	9/30/2024	14:02	14.4	28.8	71.9	7.94	<1.0	0.894 J	0.112	4.60
KMXX	9/30/2024	13:30	19.3	18.0	71.4	7.79	<1.0	1.17 J	0.114	5.10
IDBW	9/30/2024	13:04	18.0	21.4	71.7	7.84	<1.0	0.943 J	0.121	6.15
BDMZ	9/30/2024	12:37	1.78	3.50	77.7	7.90	<1.0	1.07 J	0.0765	56.1
BDXX	9/30/2024	12:20	2.39	4.80	76.5	7.76	<1.0	0.711 J	0.135	49.2
AMOG	9/30/2024	12:03	3.65	4.53	72.3	7.92	<1.0	0.648 J	0.111	4.70
AWGB	9/30/2024	9:49	2.19	2.20	70.3	8.05	<1.0	0.702 J	0.110	2.00
MCXX_DUP	9/30/2024	14:02	14.8	14.7	71.9	7.92	<1.0	0.903 J	0.112	6.25
MCXX_FB	9/30/2024	14:02	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	10/2/2024	11:40	15.4	18.1	67.8	7.91	<1.0	0.944 J	0.119	14.1
KMXX	10/2/2024	12:10	9.24	16.7	66.5	7.89	<1.0	1.00 J	0.124	7.20
IDBW	10/2/2024	12:40	4.75	8.90	67.4	7.76	<1.0	0.811 J	0.125	2.00
BDMZ	10/2/2024	11:30	1.87	6.60	70.1	7.19	<1.0	0.746 J	0.0441	75.5
BDXX	10/2/2024	11:46	2.80	7.90	70.2	7.49	<1.0	0.832 J	0.0335 J	65.2
AMOG	10/2/2024	10:50	3.52	13.1	67.8	7.79	<1.0	0.853 J	0.105	4.00
AWGB	10/2/2024	10:42	<1.0	3.50	68.6	7.35	<1.0	0.818 J	0.105	<1.0
AWGB_DUP	10/2/2024	10:42	<1.0	3.00	68.8	7.38	<1.0	1.79 J	0.102	<1.0
AWGB_DUP	10/2/2024	10:42	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
MCXX	11/7/2024	6:20	4.08	20.4	55.5	6.83	<1.0	0.726 J	0.118	10.6
KMXX	11/7/2024	6:47	4.00	12.6	55.8	6.81	<1.0	0.643 J	0.114	14.0
IDBW	11/7/2024	7:18	2.62	9.40	54.8	7.20	<1.0	0.690 J	0.112	38.2
BDMZ	11/7/2024	8:20	6.08	39.7	56.8	7.10	<1.0	2.09	0.0218 J	699
BDXX	11/7/2024	8:35	5.73	48.5	57.2	7.15	<1.0	1.44 J	0.0217 J	723
AMOG	11/7/2024	7:45	2.73	14.3	56.1	7.30	<1.0	0.742 J	0.0833	623
AWGL	11/7/2024	6:45	2.80	3.20	57.5	7.05	<1.0	0.478 J	0.117	<1.0

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG_DUP	11/7/2024	7:45	2.78	11.4	56.1	7.30	<1.0	0.834 J	0.0785	572
AMOG_FB	11/7/2024	7:45	N/A	0.400 J	N/A	N/A	<1.0	<0.433	<0.0130	<1.0

Abbreviations:
DUP = field duplicate
FB = field blank
TSS = total suspended solids
Hex. Chromium = hexavalent chromium
N/A = not applicable
<#.# = not detected at the limit of detection shown
J = the analyte was detected, but below the limit of quantitation
U = the analyte was analyzed, but not detected above the limit of quantitation

Locations:
AWGL = American Water Intake - Gary Lakeside Pump House
AWGB = American Water Intake - Breakwall
AMOG = American Water Intake - Ogden
BDXX = Burns Ditch
BDMZ = Burns Ditch / Lake Michigan Mixing Zone
IDBW = Indiana Dunes Beach - Western Area
KMXX = Kemil Beach
MCXX = Michigan City

Units:
NTU = nephelometric turbidity unit
mg/L = milligrams per liter
°F = degrees Celsius
s.u. = standard units
µg/L = micrograms per liter
MPN = Most Probable Number per 100 milliliters

4. PUBLIC REPORTING

Paragraph 17 of the Revised Consent Decree Section VII outlines the public reporting requirements of the State-Only EBP. U. S. Steel made all reasonable attempts to meet these requirements.

Paragraph 17.a. mandated U. S. Steel submit analytical data reports weekly from May 1 through September 30 and monthly from October through April. These weekly/monthly reports, as well as annual data summary reports, were submitted within the time listed in the Revised Consent Decree, except as noted in Section 3, above. The data were also uploaded to a dedicated website created by U. S. Steel: https://midwest.uss.com/lake-mi[4].

U. S. Steel and ALS Valparaiso attempted to submit *E. coli* concentrations to the Beach Guard notification system from November 2021 through March 2022, as required at Paragraph 17.b. IDEM confirmed in an email dated February 28, 2022 that the Beach Guard program was no longer a functional database and therefore posting data to the Beach*Alert* website would not be possible. U. S. Steel continued to submit the *E. coli* data along with the other results to the three IDEM email addresses provided in the letter within eight hours of receipt of the data (per Paragraph 17.d).

For all sampling events, ALS Valparaiso prepared electronic data interchange (EDI) files in ASCII format that met data quality assessment Level 3 criteria outlined in IDEM (2015) Technical Guidance for the Office of Water Quality External Data Framework, as required at Paragraph 17.c.

[4] The correct web address to reach the Midwest Plant page is https://midwest.uss.com. "www.midwest.uss.com" as stated in the Consent Decree leads to an error page.

APPENDIX 1
2021-2023 ANALYTICAL RESULTS

Table 1. 2021 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°C)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
AWGL	11/16/2021	08:17	7.27	9.10	49.1	7.74	<1.0	0.833 J	0.114	<1.0
AMOG	11/16/2021	09:36	8.80	8.27	46.4	7.73	<1.0	0.676 J	0.125	1.0
BDXX	11/16/2021	10:00	6.83	5.89	50.9	7.54	<1.0	0.689 J	0.0960	78.2
BDMZ	11/16/2021	10:28	8.41	5.78	46.9	7.63	<1.0	0.729 J	0.0994	103.4
IDBW	11/16/2021	11:23	16.1	11.7	50.1	7.72	<1.0	0.764 J	0.134	7.6
KMXX	11/16/2021	12:02	24.9	20.2	49.6	7.71	<1.0	0.986 J	0.145	6.0
MCXX	11/16/2021	12:47	21.0	16.3	50.5	7.77	<1.0	1.02 J	0.146	10.0
AWGL-DUP	11/16/2021	08:20	7.23	10.1	48.7	7.73	<1.0	0.530 J	0.121	<1.0
AWGL-FB	11/16/2021	08:10	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0154 J	<1.0
AWGL	12/02/2021	11:20	4.83	4.97	51.8	7.61	<1.0	0.554 J	0.142	<1.0
AMOG	12/02/2021	10:35	14.4	14.2	48.7	7.62	<1.0	0.992 J	0.141	<1.0
BDXX	12/02/2021	09:51	8.32	8.79	51.4	7.62	<1.0	0.724 J	0.0817	54.7
BDMZ	12/02/2021	10:09	8.55	9.16	49.4	7.47	<1.0	0.647 J	0.0857	48.4
IDBW	12/02/2021	09:07	8.85	10.8	47.8	7.71	<1.0	0.525 J	0.137	<1.0
KMXX	12/02/2021	08:24	27.1	20.0	45.8	7.63	<1.0	0.689 J	0.140	<1.0
MCXX	12/02/2021	07:38	14.4	12.2	44.7	7.81	<1.0	0.581 J	0.137	5.2
MCXX-DUP	12/02/2021	07:40	15.0	13.8	44.7	7.55	<1.0	0.719 J	0.135	5.8
MCXX-FB	12/02/2021	07:45	N/A	0.622 J	N/A	N/A	<1.0	<0.433	0.0375	<1.0

Abbreviations:
TSS = total suspended solids
Hex. Chromium = hexavalent chromium
N/A = not applicable
<#.# = not detected at the limit of detection shown
J = the analyte was detected, but below the limit of quantitation
DUP = field duplicate
FB = field blank

Locations:
AWGL = American Water Intake - Gary Lakeside Pump House
AMOG = American Water Intake - Ogden
BDXX = Burns Ditch
BDMZ = Burns Ditch / Lake Michigan Mixing Zone
IDBW = Indiana Dunes Beach - Western Area
KMXX = Kemil Beach
MCXX = Michigan City

Units:
NTU = nephelometric turbidity unit
mg/L = milligrams per liter
°C = degrees Celsius
s.u. = standard units
ug/L = micrograms per liter
MPN = Most Probable Number per 100 milliliters

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
AWGL	1/4/2022	13:20	9.41	9.10	36.1	7.92	<1.0	<0.433	0.151	<1.0
AMOG	1/4/2022	11:00	9.86	5.33	36.6	8.00	<1.0	0.890 J	0.214	5.1
BDXX	1/4/2022	10:00	13.1	8.40	36.6	7.96	<1.0	1.24 J	0.0975	233.7
BDMZ	1/4/2022	10:24	11.9	11.7	35.9	7.65	<1.0	1.24 J	0.112	287
IDBW	1/4/2022	9:22	9.13	8.22	35.6	8.05	<1.0	0.520 J	0.182	<1.0
KMXX	1/4/2022	8:38	17.4	15.2	34.5	7.91	<1.0	0.602 J	0.178	<1.0
MCXX	1/4/2022	8:00	14.7	10.8	36.5	7.64	<1.0	0.593 J	0.116	<1.0
MCXX-DUP	1/4/2022	8:02	16.2	19.5	36.5	7.68	<1.0	0.995 J	0.148	<1.0
MCXX-FB	1/4/2022	7:58	N/A	0.410 J	N/A	N/A	<1.0	<0.433	0.0351	<1.0
AWGL	2/24/2022	8:59	16.1	17.1	34.3	7.97	<1.0	0.966 J	0.168	1.0
AMOG	2/24/2022	Sample not collected due to ice cover.								
BDXX	2/24/2022	7:52	41.7	33.0	32.0	7.10	<1.0	2.66	0.212	1,139
BDMZ	2/24/2022	7:33	44.1	30.2	31.8	7.47	<1.0	2.42	0.212	645.9
IDBW	2/24/2022	Sample not collected due to ice cover.								
KMXX	2/24/2022	Sample not collected due to ice cover.								
MCXX	2/24/2022	Sample not collected due to ice cover.								
AWGL-DUP	2/24/2022	8:54	12.5	17.3	33.0	7.97	<1.0	0.992 J	0.198	2.0
AWGL-FB	2/24/2022	8:50	N/A	0.321	N/A	N/A	<1.0	<0.433	<0.0130	<1.0
AWGL	3/21/2022	9:10	5.94	2.86	41.3	8.00	<1.0	0.509 J	0.213	<1.0
AMOG	3/21/2022	8:26	5.32	3.04	44.7	7.90	<1.0	0.569 J	0.246	<1.0
BDXX	3/21/2022	8:05	17.5	15.9	48.9	7.54	<1.0	1.22 J	0.222	344.7
BDMZ	3/21/2022	7:47	18.0	14.5	50.1	7.70	<1.0	1.22 J	0.163	348.8
IDBW	3/21/2022	7:06	6.28	3.60	41.3	8.11	<1.0	0.557 J	0.241	2.6
KMXX	3/21/2022	6:34	8.94	4.24	41.7	8.04	<1.0	0.568 J	0.276	2.0
MCXX	3/21/2022	5:50	10.3	5.20	40.2	8.58	<1.0	0.660 J	0.261	7.3
AWGL-DUP	3/21/2022	9:10	5.91	2.78	41.5	8.01	<1.0	1.58 J	0.189	1.0
AWGL-FB	3/21/2022	9:14	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0159 J	<1.0
AWGL	4/4/2022	8:25	7.88	3.00	44.5	7.91	<1.0	0.551 J	0.189	<1.0
AMOG	4/4/2022	7:38	8.23	10.3	43.9	7.84	<1.0	1.35 J	0.156	41.9
BDXX	4/4/2022	6:51	9.76	28.3	46.7	8.07	<1.0	2.56	0.134	1,158.8
BDMZ	4/4/2022	7:14	9.08	26.6	46.2	8.09	<1.0	2.47	0.159	922.2
IDBW	4/4/2022	6:24	8.15	4.77	43.6	8.11	<1.0	0.809 J	0.218	4.5
KMXX	4/4/2022	5:58	8.24	7.49	44.7	7.95	<1.0	0.606 J	0.190	<1.0
MCXX	4/4/2022	5:31	7.15	6.49	46.5	8.02	<1.0	0.727 J	0.190	5.5
AMOG-DUP	4/4/2022	7:59	8.20	11.0	43.8	8.04	<1.0	1.28 J	0.172	46.9
AMOG-FB	4/4/2022	7:37	N/A	<0.300	N/A	N/A	<1.0	0.524 J	0.0229 J	<1.0

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	5/2/2022	8:41	9.04	8.30	44.6	8.21	<1.0	0.603 J	0.165	2.0
KMXX	5/2/2022	8:04	9.00	12.2	46.1	8.09	<1.0	0.609 J	0.148	41.2
IDBW	5/2/2022	7:37	8.92	4.50	45.2	8.11	<1.0	0.533 J	0.174	1.0
BDMZ	5/2/2022	6:38	8.04	14.3	53.7	7.90	<1.0	0.965 J	0.0812	1230.5
BDXX	5/2/2022	6:24	8.91	17.4	53.3	8.03	<1.0	1.03 J	0.114	821.2
AMOG	5/2/2022	6:53	8.31	1.30 J	44.3	8.06	<1.0	<0.433	0.162	<1
AWGL	5/2/2022	6:03	7.73	7.44	45.1	7.84	<1.0	1.02 J	0.146	5.1
BDXX-DUP	5/2/2022	6:52	7.92	17.9	53.5	7.90	<1.0	1.06 J	0.115	637.3
BDXX-FB	5/2/2022	6:23	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0193 J	<1
KMXX	5/4/2022	12:00	17.8	20.7	50.1	7.63	<1.0	0.697 J	0.163	14.7
IDBW	5/4/2022	11:12	14.1	23.4	50.9	7.72	<1.0	0.809 J	0.153	16.3
BDMZ	5/4/2022	10:35	28.6	25.5	53.2	7.34	<1.0	1.35 J	0.119	>2419.6
BDXX	5/4/2022	10:20	26.4	25.3	50.4	7.51	<1.0	1.58 J	0.105	>2419.6
KMXX-DUP	5/4/2022	12:01	17.6	21.7	50.1	7.60	<1.0	0.918 J	0.148	14.5
KMXX-FB	5/4/2022	11:50	N/A	0.609 J	N/A	N/A	<1.0	<0.433	0.0297 J	<1
MCXX	5/9/2022	6:50	2.87	2.50	50.1	8.20	<1.0	0.452 J	0.145	5.9
KMXX	5/9/2022	7:24	2.87	2.60	50.5	8.15	<1.0	<0.433	0.158	3.1
IDBW	5/9/2022	8:02	3.37	4.40	55.9	6.90	<1.0	<0.433	0.157	2.0
BDMZ	5/9/2022	9:41	18.0	18.4	64.9	7.12	<1.0	1.36 J	0.0794	100.8
BDXX	5/9/2022	9:19	17.8	17.3	65.3	7.46	<1.0	1.11 J	0.0794	223.1
AMOG	5/9/2022	8:40	7.88	8.80	56.1	7.63	<1.0	0.558 J	0.146	17.4
AWGL	5/9/2022	11:40	2.40	4.51	52.7	7.69	<1.0	0.438 J	0.152	2.0
BDMZ-DUP	5/9/2022	9:49	17.8	17.6	65.1	7.48	<1.0	1.12 J	0.113	339.1
BDMZ-FB	5/9/2022	9:55	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0258 J	<1
KMXX	5/11/2022	7:07	1.45	1.51 J	56.8	7.68	<1.0	<0.433	0.143	<1
IDBW	5/11/2022	7:34	1.87	2.50	57.0	7.59	<1.0	0.440 J	0.135	<1
BDMZ	5/11/2022	8:03	12.3	11.6	72.1	7.44	<1.0	0.827 J	0.0783	52.0
BDXX	5/11/2022	8:24	10.5	12.2	70.5	7.26	<1.0	1.01 J	0.0727	81.5
BDXX-DUP	5/11/2022	8:24	11.9	17.3	72.1	7.48	<1.0	0.750 J	0.0766	62.8
BDXX-FB	5/11/2022	8:20	N/A	4.54	N/A	N/A	<1.0	<0.433	0.0187 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	5/16/2022	5:52	9.75	13.9	53.2	8.13	<1.0	0.905 J	0.216	29.0
KMXX	5/16/2022	6:21	4.09	7.11	54.6	8.20	<1.0	0.881 J	0.195	23.0
IDBW	5/16/2022	6:52	5.96	12.7	54.5	7.92	<1.0	0.891 J	0.200	107
BDMZ	5/16/2022	7:49	9.61	16.0	59.9	8.12	<1.0	1.22 J	0.0932	61.1
BDXX	5/16/2022	7:28	9.03	16.0	57.8	8.21	<1.0	1.00 J	0.0758	75.6
AMOG	5/16/2022	7:17	5.72	16.5	53.6	8.30	<1.0	0.984 J	0.176	2,330
AWGL	5/16/2022	8:35	4.34	1.21 J	51.4	8.01	<1.0	0.634 J	0.202	<1.0
IDBW-DUP	5/16/2022	6:59	5.97	11.1	54.5	7.92	<1.0	0.879 J	0.197	84.4
IDBW-FB	5/16/2022	6:50	N/A	<0.300	N/A	N/A	<1.0	0.434 J	0.0366	<1.0
KMXX	5/18/2022	9:20	5.22	8.90	51.4	7.76	<1.0	0.868 J	0.180	<1
IDBW	5/18/2022	8:40	4.61	3.90	53.9	8.03	<1.0	0.676 J	0.179	7.9
BDMZ	5/18/2022	7:57	24.0	15.8	65.3	7.91	<1.0	1.09 J	0.0694	37.3
BDXX	5/18/2022	8:13	17.7	17.9	65.4	7.76	<1.0	1.73 J	0.0740	65.5
BDMZ-DUP	5/18/2022	8:01	17.9	14.8	65.4	7.78	<1.0	1.18 J	0.0687	29.2
BDMZ-FB	5/18/2022	7:40	N/A	<0.300	N/A	N/A	<1.0	0.441 J	0.0329 J	<1
MCXX	5/23/2022	5:20	8.66	29.3	54.8	8.08	<1.0	0.772 J	0.180	18.8
KMXX	5/23/2022	5:43	9.12	14.5	54.1	8.17	<1.0	0.559 J	0.167	6.25
IDBW	5/23/2022	6:04	8.50	10.6	54.3	8.01	<1.0	0.718 J	0.168	11.2
BDMZ	5/23/2022	6:44	11.6	12.7	59.2	7.84	<1.0	1.04 J	0.0708	75
BDXX	5/23/2022	6:31	21.3	14.9	58.4	7.69	<1.0	0.761 J	0.0644	79.1
AMOG	5/23/2022	7:13	9.06	9.85	54.4	8.24	<1.0	0.525 J	0.146	9
AWGB	5/23/2022	8:41	10.3	2.50	53.9	8.21	<1.0	<0.433	0.172	1
KMXX-DUP	5/23/2022	5:53	9.12	16.7	54.0	8.18	<1.0	0.653 J	0.175	8.3
KMXX-FB	5/23/2022	5:40	N/A	<0.303	N/A	N/A	<1.0	<0.433	0.0176 J	<1
KMXX	5/25/2022	8:51	10.1	21.1	54.9	8.20	<1.0	0.679 J	0.186	11.8
IDBW	5/25/2022	8:11	9.90	19.6	55.6	8.21	<1.0	0.831 J	0.181	11.8
BDMZ	5/25/2022	7:39	14.4	15.6	58.5	8.14	<1.0	0.845 J	0.0694	55.4
BDXX	5/25/2022	7:28	11.9	19.0	58.3	8.17	<1.0	0.875 J	0.0656	62.7
IDBW-DUP	5/25/2022	8:23	10.2	18.7	55.4	8.20	<1.0	0.691 J	0.174	13.9
IDBW-FB	5/25/2022	8:10	N/A	0.500 J	N/A	N/A	<1.0	<0.433	0.0271 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	5/31/2022	6:25	3.74	2.40	61.8	8.15	<1.0	<0.433	0.192	5.15
KMXX	5/31/2022	6:50	2.11	2.90	61.5	8.19	<1.0	0.529 J	0.185	2.50
IDBW	5/31/2022	7:23	3.38	2.90	62.0	8.22	<1.0	0.710 J	0.173	<1.0
BDMZ	5/31/2022	8:32	8.44	14.3	63.8	8.12	<1.0	1.08 J	0.0690	36.9
BDXX	5/31/2022	8:07	9.03	15.3	64.1	8.01	<1.0	1.07 J	0.0624	43.0
AMOG	5/31/2022	7:41	3.84	2.90	61.9	8.13	<1.0	0.595 J	0.176	<1.0
AWGB	5/31/2022	10:26	3.35	2.14 J	62.4	8.27	<1.0	1.02 J	0.245	<1.0
MCXX-DUP	5/31/2022	6:31	3.74	1.80 J	61.8	8.15	<1.0	0.499 J	0.174	5.15
MCXX-FB	5/31/2022	6:20	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0305 J	<1
KMXX	6/1/2022	7:10	3.64	2.20	61.1	8.13	<1.0	0.502 J	0.186	36.4
IDBW	6/1/2022	7:58	3.81	2.00	61.4	8.17	<1.0	0.512 J	0.196	5.65
BDMZ	6/1/2022	8:38	8.95	12.7	63.2	8.07	<1.0	0.967 J	0.0492	63.4
BDXX	6/1/2022	8:19	9.23	13.1	63.3	8.11	<1.0	0.845 J	0.0737	88.7
KMXX-DUP	6/1/2022	7:14	3.60	2.40	61.2	8.13	<1.0	0.502 J	0.207	5.15
KMXX-FB	6/1/2022	7:07	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0460	<1
MCXX	6/6/2022	5:45	4.60	6.80	55.9	8.00	<1.0	0.527 J	0.198	6.25
KMXX	6/6/2022	6:05	3.20	3.20	56.2	8.20	<1.0	0.566 J	0.221	3.00
IDBW	6/6/2022	6:25	2.90	2.80	55.8	7.90	<1.0	0.679 J	0.190	3.55
BDMZ	6/6/2022	7:00	8.40	11.0	67.4	7.80	<1.0	0.949 J	0.165	55.8
BDXX	6/6/2022	6:45	9.00	12.6	68.1	7.40	<1.0	0.660 J	0.109	111
AMOG	6/6/2022	7:35	5.40	4.20	60.6	7.80	<1.0	0.451 J	0.188	11.0
AWGB	6/6/2022	8:10	9.00	1.20 J	61.7	7.80	<1.0	0.466 J	0.214	<1
AWGB-DUP	6/6/2022	8:12	8.90	1.10 J	61.8	7.80	<1.0	0.488 J	0.211	<1
AWGB-FB	6/6/2022	8:08	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0636	<1
KMXX	6/9/2022	5:29	9.76	6.90	58.1	8.20	<1.0	<0.433	0.184	20.2
IDBW	6/9/2022	5:43	10.1	5.10	58.4	8.23	<1.0	0.548 J	0.161	11.2
BDMZ	6/9/2022	6:02	22.4	15.6	62.4	8.03	<1.0	1.04 J	0.0854	572
BDXX	6/9/2022	6:21	19.8	16.6	61.7	8.07	<1.0	0.878 J	0.0806	506
BDXX-DUP	6/9/2022	6:25	21.9	17.5	62.3	8.03	<1.0	0.817 J	0.0818	661
BDXX-FB	6/9/2022	9:18	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0411	<1.0

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	6/14/2022	6:28	8.01	7.80	64.5	7.96	<1.0	0.609 J	0.179	2.00
KMXX	6/14/2022	7:06	3.63	1.70 J	65.1	7.82	<1.0	0.493 J	0.203	1.00
IDBW	6/14/2022	7:37	4.67	3.20	67.1	7.22	<1.0	<0.433	0.192	<1
BDMZ	6/14/2022	9:20	14.6	15.9	80.2	7.48	<1.0	1.28 J	0.324	432
BDXX	6/14/2022	9:05	13.1	22.0	81.6	7.54	<1.0	0.896 J	0.125	574
AMOG	6/14/2022	8:31	3.54	3.00	71.0	7.73	<1.0	0.664 J	0.228	5.15
AWGB	6/14/2022	10:25	1.30	1.10 J	72.6	8.10	<1.0	0.625 J	0.188	3.00
AMOG-DUP	6/14/2022	8:40	2.96	2.50	69.8	7.65	<1.0	0.706 J	0.194	4.60
AMOG-FB	6/14/2022	8:22	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0381 J	<1
KMXX	6/15/2022	9:41	3.71	1.50 J	65.4	7.79	<1.0	0.446 J	0.174	1.00
IDBW	6/15/2022	9:05	3.01	1.50 J	66.3	7.49	<1.0	0.462 J	0.161	2.00
BDMZ	6/15/2022	7:07	12.9	11.0	80.3	7.36	<1.0	0.606 J	0.119	303
BDXX	6/15/2022	6:48	13.1	10.9	80.4	7.41	<1.0	0.813 J	0.111	469
BDMZ-DUP	6/15/2022	7:10	19.0	11.8	80.5	7.45	<1.0	0.894 J	0.111	342
BDMZ-FB	6/15/2022	7:05	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0239 J	<1
MCXX	6/20/2022	6:15	1.80	0.900 J	64.8	8.10	<1.0	0.470 J	0.225	3.55
KMXX	6/20/2022	6:40	1.41	1.00 J	65.1	8.20	<1.0	<0.433	0.233	1.00
IDBW	6/20/2022	6:58	1.60	1.00 J	66.3	7.70	<1.0	<0.433	0.217	2.00
BDMZ	6/20/2022	7:20	9.30	8.70	75.7	8.10	<1.0	0.494 J	0.125	77.2
BDXX	6/20/2022	7:40	8.20	7.80	74.8	7.90	<1.0	0.531 J	0.140	74.0
AMOG	6/20/2022	8:15	3.10	1.60 J	68.6	8.00	<1.0	0.466 J	0.242	<1
AWGB	6/20/2022	8:50	2.70	1.20 J	69.6	7.80	<1.0	1.08 J	0.305	<1
BDXX-DUP	6/20/2022	7:40	9.10	7.50	75.7	8.10	<1.0	0.496 J	0.121	91.7
BDXX-FB	6/20/2022	7:37	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0873	<1
KMXX	6/22/2022	6:20	4.03	4.95	72.1	7.40	<1.0	0.581 J	0.225	17.0
IDBW	6/22/2022	6:45	6.10	6.60	72.7	7.60	<1.0	0.473 J	0.227	14.0
BDMZ	6/22/2022	7:50	5.69	11.6	80.0	7.70	<1.0	0.670 J	0.120	60.5
BDXX	6/22/2022	8:05	4.64	9.50	79.8	7.90	<1.0	0.553 J	0.118	88.8
IDBW-DUP	6/22/2022	6:46	5.96	6.40	72.6	7.60	<1.0	0.466 J	0.186	15.9
IDBW-FB	6/22/2022	6:40	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0460	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	6/27/2022	5:31	10.2	9.70	67.4	8.04	<1.0	1.07 J	0.175	11.6
KMXX	6/27/2022	6:02	9.40	4.70	66.8	7.96	<1.0	0.572 J	0.166	4.50
IDBW	6/27/2022	6:30	9.80	5.20	66.8	8.02	<1.0	0.696 J	0.177	2.00
BDMZ	6/27/2022	7:10	17.6	17.0	69.3	7.63	<1.0	1.23 J	0.289	294
BDXX	6/27/2022	6:52	18.5	15.7	69.5	7.71	<1.0	1.49 J	0.0736	485
AMOG	6/27/2022	7:28	11.3	9.00	65.2	8.03	<1.0	1.51 J	0.205	11.9
AWGB	6/27/2022	7:59	20.3	17.8	65.4	7.96	<1.0	0.944 J	0.199	144
BDMZ-DUP	6/27/2022	7:12	17.9	15.7	69.4	7.70	<1.0	1.42 J	0.278	299
BDMZ-FB	6/27/2022	7:08	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1
KMXX	6/29/2022	5:54	4.13	1.70 J	66.4	8.08	<1.0	0.523 J	0.218	2.00
IDBW	6/29/2022	6:28	2.87	1.72 J	66.5	8.11	<1.0	0.667 J	0.197	1.50
BDMZ	6/29/2022	8:48	10.1	12.4	68.5	7.54	<1.0	0.652 J	0.111	471
BDXX	6/29/2022	8:59	9.82	14.3	68.8	8.63	<1.0	0.695 J	0.116	314
KMXX-DUP	6/29/2022	5:59	4.00	2.25 J	66.1	8.08	<1.0	0.544 J	0.203	3.00
KMXX-FB	6/29/2022	5:51	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0413	<1
MCXX	7/5/2022	9:31	3.82	0.700 J	67.1	8.13	<1.0	0.462 J	0.190	<1
KMXX	7/5/2022	8:59	4.22	0.400 J	67.3	8.01	<1.0	<0.433	0.193	4.10
IDBW	7/5/2022	8:09	2.71	0.600 J	66.9	7.90	<1.0	0.623 J	0.188	3.60
BDMZ	7/5/2022	7:12	10.3	13.1	69.6	7.57	<1.0	0.602 J	0.0621	710
BDXX	7/5/2022	7:36	9.76	12.7	69.9	7.64	<1.0	0.515 J	0.0662	511
AMOG	7/5/2022	6:50	3.21	0.600 J	66.8	7.94	<1.0	<0.433	0.186	<1
AWGB	7/5/2022	6:10	5.63	0.400 J	67.2	7.91	<1.0	<0.433	0.179	1.50
IDBW-DUP	7/5/2022	8:14	3.03	0.700 J	67.0	7.93	<1.0	<0.433	0.191	<1
IDBW-FB	7/5/2022	8:05	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0315 J	<1
KMXX	7/6/2022	5:12	4.18	7.00	66.1	8.04	<1.0	0.599 J	0.205	66.6
IDBW	7/6/2022	5:47	3.00	6.80	65.9	7.76	<1.0	0.829 J	0.245	39.0
BDMZ	7/6/2022	8:18	8.51	14.4	71.2	7.66	<1.0	0.536 J	0.0872	168
BDXX	7/6/2022	7:56	8.12	13.3	70.8	7.45	<1.0	0.578 J	0.0919	2,190
BDXX-DUP	7/6/2022	7:59	8.92	12.2	70.7	7.67	<1.0	0.638 J	0.100	2,760
BDXX-FB	7/6/2022	7:53	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0448 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	7/11/2022	6:22	2.71	1.80 J	66.3	8.01	<1.0	0.746 J	0.201	14.7
KMXX	7/11/2022	6:51	3.83	1.60 J	66.8	8.00	<1.0	0.696 J	0.192	<1
IDBW	7/11/2022	7:34	2.01	1.70 J	67.1	7.93	<1.0	0.783 J	0.182	2.00
BDMZ	7/11/2022	8:20	8.12	17.7	69.8	7.54	<1.0	0.923 J	0.0921	262
BDXX	7/11/2022	8:01	8.11	12.2	69.3	7.36	<1.0	0.803 J	0.0924	162
AMOG	7/11/2022	8:31	3.07	1.80 J	67.4	7.91	<1.0	0.692 J	0.156	1.00
AWGB	7/11/2022	9:01	4.60	1.30 J	66.9	7.82	<1.0	0.975 J	0.217	2.00
KMXX-DUP	7/11/2022	6:56	3.40	1.40 J	66.8	8.01	<1.0	0.691 J	0.196	<1
KMXX-FB	7/11/2022	6:49	N/A	<0.300	N/A	N/A	<1.0	0.463 J	0.0395 J	<1
KMXX	7/13/2022	7:29	3.27	4.70	70.5	8.06	<1.0	0.665 J	0.173	3.00
IDBW	7/13/2022	6:58	3.39	4.70	70.3	8.02	<1.0	0.520 J	0.165	5.60
BDMZ	7/13/2022	8:25	9.44	8.50	78.9	9.49	<1.0	0.475 J	0.0704	161
BDXX	7/13/2022	8:39	9.02	9.40	79.1	9.02	<1.0	<0.433	0.0713	112
BDMZ-DUP	7/13/2022	8:27	9.00	8.90	78.8	9.00	<1.0	<0.433	0.0712	126
BDMZ-FB	7/13/2022	8:22	N/A	0.600 J	N/A	N/A	<1.0	<0.433	0.0351 J	<1
MCXX	7/18/2022	6:03	7.61	16.8	70.4	7.96	<1.0	0.731 J	0.188	4.05
KMXX	7/18/2022	6:18	8.12	19.2	70.8	7.81	<1.0	0.697 J	0.184	16.6
IDBW	7/18/2022	6:40	8.93	9.10	70.5	7.90	<1.0	0.671 J	0.187	2.00
BDMZ	7/18/2022	7:14	14.4	5.20	74.4	7.57	<1.0	0.620 J	0.103	190
BDXX	7/18/2022	7:04	18.5	5.50	74.9	7.41	<1.0	0.528 J	0.0822	397
AMOG	7/18/2022	7:37	19.0	11.7	70.2	7.88	<1.0	<0.433	0.179	<1
AWGB	7/18/2022	8:19	8.08	3.31	71.1	7.62	<1.0	0.641 J	0.183	2.00
MCXX-DUP	7/18/2022	6:05	7.70	18.6	70.3	7.96	<1.0	0.631 J	0.187	4.15
MCXX-FB	7/18/2022	5:59	N/A	<0.300	N/A	N/A	<1.0	0.471 J	0.0231 J	<1
KMXX	7/20/2022	6:56	2.02	1.60 J	70.2	7.82	<1.0	<0.433	0.172	3.1
IDBW	7/20/2022	6:34	2.16	1.60 J	70.5	7.87	<1.0	0.510 J	0.175	1.00
BDMZ	7/20/2022	7:41	9.11	6.77	75.1	7.53	<1.0	0.465 J	0.0845	132
BDXX	7/20/2022	7:30	9.07	6.80	74.9	7.49	<1.0	0.454 J	0.0885	235
IDBW-DUP	7/20/2022	6:38	2.10	1.60 J	70.2	7.87	<1.0	<0.433	0.184	1.00
IDBW-FB	7/20/2022	6:33	N/A	<0.370	N/A	N/A	<1.0	<0.433	0.0271 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	7/25/2022	6:11	8.11	6.00	76.2	7.55	<1.0	0.525 J	0.197	12.2
KMXX	7/25/2022	6:38	8.03	6.50	76.5	7.58	<1.0	<0.433	0.182	4.05
IDBW	7/25/2022	7:04	7.77	5.30	76.1	7.69	<1.0	0.606 J	0.157	3.60
BDMZ	7/25/2022	7:36	14.1	11.5	78.0	7.51	<1.0	1.25 J	0.102	858
BDXX	7/25/2022	7:21	13.2	10.5	78.4	7.59	<1.0	0.496 J	0.141	875
AMOG	7/25/2022	7:51	8.12	6.40	75.2	7.88	<1.0	0.780 J	0.172	5.15
AWGB	7/25/2022	8:27	5.19	5.70	76.4	7.87	<1.0	0.609 J	0.182	9.50
AWGB-DUP	7/25/2022	7:30	5.49	6.30	76.1	7.84	<1.0	0.597 J	0.176	9.45
AWGB-FB	7/25/2022	8:25	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
KMXX	7/27/2022	7:45	4.60	1.60 J	73.6	7.49	<1.0	<0.433	0.215	3.00
IDBW	7/27/2022	8:30	5.70	1.90 J	74.3	7.71	<1.0	0.436 J	0.188	4.10
BDMZ	7/27/2022	9:35	14.1	11.7	78.9	7.84	<1.0	0.884 J	0.235	148
BDXX	7/27/2022	9:55	12.7	9.10	79.6	7.73	<1.0	0.550 J	0.135	280
KMXX-DUP	7/27/2022	7:45	4.10	1.90 J	73.2	7.29	<1.0	0.439 J	0.187	1.00
KMXX-FB	7/27/2022	7:40	N/A	<0.300	N/A	N/A	<1.0	0.674 J	0.0407 J	<1
MCXX	8/1/2022	5:48	2.04	3.30	70.4	7.83	<1.0	<0.433	0.201	12.8
KMXX	8/1/2022	6:11	2.13	1.60 J	72.3	7.65	<1.0	0.473 J	0.194	11.2
IDBW	8/1/2022	6:38	3.83	0.900 J	72.9	7.72	<1.0	0.988 J	0.190	2.00
BDMZ	8/1/2022	7:20	10.1	7.10	73.1	7.67	<1.0	1.00 J	0.123	97.3
BDXX	8/1/2022	7:08	8.99	7.50	72.6	7.61	<1.0	0.944 J	0.110	133
AMOG	8/1/2022	7:57	4.16	2.10	72.2	7.70	<1.0	0.543 J	0.159	4.60
AWGB	8/1/2022	8:37	2.47	1.60 J	72.2	7.88	<1.0	0.678 J	0.224	4.15
AMOG-DUP	8/1/2022	8:04	4.06	3.10	72.1	7.56	<1.0	0.576 J	0.174	6.70
AMOG-FB	8/1/2022	7:55	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0338 J	<1
KMXX	8/3/2022	8:52	2.12	1.70 J	72.3	7.81	<1.0	0.496 J	0.210	<1
IDBW	8/3/2022	8:16	2.41	1.70 J	72.5	7.88	<1.0	<0.433	0.187	<1
BDMZ	8/3/2022	7:14	7.79	10.3	77.6	7.57	<1.0	0.551 J	0.106	130
BDXX	8/3/2022	6:59	7.11	12.6	78.1	7.59	<1.0	0.555 J	0.0913	12.1
BDXX-DUP	8/3/2022	7:04	7.15	11.3	78.0	7.61	<1.0	0.698 J	0.0916	156
BDXX-FB	8/3/2022	6:56	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0218 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	8/8/2022	5:52	3.16	2.20	73.1	8.01	<1.0	0.468 J	0.194	9.80
KMXX	8/8/2022	6:11	3.84	1.40 J	73.4	7.96	<1.0	0.544 J	0.199	5.65
IDBW	8/8/2022	6:38	2.89	1.40 J	72.6	7.88	<1.0	0.558 J	0.194	1.00
BDMZ	8/8/2022	7:30	10.1	8.00	75.1	7.54	<1.0	0.551 J	0.105	159
BDXX	8/8/2022	7:04	9.91	7.10	75.8	7.55	<1.0	0.582 J	0.105	182
AMOG	8/8/2022	8:01	4.16	1.20 J	72.8	7.68	<1.0	0.935 J	0.184	15.4
AWGB	8/8/2022	8:49	4.03	1.20 J	73.0	7.68	<1.0	0.590 J	0.227	5.10
BDXX-DUP	8/8/2022	7:06	9.82	7.60	75.4	7.55	<1.0	0.643 J	0.129	199
BDXX-FB	8/8/2022	7:02	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0271 J	<1
KMXX	8/10/2022	7:29	2.11	9.50	83.6	7.81	<1.0	0.590 J	0.158	9.30
IDBW	8/10/2022	6:54	2.26	9.10	82.3	7.92	<1.0	0.790 J	0.162	10.6
BDMZ	8/10/2022	8:26	9.39	7.70	86.1	7.64	<1.0	0.590 J	0.0650	495
BDXX	8/10/2022	8:18	8.14	8.40	85.2	7.61	<1.0	0.547 J	0.0468	515
BDMZ-DUP	8/10/2022	8:28	9.41	7.30	86.0	7.57	<1.0	0.700 J	0.0541	283
BDMZ-FB	8/10/2022	8:25	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0239 J	<1
MCXX	8/15/2022	6:31	8.64	11.0	83.7	8.04	<1.0	0.658 J	0.178	6.80
KMXX	8/15/2022	6:57	8.91	16.6	84.2	7.92	<1.0	0.747 J	0.162	32.1
IDBW	8/15/2022	7:36	8.99	10.0	84.9	8.09	<1.0	0.606 J	0.175	12.8
BDMZ	8/15/2022	8:01	16.3	6.80	87.5	7.86	<1.0	0.487 J	0.114	51.2
BDXX	8/15/2022	8:21	14.1	6.70	87.3	7.92	<1.0	0.632 J	0.0736	101
AMOG	8/15/2022	8:40	9.02	8.30	84.1	7.91	<1.0	0.879 J	0.162	16.1
AWGB	8/15/2022	9:05	9.31	3.50	84.4	7.88	<1.0	0.840 J	0.161	2.50
BDMZ-DUP	8/15/2022	8:04	16.3	7.20	83.9	7.86	<1.0	0.792 J	0.124	77.5
BDMZ-FB	8/15/2022	8:00	N/A	0.300 J	N/A	N/A	<1.0	0.433 J	0.0293 J	<1
KMXX	8/17/2022	7:58	8.88	2.80	83.4	7.94	<1.0	0.463 J	0.164	5.70
IDBW	8/17/2022	7:18	7.58	3.90	83.1	7.92	<1.0	0.638 J	0.157	4.60
BDMZ	8/17/2022	9:11	14.7	7.50	83.8	7.43	<1.0	0.572 J	0.0940	99.4
BDXX	8/17/2022	8:59	13.2	8.40	84.1	7.51	<1.0	0.600 J	0.0988	51.5
IDBW-DUP	8/17/2022	7:20	7.57	3.90	83.0	7.92	<1.0	0.610 J	0.140	2.00
IDBW-FB	8/17/2022	7:58	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0222 J	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	8/22/2022	6:02	7.16	14.8	83.2	7.84	<1.0	0.740 J	0.0823	28.2
KMXX	8/22/2022	6:21	8.87	15.2	83.7	8.01	<1.0	0.649 J	0.178	22.4
IDBW	8/22/2022	6:33	8.61	11.7	83.6	8.00	<1.0	0.696 J	0.170	33.2
BDMZ	8/22/2022	7:24	18.2	6.80	86.5	7.49	<1.0	0.880 J	0.0834	63.9
BDXX	8/22/2022	7:08	14.4	6.40	86.1	7.51	<1.0	1.40 J	0.146	83.6
AMOG	8/22/2022	7:41	9.12	9.60	83.5	7.73	<1.0	1.30 J	0.117	41.4
AWGB	8/22/2022	8:28	8.11	6.10	83.4	7.77	<1.0	0.693 J	0.179	2.00
IDBW-DUP	8/22/2022	6:34	8.60	11.1	83.2	8.01	<1.0	0.496 J	0.177	35.4
IDBW-FB	8/22/2022	6:32	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0467	<1
KMXX	8/24/2022	8:39	2.18	1.60 J	82.7	7.81	<1.0	0.503 J	0.241	5.65
IDBW	8/24/2022	7:54	2.40	2.10	82.9	7.85	<1.0	0.538 J	0.192	9.15
BDMZ	8/24/2022	6:59	8.90	7.20	84.9	7.52	<1.0	0.514 J	0.137	63.8
BDXX	8/24/2022	6:53	8.66	7.50	84.7	7.43	<1.0	0.475 J	0.132	73.3
KMXX-DUP	8/24/2022	8:41	2.20	1.70 J	82.7	7.81	<1.0	0.458 J	0.177	11.8
KMXX-FB	8/24/2022	8:38	N/A	0.600 J	N/A	N/A	<1.0	<0.433	0.0458	<1
MCXX	8/29/2022	6:22	2.90	2.20	79.4	8.01	<1.0	<0.433	0.190	6.15
KMXX	8/29/2022	7:16	1.68	1.50 J	73.4	8.23	<1.0	0.449 J	0.212	2.00
IDBW	8/29/2022	7:52	1.46	2.00	71.4	8.10	<1.0	0.487 J	0.188	3.00
BDMZ	8/29/2022	8:57	7.80	9.84	77.7	7.92	<1.0	0.593 J	0.0768	71.2
BDXX	8/29/2022	9:18	6.24	7.38	79.7	7.90	<1.0	0.499 J	0.121	74.2
AMOG	8/29/2022	9:34	1.42	1.89 J	76.2	8.06	<1.0	<0.433	0.170	7.35
AWGB	8/29/2022	10:15	1.49	1.80 J	76.8	8.31	<1.0	1.33 J	0.244	3.55
KMXX-DUP	8/29/2022	7:18	1.68	1.20 J	73.4	8.22	<1.0	<0.433	0.219	2.50
KMXX-FB	8/29/2022	7:15	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0444	<1
KMXX	8/31/2022	6:52	1.66	5.10	71.6	8.16	<1.0	<0.433	0.170	3.55
IDBW	8/31/2022	6:29	1.42	5.10	72.1	8.22	<1.0	0.438 J	0.169	3.60
BDMZ	8/31/2022	8:00	8.92	7.20	74.9	7.84	<1.0	0.705 J	0.111	214
BDXX	8/31/2022	7:48	8.37	7.37	74.3	7.90	<1.0	0.454 J	0.0739	7.05
BDXX-DUP	8/31/2022	7:49	8.30	7.53	74.3	7.90	<1.0	1.16 J	0.0728	1.00
BDXX-FB	8/31/2022	7:46	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	9/6/2022	6:04	8.44	9.40	72.3	8.21	<1.0	0.555 J	0.226	3.05
KMXX	9/6/2022	6:22	9.01	21.5	72.1	8.20	<1.0	0.629 J	0.217	15.3
IDBW	9/6/2022	6:45	9.46	13.3	72.7	8.10	<1.0	0.508 J	0.200	13.5
BDMZ	9/6/2022	7:40	17.9	4.80	77.8	7.74	<1.0	0.507 J	0.153	140
BDXX	9/6/2022	7:23	18.3	6.20	76.7	7.80	<1.0	<0.433	0.0976	158
AMOG	9/6/2022	7:51	10.2	5.40	72.3	8.22	<1.0	0.530 J	0.194	44.4
AWGB	9/6/2022	8:34	9.11	2.20	72.0	8.19	<1.0	0.570 J	0.199	1.50
AWGB-DUP	9/6/2022	8:36	9.11	2.80	72.1	8.19	<1.0	0.554 J	0.196	6.70
AWGB-FB	9/6/2022	8:33	N/A	0.500 J	N/A	N/A	<1.0	<0.433	0.0617	<1
KMXX	9/7/2022	7:38	8.89	4.20	71.4	8.18	<1.0	0.654 J	0.233	6.15
IDBW	9/7/2022	7:07	8.93	6.20	71.3	8.24	<1.0	0.513 J	0.217	12.4
BDMZ	9/7/2022	8:29	14.6	7.50	75.4	7.70	<1.0	0.449 J	0.110	78.2
BDXX	9/7/2022	8:44	14.7	4.80	74.9	7.71	<1.0	0.493 J	0.123	47.8
BDMZ-DUP	9/7/2022	8:31	14.6	5.00	75.3	7.70	<1.0	0.514 J	0.117	47.8
BDMZ-FB	9/7/2022	8:28	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0386	<1
MCXX	9/14/2022	7:17	8.90	50.4	71.4	8.18	<1.0	1.18 J	0.183	13.6
KMXX	9/14/2022	6:57	8.92	16.8	71.1	8.27	<1.0	0.870 J	0.171	34.8
IDBW	9/14/2022	6:33	8.89	17.2	71.1	8.21	<1.0	0.676 J	0.158	7.25
BDMZ	9/14/2022	8:29	16.7	7.80	75.3	7.91	<1.0	0.849 J	0.104	93.8
BDXX	9/14/2022	8:16	16.6	7.60	75.5	7.84	<1.0	0.620 J	0.0960	135
AMOG	9/14/2022	8:54	9.10	15.2	71.3	8.22	<1.0	0.602 J	0.175	2.00
AWGB	9/14/2022	10:11	9.09	13.3	71.2	8.18	<1.0	0.564 J	0.152	113
AMOG-DUP	9/14/2022	8:56	9.10	16.5	71.3	8.22	<1.0	0.721 J	0.171	8.90
AMOG-FB	9/14/2022	8:52	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0309 J	<1
KMXX	9/15/2022	5:30	9.84	13.0	71.1	8.20	<1.0	0.647 J	0.191	4.15
IDBW	9/15/2022	5:49	9.87	6.52	71.3	8.24	<1.0	0.618 J	0.174	4.15
BDMZ	9/15/2022	6:18	16.0	9.60	74.5	7.92	<1.0	0.557 J	0.0936	82.3
BDXX	9/15/2022	6:27	15.9	6.40	74.6	7.89	<1.0	0.547 J	0.0932	87.2
IDBW-DUP	9/15/2022	5:50	9.87	6.81	71.3	8.23	<1.0	0.661 J	0.180	1.00
IDBW-FB	9/15/2022	6:48	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0142 J	<1.0

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	9/19/2022	7:59	9.02	2.55	71.1	8.07	<1.0	0.702 J	0.174	2.00
KMXX	9/19/2022	7:38	9.15	1.80 J	71.5	8.10	<1.0	0.618 J	0.180	4.10
IDBW	9/19/2022	7:12	8.90	2.60	71.4	8.07	<1.0	0.494 J	0.175	6.70
BDMZ	9/19/2022	7:35	18.9	11.8	75.1	7.65	<1.0	0.698 J	0.129	112
BDXX	9/19/2022	7:59	17.4	13.3	75.3	7.67	<1.0	0.706 J	0.127	177
AMOG	9/19/2022	7:00	8.71	2.70	71.2	8.19	<1.0	0.883 J	0.336	6.80
AWGB	9/19/2022	6:25	8.26	2.46	71.1	8.16	<1.0	0.796 J	0.295	6.30
BDXX-DUP	9/19/2022	8:12	17.4	12.5	75.3	7.67	<1.0	0.763 J	0.128	148
BDXX-FB	9/19/2022	7:58	N/A	<0.321	N/A	N/A	<1.0	0.435 J	0.0239 J	<1.0
KMXX	9/21/2022	6:59	5.11	3.40	70.8	8.14	<1.0	0.945 J	0.179	5.20
IDBW	9/21/2022	6:34	4.72	3.70	71.3	8.16	<1.0	0.769 J	0.159	1.00
BDMZ	9/21/2022	7:56	9.00	9.90	73.2	7.90	<1.0	0.743 J	0.0920	83.6
BDXX	9/21/2022	7:45	9.18	9.80	73.5	7.88	<1.0	0.728 J	0.0939	64.0
KMXX-DUP	9/21/2022	7:02	5.21	3.70	70.9	8.14	<1.0	0.592 J	0.162	3.55
KMXX-FB	9/21/2022	6:58	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0382	<1.0
MCXX	9/29/2022	6:47	17.3	17.7	77.0	7.64	<1.0	0.637 J	0.155	5.70
KMXX	9/29/2022	7:29	20.3	28.2	54.9	8.06	<1.0	0.755 J	0.136	6.85
IDBW	9/29/2022	8:18	16.7	22.4	59.7	8.40	<1.0	0.648 J	0.133	4.65
BDMZ	9/29/2022	9:36	5.94	6.38	65.8	7.38	<1.0	1.10 J	0.0983	46.0
BDXX	9/29/2022	9:56	6.95	7.24	67.6	7.76	<1.0	0.471 J	0.0955	46.1
AMOG	9/29/2022	10:24	6.72	8.00	64.6	7.91	<1.0	0.542 J	0.119	3.50
AWGB	9/29/2022	11:28	4.63	5.41	66.9	7.48	<1.0	0.611 J	0.128	2.00
BDMZ-DUP	9/29/2022	9:37	5.55	6.20	65.8	7.38	<1.0	0.670 J	0.104	77.8
BDMZ-FB	9/29/2022	9:35	N/A	<0.316	N/A	N/A	<1.0	<0.433	0.0150 J	<1
KMXX	9/30/2022	7:39	4.71	4.55	58.6	8.03	<1.0	0.721 J	0.144	1.00
IDBW	9/30/2022	7:08	4.28	4.69	58.8	7.74	<1.0	<0.433	0.156	1.00
BDMZ	9/30/2022	6:43	5.71	5.52	62.7	7.61	<1.0	0.572 J	0.0716	62.9
BDXX	9/30/2022	6:28	5.56	5.45	61.2	7.70	<1.0	1.78 J	0.0624	56.5
BDXX-DUP	9/30/2022	6:30	5.56	5.33	61.2	7.70	<1.0	0.621 J	0.0689	46.5
BDXX-FB	9/30/2022	6:27	N/A	<0.305	N/A	N/A	<1.0	<0.433	0.0395	<1

See last page for abbreviations and notes.

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (µg/L)	Chromium (µg/L)	Hex. Chromium (µg/L)	*E. coli* (MPN/100 mL)
MCXX	10/3/2022	8:44	3.16	11.0	63.5	7.99	<1.0	0.533 J	0.186	3.55
KMXX	10/3/2022	8:29	2.94	10.6	63.3	8.03	<1.0	0.709 J	0.167	2.55
IDBW	10/3/2022	8:05	3.01	8.20	63.5	8.02	<1.0	0.546 J	0.159	4.10
BDMZ	10/3/2022	6:37	5.09	6.40	64.8	7.58	<1.0	0.613 J	0.129	73.5
BDXX	10/3/2022	6:29	5.92	6.20	64.6	7.66	<1.0	0.444 J	0.128	53.4
AMOG	10/3/2022	6:53	4.16	8.90	63.3	8.04	<1.0	0.500 J	0.172	3.55
AWGB	10/3/2022	7:27	3.99	8.90	62.8	8.09	<1.0	0.537 J	0.158	7.25
IDBW-DUP	10/3/2022	8:07	3.01	10.7	63.5	8.02	<1.0	0.561 J	0.170	3.00
IDBW-FB	10/3/2022	8:04	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.101	<1
MCXX	11/9/2022	11:15	7.10	2.90	53.4	7.70	<1.0	0.962 J	0.147	1.50
KMXX	11/9/2022	11:52	7.10	4.20	52.7	7.70	<1.0	0.692 J	0.137	2.00
IDBW	11/9/2022	12:30	11.4	4.20	53.4	7.60	<1.0	0.495 J	0.147	1.00
BDMZ	11/9/2022	10:00	9.00	7.80	56.5	7.10	<1.0	0.981 J	0.0866	109
BDXX	11/9/2022	10:20	5.60	21.9	58.3	7.54	<1.0	0.681 J	0.0955	157
AMOG	11/9/2022	13:10	3.90	2.90	54.7	7.60	<1.0	0.586 J	0.155	9.60
AWGL	11/9/2022	8:50	2.80	3.10	54.7	7.70	<1.0	0.610 J	0.135	<1
KMXX-DUP	11/9/2022	11:52	7.10	4.30	52.7	7.70	<1.0	0.549 J	0.155	1.00
KMXX-FB	11/9/2022	11:50	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0215 J	<1
MCXX	12/12/2022	13:35	16.9	8.10	40.8	7.40	<1.0	1.03 J	0.153	13.1
KMXX	12/12/2022	12:58	17.0	9.10	41.6	7.50	<1.0	1.04 J	0.154	2.00
IDBW	12/12/2022	12:32	10.9	7.16	42.6	7.30	<1.0	1.40 J	0.159	<1
BDMZ	12/12/2022	11:26	7.28	5.40	47.6	7.50	<1.0	0.710 J	0.0699	28.8
BDXX	12/12/2022	11:08	7.64	6.43	48.6	7.50	<1.0	0.922 J	0.0626	76.2
AMOG	12/12/2022	12:05	4.95	4.40	42.5	7.52	<1.0	0.701 J	0.138	2.00
AWGL	12/12/2022	10:27	6.29	2.80	42.1	7.80	<1.0	0.820 J	0.145	<1
MCXX-DUP	12/12/2022	13:37	16.8	10.0	40.8	7.44	<1.0	0.997 J	0.144	14.6
MCXX-FB	12/12/2022	13:33	N/A	0.308 J	N/A	N/A	<1.0	<0.433	0.0274 J	<1

Abbreviations:

TSS = total suspended solids

Hex. Chromium = hexavalent chromium

N/A = not applicable

<#.# = not detected at the limit of detection shown

J = the analyte was detected, but below the limit of quantitation

DUP = field duplicate

FB = field blank

Locations:

AWGL = American Water Intake - Gary Lakeside Pump House

AWGB = American Water Intake - Breakwall

AMOG = American Water Intake - Ogden

BDXX = Burns Ditch

BDMZ = Burns Ditch / Lake Michigan Mixing Zone

IDBW = Indiana Dunes Beach - Western Area

KMXX = Kemil Beach

MCXX = Michigan City

Units:

NTU = nephelometric turbidity unit

mg/L = milligrams per liter

°C = degrees Celcius

s.u. = standard units

µg/L = micrograms per liter

MPN = Most Probable Number per 100 milliliters

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	1/9/2023	11:30	3.20	1.20 J	36.5	7.80	<1.0	0.797 J	0.174	<1
AWGL	1/9/2023	10:10	3.80	1.30 J	35.9	7.70	<1.0	0.466 J	0.180	<1
AWGL-DUP	1/9/2023	10:12	3.60	1.10 J	36.0	7.70	<1.0	0.600 J	0.179	<1
AWGL-FB	1/9/2023	10:08	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0284 J	<1
BDMZ	1/9/2023	11:00	5.30	9.44	40.9	7.60	<1.0	0.804 J	0.0885	167
BDXX	1/9/2023	10:45	6.20	8.40	42.6	7.30	<1.0	1.09 J	0.178	227
IDBW	1/9/2023	11:50	2.80	1.80 J	36.2	7.40	<1.0	0.499 J	0.178	<1
KMXX	1/9/2023	12:25	6.10	7.45	33.9	7.50	<1.0	0.609 J	0.173	<1
MCXX	1/9/2023	13:00	2.90	6.60	33.7	7.80	<1.0	0.556 J	0.177	<1
AMOG	2/20/2023	10:55	11.9	14.6	39.9	8.17	<1.0	1.02 J	0.176	<1
AMOG-DUP	2/20/2023	10:55	10.8	16.2	39.9	8.10	<1.0	1.02 J	0.169	<1
AMOG-FB	2/20/2023	10:55	N/A	1.01 J	N/A	N/A	<1.0	<0.433	0.0371 J	<1
AWGL	2/20/2023	9:10	6.70	8.00	39.7	8.20	<1.0	0.792 J	0.170	<1
BDMZ	2/20/2023	10:35	11.9	8.89	46.3	8.00	<1.0	0.836 J	0.0705	87.5
BDXX	2/20/2023	10:10	12.6	12.6	46.2	8.10	<1.0	0.943 J	0.0892	132
IDBW	2/20/2023	11:20	31.7	33.2	39.2	8.10	<1.0	1.33 J	0.157	2.50
KMXX	2/20/2023	11:40	37.4	32.6	39.0	8.20	<1.0	1.43 J	0.158	1.00
MCXX	2/20/2023	12:00	28.4	31.4	39.4	8.00	<1.0	1.26 J	0.163	<1
AMOG	3/15/2023	12:52	10.9	10.0	41.7	8.26	<1.0	0.861 J	0.151	1.50
AWGL	3/15/2023	10:40	10.9	8.90	46.7	8.10	<1.0	0.703 J	0.158	<1
BDMZ	3/15/2023	12:20	15.4	16.5	44.7	8.05	<1.0	0.801 J	0.0776	90.4
BDXX	3/15/2023	11:55	14.3	13.4	45.5	7.96	<1.0	0.927 J	0.0935	99.3
BDXX-DUP	3/15/2023	11:56	13.9	13.5	45.4	7.92	<1.0	1.39 J	N/A	84.0
BDXX-FB	3/15/2023	11:50	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0214 J	<1
IDBW	3/15/2023	13:35	19.7	14.9	42.6	8.15	<1.0	1.05 J	0.155	<1
KMXX	3/15/2023	14:02	25.5	19.5	44.4	8.17	<1.0	1.06 J	0.164	<1
MCXX	3/15/2023	14:37	31.9	24.4	45.5	8.20	<1.0	1.02 J	0.158	<1
AMOG	4/11/2023	11:05	7.15	6.90	55.4	8.20	<1.0	0.714 J	0.177	<1
AWGL	4/11/2023	9:00	3.60	2.80	49.3	8.10	<1.0	0.478 J	0.188	<1
BDMZ	4/11/2023	9:55	22.9	18.6	60.8	8.00	<1.0	1.29 J	0.0918	30.6
BDMZ-DUP	4/11/2023	9:55	24.6	19.0	61.8	8.07	<1.0	1.09 J	0.0947	105
BDMZ-FB	4/11/2023	9:00	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0393	<1
BDXX	4/11/2023	10:30	22.2	17.3	61.7	7.95	<1.0	1.06 J	0.0874	50.2
IDBW	4/11/2023	11:51	5.02	4.50	57.3	8.20	<1.0	0.537 J	0.176	<1
KMXX	4/11/2023	12:30	5.16	4.30	57.3	8.20	<1.0	0.476 J	0.190	<1
MCXX	4/11/2023	13:00	5.35	4.70	57.2	8.20	<1.0	0.526 J	0.195	<1

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	5/2/2023	11:53	8.00	9.00	48.2	8.20	<1.0	0.640 J	0.107	8.90
AWGL	5/2/2023	10:15	5.29	5.60	50.0	7.60	<1.0	0.438 J	0.117	<1
BDMZ	5/2/2023	11:28	7.82	10.2	49.4	8.10	<1.0	0.558 J	0.139	41.1
BDXX	5/2/2023	11:00	10.6	12.0	52.8	8.20	<1.0	0.581 J	0.082	217
IDBW	5/2/2023	12:28	13.4	10.8	47.4	8.13	<1.0	0.501 J	0.167	6.25
IDBW-DUP	5/2/2023	12:28	13.9	9.60	47.6	8.20	<1.0	0.753 J	0.170	3.55
IDBW-FB	5/2/2023	12:25	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
KMXX	5/2/2023	13:05	24.7	18.0	46.4	8.07	<1.0	0.791 J	0.167	3.00
MCXX	5/2/2023	13:36	20.4	20.2	46.0	8.10	<1.0	0.706 J	0.164	7.40
BDMZ	5/4/2023	12:11	10.4	10.4	59.9	8.40	<1.0	0.780 J	0.107	17.2
BDMZ-DUP	5/4/2023	12:19	10.0	9.00	59.7	8.20	<1.0	0.737 J	0.112	15.4
BDMZ-FB	5/4/2023	12:19	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.023 J	<1
BDXX	5/4/2023	12:34	11.6	12.2	58.2	8.20	<1.0	0.598 J	0.0837	21.6
IDBW	5/4/2023	11:25	28.0	19.2	53.9	8.10	<1.0	0.837 J	0.160	3.60
KMXX	5/4/2023	10:38	27.4	19.9	52.1	8.20	<1.0	0.818 J	0.156	5.15
AMOG	5/8/2023	10:50	4.07	5.90	50.2	7.90	<1.0	<0.433	0.149	5.70
AWGL	5/8/2023	11:44	4.34	3.10	48.5	7.40	<1.0	0.633 J	0.160	<1
BDMZ	5/8/2023	10:00	16.0	21.2	62.6	7.40	<1.0	0.814 J	0.0702	45.8
BDXX	5/8/2023	10:23	15.3	19.2	61.6	7.90	<1.0	0.666 J	0.0595	50.4
IDBW	5/8/2023	9:25	9.60	8.40	51.2	7.80	<1.0	0.587 J	0.160	25.2
KMXX	5/8/2023	8:35	10.5	9.70	50.9	8.00	<1.0	<0.433	0.159	11.3
KMXX-DUP	5/8/2023	8:40	10.6	12.4	50.9	8.00	<1.0	2.17	0.163	5.05
KMXX-FB	5/8/2023	8:44	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0256 J	<1
MCXX	5/8/2023	8:05	18.9	25.6	51.2	7.90	<1.0	0.792 J	0.163	23.5
BDMZ	5/10/2023	10:17	10.8	11.1	66.3	8.20	<1.0	0.740 J	0.0916	18.3
BDXX	5/10/2023	10:40	14.0	12.6	66.4	8.00	<1.0	0.761 J	0.0866	20.9
IDBW	5/10/2023	9:05	8.70	2.80	54.6	8.10	<1.0	1.34 J	0.161	<1
IDBW-DUP	5/10/2023	9:15	8.60	2.10	54.6	8.10	<1.0	0.493 J	0.161	<1
IDBW-FB	5/10/2023	9:20	N/A	0.300 J	N/A	N/A	<1.0	0.507 J	0.0276 J	<1
KMXX	5/10/2023	8:30	3.30	1.80 J	52.7	8.20	<1.0	0.537 J	0.155	12.6
AMOG	5/15/2023	9:41	12.0	13.7	57.3	8.21	<1.0	0.603 J	0.142	49.1
AWGL	5/15/2023	11:30	2.76	1.80 J	56.6	8.19	<1.0	<0.433	0.171	<1
BDMZ	5/15/2023	10:20	11.0	12.5	65.3	8.12	<1.0	0.533 J	0.0811	98.1
BDXX	5/15/2023	10:44	12.4	14.2	66.5	8.13	<1.0	0.724 J	0.0776	118
IDBW	5/15/2023	9:09	9.30	7.40	53.9	8.02	<1.0	0.437 J	0.161	11.5
KMXX	5/15/2023	8:44	14.1	11.4	52.1	7.97	<1.0	0.572 J	0.162	11.7
MCXX	5/15/2023	8:21	34.8	22.9	51.8	7.97	<1.0	0.727 J	0.163	6.80
MCXX-DUP	5/15/2023	8:22	34.1	24.0	51.7	8.01	<1.0	0.822 J	0.175	7.30
MCXX-FB	5/15/2023	8:25	N/A	<0.303	N/A	N/A	<1.0	<0.433	0.0221 J	<1

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	5/17/2023	9:05	14.3	14.3	64.9	7.73	<1.0	0.937 J	0.0924	40.7
BDXX	5/17/2023	9:24	17.3	17.7	65.4	7.80	<1.0	0.784 J	0.0669	96.4
IDBW	5/17/2023	8:31	21.4	22.7	57.2	7.90	<1.0	0.931 J	0.163	16.4
KMXX	5/17/2023	8:00	28.2	23.4	56.8	7.90	<1.0	1.15 J	0.152	14.7
KMXX-DUP	5/17/2023	8:05	28.3	25.7	56.7	7.90	<1.0	1.05 J	0.154	19.8
KMXX-FB	5/17/2023	8:07	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0221 J	<1
AMOG	5/22/2023	7:55	2.05	1.50 J	56.8	8.19	<1.0	0.503 J	0.159	<1
AWGL	5/22/2023	11:55	4.76	1.40 J	59.7	7.96	<1.0	0.841 J	0.166	<1
AWGL-DUP	5/22/2023	12:05	4.77	1.30 J	59.7	7.95	<1.0	0.580 J	0.164	<1
AWGL-FB	5/22/2023	12:10	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
BDMZ	5/22/2023	8:40	8.72	9.70	69.2	8.19	<1.0	0.836 J	0.0786	13.2
BDXX	5/22/2023	8:59	11.7	13.0	70.4	8.14	<1.0	0.755 J	0.0653	36.7
IDBW	5/22/2023	7:18	3.12	2.40	55.4	7.88	<1.0	0.547 J	0.147	2.50
KMXX	5/22/2023	6:44	2.51	2.10	54.3	7.86	<1.0	0.605 J	0.152	5.60
MCXX	5/22/2023	6:21	3.02	3.30	53.2	7.61	<1.0	0.531 J	0.158	1.00
BDMZ	5/24/2023	7:53	8.10	9.30	57.4	8.20	<1.0	0.831 J	0.0555	36.4
BDXX	5/24/2023	8:08	9.84	11.9	69.0	8.20	<1.0	0.783 J	0.0685	74.9
BDXX-DUP	5/24/2023	8:12	9.86	12.9	69.0	8.20	<1.0	0.766 J	0.0671	30.8
BDXX-FB	5/24/2023	8:15	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
IDBW	5/24/2023	6:34	2.13	3.20	54.1	7.80	<1.0	0.557 J	0.134	<1
KMXX	5/24/2023	6:07	2.46	3.60	53.4	7.80	<1.0	0.761 J	0.127	<1
AMOG	5/30/2023	7:47	3.36	5.40	63.3	8.10	<1.0	0.520 J	0.164	6.25
AMOG-DUP	5/30/2023	7:47	3.29	5.00	63.3	8.10	<1.0	0.612 J	0.158	3.05
AMOG-FB	5/30/2023	7:45	N/A	<0.316	N/A	N/A	<1.0	<0.433	<0.0130	<1
AWGL	5/30/2023	9:10	4.23	1.30 J	61.2	7.70	<1.0	0.522 J	0.157	<1
BDMZ	5/30/2023	8:04	8.52	7.70	71.4	8.30	<1.0	0.769 J	0.0791	54.8
BDXX	5/30/2023	8:22	12.9	10.1	71.0	8.20	<1.0	0.735 J	0.0506	29.6
IDBW	5/30/2023	7:14	3.44	3.30	58.6	7.90	<1.0	0.532 J	0.144	<1
KMXX	5/30/2023	6:50	2.97	1.40 J	57.9	7.80	<1.0	0.626 J	0.142	<1
MCXX	5/30/2023	6:25	3.22	3.00	58.2	7.90	<1.0	0.484 J	0.151	2.00
BDMZ	5/31/2023	10:42	9.92	11.6	76.6	8.14	<1.0	0.735 J	0.075	39.4
BDMZ-DUP	5/31/2023	10:45	9.92	16.5	76.6	8.13	<1.0	0.658 J	0.0797	38.0
BDMZ-FB	5/31/2023	10:47	N/A	0.933 J	N/A	N/A	<1.0	<0.433	0.019 J	<1
BDXX	5/31/2023	10:30	8.92	11.7	82.5	8.23	<1.0	0.902 J	0.0784	60.4
IDBW	5/31/2023	12:10	3.48	8.67	65.8	7.94	<1.0	0.520 J	0.169	<1
KMXX	5/31/2023	12:35	3.31	13.1	64.9	7.93	<1.0	1.63 J	0.171	2.00

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	6/5/2023	10:12	4.67	8.60	68.5	8.13	<1.0	<0.433	0.166	8.00
AWGL	6/5/2023	12:35	1.51	1.60 J	66.3	7.77	<1.0	<0.433	0.147	25.4
BDMZ	6/5/2023	11:30	8.24	9.40	76.2	7.97	<1.0	<0.433	0.0956	25.1
BDXX-FB	6/5/2023	11:05	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0157 J	<1
BDXX	6/5/2023	10:55	10.9	12.3	74.6	7.96	<1.0	<0.433	0.0712	35.9
BDXX-DUP	6/5/2023	11:00	10.7	12.6	75.7	8.10	<1.0	0.446 J	0.082	62.5
IDBW	6/5/2023	9:30	2.42	1.70 J	67.1	8.13	<1.0	<0.433	0.161	6.15
KMXX	6/5/2023	8:48	4.86	3.70	62.2	8.05	<1.0	<0.433	0.174	11.3
MCXX	6/5/2023	8:15	15.5	9.00	59.9	8.02	<1.0	<0.433	0.166	16.4
BDMZ	6/7/2023	8:39	8.39	15.0	76.8	7.98	<1.0	0.691 J	0.0229 J	47.7
BDXX	6/7/2023	8:43	11.1	14.5	75.5	8.03	<1.0	0.913 J	0.0348 J	49.2
IDBW	6/7/2023	6:48	6.75	5.70	64.0	7.81	<1.0	0.539 J	0.153	22.8
IDBW-DUP	6/7/2023	6:55	6.75	6.60	64.1	7.82	<1.0	0.697 J	0.145	28.0
IDBW-FB	6/7/2023	7:00	N/A	0.529 J	N/A	N/A	<1.0	<0.433	<0.0130	<1
KMXX	6/7/2023	6:12	14.2	14.9	63.1	7.55	<1.0	0.612 J	0.146	6.15
AMOG	6/13/2023	7:32	8.51	9.80	61.5	7.90	<1.0	1.05 J	0.156	11.8
AWGB	6/13/2023	8:55	8.36	4.00	64.4	8.00	<1.0	1.16 J	0.590	48.5
BDMZ	6/13/2023	8:00	11.7	9.20	69.6	8.01	<1.0	0.621 J	0.0933	24.0
BDMZ-DUP	6/13/2023	8:05	11.8	9.70	69.7	8.02	<1.0	0.843 J	0.0904	78.6
BDMZ-FB	6/13/2023	8:10	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1
BDXX	6/13/2023	8:21	17.7	10.3	68.0	8.00	<1.0	0.777 J	0.0587	62.3
IDBW	6/13/2023	7:03	14.9	12.2	60.9	7.80	<1.0	0.761 J	0.161	37.6
KMXX	6/13/2023	6:39	23.6	19.4	61.1	7.90	<1.0	0.911 J	0.144	36.2
MCXX	6/13/2023	6:15	22.4	21.2	61.5	7.90	<1.0	0.894 J	0.168	4.60
BDMZ	6/14/2023	8:12	12.1	10.9	69.7	8.02	<1.0	0.801 J	0.0965	861
BDXX	6/14/2023	8:29	17.8	13.8	68.7	8.05	<1.0	1.58 J	0.101	1,150
IDBW	6/14/2023	6:35	10.3	5.80	62.2	7.90	<1.0	0.676 J	0.159	15.2
KMXX	6/14/2023	5:58	11.5	6.50	61.5	7.82	<1.0	0.741 J	0.158	6.95
KMXX-DUP	6/14/2023	6:03	11.6	7.00	61.5	7.82	<1.0	0.718 J	0.177	9.4
KMXX-FB	6/14/2023	6:07	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0174 J	<1
AMOG	6/19/2023	7:18	3.50	2.80	64.4	7.90	<1.0	0.445 J	0.171	2.55
AWGB	6/19/2023	8:26	4.40	1.10 J	63.6	7.80	<1.0	0.537 J	0.161	<1
BDMZ	6/19/2023	7:33	9.40	8.40	72.1	8.40	<1.0	0.812 J	0.122	56.2
BDXX	6/19/2023	7:43	11.7	9.40	73.7	8.50	<1.0	0.563 J	0.0831	109
IDBW	6/19/2023	7:00	3.80	3.00	61.9	7.90	<1.0	<0.433	0.168	1.00
IDBW-DUP	6/19/2023	7:00	3.60	3.10	61.8	7.90	<1.0	0.435 J	0.158	2.50
IDBW-FB	6/19/2023	6:58	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0212 J	<1
KMXX	6/19/2023	6:31	3.70	3.10	62.2	7.80	<1.0	<0.433	0.152	3.00
MCXX	6/19/2023	6:10	4.00	3.50	62.0	7.70	<1.0	1.75 J	0.163	2.00

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	6/21/2023	8:34	11.5	11.3	77.5	8.31	<1.0	0.868 J	0.0774	59.1
BDXX	6/21/2023	8:46	16.1	12.9	78.2	8.20	<1.0	0.642 J	0.0893	42.5
BDXX-DUP	6/21/2023	8:50	20.2	11.5	78.3	8.21	<1.0	0.660 J	0.0931	99.2
BDXX-FB	6/21/2023	8:50	N/A	0.500 J	N/A	N/A	<1.0	<0.433	0.0228 J	<1
IDBW	6/21/2023	6:41	7.70	7.10	64.1	7.50	<1.0	0.569 J	0.160	4.10
KMXX	6/21/2023	6:05	12.4	9.00	63.1	7.70	<1.0	0.581 J	0.153	14.2
AMOG	6/26/2023	7:49	5.28	2.80	67.6	8.00	<1.0	<0.433	0.151	17.1
AWGB	6/26/2023	9:15	4.85	1.20 J	68.5	7.80	<1.0	<0.433	0.153	7.90
BDMZ	6/26/2023	8:23	13.0	10.8	75.0	7.80	<1.0	<0.433	0.0771	1,210
BDXX	6/26/2023	8:45	23.7	14.3	75.2	7.90	<1.0	<0.433	0.0882	1,920
IDBW	6/26/2023	7:14	13.9	4.00	67.1	7.90	<1.0	<0.433	0.163	15.1
KMXX	6/26/2023	6:40	23.8	16.1	66.7	8.10	<1.0	<0.433	0.165	23.4
KMXX-DUP	6/26/2023	6:50	23.1	13.7	66.7	8.10	<1.0	<0.433	0.161	19.1
KMXX-FB	6/26/2023	6:55	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0239 J	<1
MCXX	6/26/2023	6:10	22.8	17.3	66.8	7.90	<1.0	<0.433	0.151	27.6
BDMZ	6/28/2023	8:20	20.2	11.4	73.5	7.80	<1.0	0.995 J	0.070	186
BDMZ-DUP	6/28/2023	8:27	20.1	11.1	73.4	7.80	<1.0	0.853 J	0.071	225
BDMZ-FB	6/28/2023	8:27	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0175 J	<1
BDXX	6/28/2023	8:35	26.8	14.8	72.5	7.70	<1.0	0.706 J	0.0617	275
IDBW	6/28/2023	6:45	22.5	17.8	64.0	7.76	<1.0	0.655 J	0.166	14.3
KMXX	6/28/2023	6:05	21.1	22.2	63.6	7.81	<1.0	0.844 J	0.150	22.8
AMOG	7/3/2023	7:51	6.96	7.00	67.6	8.70	<1.0	0.653 J	0.106	46.1
AWGB	7/3/2023	8:33	5.50	1.80 J	69.6	8.70	<1.0	0.619 J	0.117	16.8
BDMZ	7/3/2023	7:25	11.2	8.30	75.2	8.20	<1.0	1.88 J	0.596	495
BDXX	7/3/2023	7:35	14.1	9.70	73.9	8.20	<1.0	0.587 J	0.0318 J	437
IDBW	7/3/2023	6:55	10.9	11.5	68.9	8.60	<1.0	0.551 J	0.124	8.40
KMXX	7/3/2023	6:30	9.40	4.70	68.5	8.60	<1.0	0.588 J	0.119	10.6
MCXX	7/3/2023	6:10	10.0	9.02	68.3	8.70	<1.0	0.722 J	0.160	9.85
MCXX-DUP	7/3/2023	6:10	10.3	5.20	68.3	8.60	<1.0	0.577 J	0.161	6.70
MCXX-FB	7/3/2023	6:08	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
BDMZ	7/4/2023	7:45	8.40	10.9	77.9	8.10	<1.0	0.642 J	0.015 J	188
BDXX	7/4/2023	7:59	7.80	12.3	77.7	8.30	<1.0	0.993 J	0.0328 J	192
IDBW	7/4/2023	7:08	6.70	2.80	69.1	7.90	<1.0	0.569 J	0.158	3.55
IDBW-DUP	7/4/2023	7:08	6.50	2.40	69.0	7.90	<1.0	<0.433	0.156	1.00
IDBW-FB	7/4/2023	7:06	N/A	<0.300	N/A	N/A	<1.0	0.500 J	<0.0130	<1
KMXX	7/4/2023	6:35	5.30	2.20	69.4	7.80	<1.0	0.651 J	0.114	3.55

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	7/10/2023	7:21	2.94	2.10	68.9	8.21	<1.0	0.591 J	0.115	5.70
AWGB	7/10/2023	8:35	3.32	1.10 J	71.1	8.23	<1.0	0.499 J	0.168	1.00
AWGB-DUP	7/10/2023	8:40	3.31	1.10 J	71.0	8.22	<1.0	0.555 J	0.175	3.00
AWGB-FB	7/10/2023	8:45	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
BDMZ	7/10/2023	7:58	20.2	9.80	75.1	7.93	<1.0	0.665 J	0.0311 J	123
BDXX	7/10/2023	7:41	13.6	9.10	74.6	8.06	<1.0	0.922 J	0.0294 J	183
IDBW	7/10/2023	6:54	3.65	3.00	68.0	8.23	<1.0	0.589 J	0.114	4.05
KMXX	7/10/2023	6:32	4.27	2.30	67.1	7.90	<1.0	0.554 J	0.111	10.3
MCXX	7/10/2023	6:15	5.96	2.80	67.2	7.90	<1.0	0.853 J	0.116	1.00
BDMZ	7/12/2023	8:20	19.7	12.9	75.8	8.03	<1.0	0.939 J	0.106	713
BDXX	7/12/2023	8:00	21.2	13.4	76.4	8.02	<1.0	0.996 J	0.0632	377
IDBW	7/12/2023	6:05	5.41	11.9	68.3	8.10	<1.0	0.624 J	0.160	10.2
KMXX	7/12/2023	5:45	6.27	13.9	67.4	7.90	<1.0	0.720 J	0.166	16.6
KMXX-DUP	7/12/2023	5:55	6.20	11.8	67.3	7.90	<1.0	0.943 J	0.164	14.1
KMXX-FB	7/12/2023	5:55	N/A	0.400 J	N/A	N/A	<1.0	<0.433	0.0218 J	<1
AMOG	7/17/2023	9:11	5.31	5.60	71.7	8.35	<1.0	<0.433	0.157	12.3
AMOG-DUP	7/17/2023	9:19	3.29	5.20	71.9	8.30	<1.0	0.474 J	0.159	16.6
AMOG-FB	7/17/2023	9:20	N/A	0.300 J	N/A	N/A	<1.0	<0.433	<0.0130	<1
AWGB	7/17/2023	10:37	1.87	2.50	78.0	8.30	<1.0	0.462 J	0.153	29.2
BDMZ	7/17/2023	8:21	12.8	9.50	76.6	7.90	<1.0	0.803 J	0.0841	376
BDXX	7/17/2023	8:44	18.1	14.6	77.0	7.90	<1.0	0.717 J	0.0749	292
IDBW	7/17/2023	7:45	5.88	3.50	71.6	8.30	<1.0	0.521 J	0.164	6.30
KMXX	7/17/2023	7:12	14.3	10.9	70.1	8.30	<1.0	0.480 J	0.156	74.4
MCXX	7/17/2023	6:50	13.4	6.90	70.1	7.80	<1.0	0.658 J	0.158	2.55
BDMZ	7/19/2023	7:58	12.4	11.8	76.4	8.00	<1.0	0.796 J	0.0671	73.9
BDXX	7/19/2023	8:15	18.7	8.60	76.2	7.90	<1.0	0.538 J	0.0668	98.0
BDXX-DUP	7/19/2023	8:25	18.8	10.2	76.4	8.00	<1.0	0.752 J	0.0622	108
BDXX-FB	7/19/2023	8:30	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
IDBW	7/19/2023	6:30	5.22	4.70	66.5	8.10	<1.0	0.442 J	0.156	5.10
KMXX	7/19/2023	6:00	4.12	3.30	66.2	7.90	<1.0	0.554 J	0.152	7.80
AMOG	7/24/2023	7:34	3.76	2.50	71.4	8.20	<1.0	0.437 J	0.152	31.0
AWGB	7/24/2023	9:00	5.96	5.10	78.0	8.10	<1.0	1.17 J	0.163	4.70
BDMZ	7/24/2023	8:03	17.9	10.6	78.6	8.00	<1.0	0.816 J	0.121	641
BDXX	7/24/2023	8:18	34.2	14.0	77.7	7.73	<1.0	0.947 J	0.110	702
BDXX-DUP	7/24/2023	8:22	34.5	13.3	77.6	7.75	<1.0	1.09 J	0.108	589
BDXX-FB	7/24/2023	8:26	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0184 J	<1
IDBW	7/24/2023	7:03	3.06	1.70 J	71.5	8.10	<1.0	<0.433	0.165	5.10
KMXX	7/24/2023	6:36	2.50	2.30	71.4	8.20	<1.0	0.548 J	0.165	2.55
MCXX	7/24/2023	6:15	3.32	1.70 J	70.5	8.10	<1.0	7.17	0.163	7.25

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	7/26/2023	7:55	14.2	11.3	78.4	8.00	<1.0	0.658 J	0.0907	111
BDMZ-DUP	7/26/2023	7:57	14.3	12.0	78.3	8.00	<1.0	0.718 J	0.0942	70.4
BDMZ-FB	7/26/2023	7:58	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0261 J	<1
BDXX	7/26/2023	8:05	17.3	6.60	78.8	8.10	<1.0	0.537 J	0.0775	93.3
IDBW	7/26/2023	6:15	3.03	1.90 J	72.8	7.90	<1.0	<0.433	0.163	3.05
KMXX	7/26/2023	5:50	2.45	1.60 J	72.6	7.90	<1.0	<0.433	0.166	<1
AMOG	7/31/2023	8:21	18.6	11.5	72.8	7.81	<1.0	0.880 J	0.144	55.4
AWGB	7/31/2023	9:50	6.57	5.00	78.0	7.14	<1.0	1.07 J	0.172	2.00
BDMZ	7/31/2023	8:47	17.1	10.9	76.8	7.53	<1.0	0.575 J	0.0551	209
BDMZ-DUP	7/31/2023	8:50	17.1	10.9	76.7	7.52	<1.0	0.704 J	0.0791	167
BDMZ-FB	7/31/2023	8:50	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0183 J	<1
BDXX	7/31/2023	9:10	26.9	18.3	76.8	7.38	<1.0	0.757 J	0.0484	218
IDBW	7/31/2023	7:45	8.49	4.70	72.5	7.88	<1.0	0.456 J	0.161	3.10
KMXX	7/31/2023	7:15	27.7	20.3	69.2	7.54	<1.0	0.815 J	0.147	41.6
MCXX	7/31/2023	6:50	22.5	10.9	68.3	7.47	<1.0	0.770 J	0.148	62.2
BDMZ	8/2/2023	8:47	11.6	10.2	77.9	7.83	<1.0	0.699 J	0.0643	108
BDXX	8/2/2023	9:00	13.0	10.1	78.9	7.85	<1.0	1.07 J	0.0544	114
IDBW	8/2/2023	6:52	3.84	1.60 J	69.9	8.03	<1.0	<0.433	0.168	3.05
IDBW-DUP	8/2/2023	6:58	3.83	1.00 J	69.8	8.03	<1.0	0.512 J	0.167	<1
IDBW-FB	8/2/2023	6:58	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0339 J	<1
KMXX	8/2/2023	6:18	3.33	1.80 J	71.6	8.12	<1.0	<0.433	0.136	5.15
AMOG	8/8/2023	8:53	8.12	10.8	72.3	8.05	<1.0	<0.433	0.161	30.5
AWGB	8/8/2023	10:42	1.34	2.00	76.4	8.17	<1.0	<0.433	0.163	4.55
BDMZ	8/8/2023	9:25	12.4	11.8	75.5	7.86	<1.0	<0.433	0.0607	251
BDXX	8/8/2023	9:38	14.1	13.1	77.0	7.74	<1.0	1.10 J	0.0684	270
IDBW	8/8/2023	7:01	4.96	5.80	69.8	8.10	<1.0	<0.433	0.152	8.90
IDBW-DUP	8/8/2023	8:10	4.27	6.00	70.3	8.30	<1.0	0.690 J	0.147	7.80
IDBW-FB	8/8/2023	8:10	N/A	0.600 J	N/A	N/A	<1.0	0.542 J	0.0212 J	<1
KMXX	8/8/2023	7:18	11.1	7.90	67.4	7.99	<1.0	<0.433	0.159	10.6
MCXX	8/8/2023	6:50	16.5	11.0	65.4	7.73	<1.0	<0.433	0.160	11.9
BDMZ	8/9/2023	8:38	12.1	10.1	75.3	7.76	<1.0	1.92 J	0.0694	138
BDXX	8/9/2023	8:50	16.4	16.1	75.3	7.71	<1.0	1.66 J	0.0655	184
IDBW	8/9/2023	7:00	3.57	2.90	70.5	7.80	<1.0	1.66 J	0.151	1.50
KMXX	8/9/2023	6:35	6.37	3.50	69.8	7.50	<1.0	1.12 J	0.147	40.0
KMXX-DUP	8/9/2023	6:37	6.38	4.50	69.7	7.60	<1.0	0.625 J	0.159	3.05
KMXX-FB	8/9/2023	6:41	N/A	<0.300	N/A	N/A	<1.0	0.857 J	<0.0130	<1

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	8/14/2023	8:13	11.9	2.00	71.9	7.72	<1.0	0.660 J	0.152	33.8
AWGB	8/14/2023	9:33	3.97	14.8	72.6	7.93	<1.0	0.544 J	0.151	1.00
BDMZ	8/14/2023	8:40	11.2	10.7	76.1	7.70	<1.0	0.712 J	0.0938	88.0
BDXX	8/14/2023	8:52	15.8	16.0	75.0	7.72	<1.0	0.866 J	0.0833	165
IDBW	8/14/2023	7:44	8.75	8.30	71.7	7.76	<1.0	0.601 J	0.167	4.05
KMXX	8/14/2023	7:10	16.5	8.50	71.1	7.86	<1.0	0.594 J	0.167	6.45
KMXX-DUP	8/14/2023	7:17	16.3	12.8	71.1	7.85	<1.0	0.679 J	0.157	2.50
KMXX-FB	8/14/2023	7:15	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0133 J	<1
MCXX	8/14/2023	6:50	17.1	9.50	71.2	7.73	<1.0	0.662 J	0.165	6.15
BDMZ	8/16/2023	8:31	25.5	17.8	71.3	7.73	<1.0	1.34 J	0.0988	2,110
BDXX	8/16/2023	8:40	35.7	20.6	71.7	7.80	<1.0	1.29 J	0.0634	2,240
BDXX-DUP	8/16/2023	8:45	35.6	21.2	71.7	7.80	<1.0	1.27 J	0.070	1,620
BDXX-FB	8/16/2023	8:45	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0152 J	<1
IDBW	8/16/2023	6:43	33.8	36.6	67.6	8.10	<1.0	1.14 J	0.142	60.8
KMXX	8/16/2023	6:14	37.0	36.8	68.0	8.10	<1.0	1.22 J	0.140	49.8
AMOG	8/21/2023	7:57	19.9	9.51	72.5	8.00	<1.0	1.34 J	0.148	119
AWGB	8/21/2023	8:55	9.70	3.00	73.4	8.00	<1.0	0.663 J	0.168	5.20
BDMZ	8/21/2023	8:22	13.1	11.8	76.1	7.80	<1.0	0.541 J	0.107	70.0
BDXX	8/21/2023	8:40	12.7	11.6	79.1	7.80	<1.0	1.79 J	0.121	92.6
IDBW	8/21/2023	7:25	14.9	10.3	72.1	8.00	<1.0	0.769 J	0.163	84.6
KMXX	8/21/2023	6:45	22.6	16.3	71.7	7.90	<1.0	0.502 J	0.162	24.8
MCXX	8/21/2023	6:20	21.7	21.9	71.2	7.90	<1.0	0.642 J	0.169	19.3
MCXX-DUP	8/21/2023	6:22	21.6	19.7	71.0	7.90	<1.0	1.16 J	0.178	27.8
MCXX-FB	8/21/2023	6:24	N/A	0.400 J	N/A	N/A	<1.0	0.437 J	0.0259 J	<1
BDMZ	8/23/2023	7:27	11.5	7.70	77.0	7.60	<1.0	1.10 J	0.0934	27.7
BDMZ-DUP	8/23/2023	7:30	11.6	8.40	77.1	7.60	<1.0	1.20 J	0.0991	41.2
BDMZ-FB	8/23/2023	7:30	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0311 J	<1
BDXX	8/23/2023	7:48	12.9	11.0	79.1	7.57	<1.0	1.37 J	0.101	61.4
IDBW	8/23/2023	6:30	3.39	3.00	71.4	7.43	<1.0	0.654 J	0.161	2.00
KMXX	8/23/2023	6:07	3.62	3.20	71.1	7.44	<1.0	<0.433	0.164	3.50
AMOG	8/28/2023	7:43	8.73	12.0	70.3	7.66	<1.0	0.578 J	0.140	3.55
AWGB	8/28/2023	10:20	3.64	2.10	76.6	7.60	<1.0	0.673 J	0.154	1.00
AWGB-DUP	8/28/2023	10:23	3.63	3.00	76.5	7.60	<1.0	0.571 J	0.145	<1
AWGB-FB	8/28/2023	10:25	N/A	0.600 J	N/A	N/A	<1.0	<0.433	0.0154 J	<1
BDMZ	8/28/2023	8:11	13.6	12.1	76.1	7.98	<1.0	0.771 J	0.0877	14.2
BDXX	8/28/2023	8:20	12.3	8.40	77.5	8.05	<1.0	0.663 J	0.0844	55.0
IDBW	8/28/2023	7:14	21.9	18.0	68.7	7.51	<1.0	0.737 J	0.158	6.45
KMXX	8/28/2023	6:45	26.4	18.5	69.1	7.43	<1.0	0.789 J	0.148	12.3
MCXX	8/28/2023	6:20	24.7	19.7	67.8	7.30	<1.0	0.825 J	0.139	20.2

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
BDMZ	8/29/2023	10:41	8.99	8.20	78.9	7.80	<1.0	1.20 J	0.302	25.2
BDXX	8/29/2023	10:25	9.71	10.4	78.8	7.71	<1.0	0.953 J	0.0782	48.5
IDBW	8/29/2023	11:40	8.07	5.60	76.4	7.92	<1.0	0.582 J	0.154	1.50
IDBW-DUP	8/29/2023	11:50	8.11	6.60	76.4	7.94	<1.0	0.669 J	0.156	<1
IDBW-FB	8/29/2023	11:55	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0162 J	<1
KMXX	8/29/2023	12:10	10.9	8.60	74.6	8.03	<1.0	0.607 J	0.156	4.00
AMOG	9/5/2023	8:05	2.01	2.63	74.9	7.71	<1.0	<0.433	0.117	2.00
AMOG-DUP	9/5/2023	8:08	2.02	2.20	74.9	7.73	<1.0	0.695 J	0.116	<1
AMOG-FB	9/5/2023	8:09	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0189 J	<1
AWGB	9/5/2023	9:15	2.28	2.00	75.7	8.13	<1.0	<0.433	0.145	1.00
BDMZ	9/5/2023	8:30	6.65	6.20	78.9	7.70	<1.0	0.494 J	0.0785	10.7
BDXX	9/5/2023	8:46	8.32	6.50	80.6	8.06	<1.0	0.693 J	0.0833	41.4
IDBW	9/5/2023	7:31	2.25	2.20	73.5	7.61	<1.0	<0.433	0.144	1.50
KMXX	9/5/2023	6:50	1.62	1.96 J	73.9	7.71	<1.0	<0.433	0.134	11.8
MCXX	9/5/2023	6:30	1.68	2.40	74.6	7.50	<1.0	<0.433	0.133	1.00
BDMZ	9/6/2023	8:30	6.64	8.10	77.7	8.10	<1.0	1.43 J	0.462	26.1
BDXX	9/6/2023	8:50	7.55	5.90	77.3	8.05	<1.0	0.488 J	0.0976	54.2
IDBW	9/6/2023	9:28	2.55	3.00	73.5	8.30	<1.0	<0.433	0.158	9.55
KMXX	9/6/2023	10:00	1.91	2.30	73.9	8.30	<1.0	<0.433	0.171	84.9
KMXX-DUP	9/6/2023	10:05	1.91	2.10	73.8	8.31	<1.0	<0.433	0.150	113
KMXX-FB	9/6/2023	10:07	N/A	<0.300	N/A	N/A	<1.0	0.697 J	0.0333 J	<1
AMOG	9/11/2023	8:00	3.18	5.30	68.3	7.71	<1.0	0.543 J	0.120	3.10
AWGB	9/11/2023	8:44	4.48	2.80	71.1	7.24	<1.0	0.538 J	0.118	3.00
BDMZ	9/11/2023	8:28	8.32	8.40	72.6	7.61	<1.0	1.24 J	0.560	32.0
BDXX	9/11/2023	8:37	9.41	8.10	69.5	7.73	<1.0	0.610 J	0.0437	61.2
BDXX-DUP	9/11/2023	8:38	9.44	7.20	69.5	7.74	<1.0	0.627 J	0.0375	30.6
BDXX-FB	9/11/2023	8:41	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
IDBW	9/11/2023	7:19	2.21	3.10	68.8	7.56	<1.0	0.438 J	0.130	3.05
KMXX	9/11/2023	6:48	2.43	3.30	70.1	7.88	<1.0	<0.433	0.135	3.55
MCXX	9/11/2023	6:28	2.61	3.70	69.4	7.84	<1.0	<0.433	0.127	4.55
BDMZ	9/13/2023	8:10	11.6	9.00	70.8	8.04	<1.0	0.986 J	0.0838	194
BDXX	9/13/2023	8:19	17.1	8.10	69.4	7.88	<1.0	0.645 J	0.0454	162
BDXX-DUP	9/13/2023	8:22	17.2	8.90	69.4	7.89	<1.0	0.743 J	0.0449	214
BDXX-FB	9/13/2023	8:23	N/A	<0.300	N/A	N/A	<1.0	<0.433	<0.0130	<1
IDBW	9/13/2023	6:46	19.7	15.5	63.4	7.42	<1.0	0.751 J	0.114	7.30
KMXX	9/13/2023	6:15	20.7	19.4	65.4	7.51	<1.0	0.730 J	0.103	21.9

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	9/19/2023	8:18	3.46	3.80	65.4	8.21	<1.0	0.561 J	0.0978	11.2
AWGB	9/19/2023	10:00	4.06	3.00	67.1	8.23	<1.0	0.764 J	0.099	2.50
BDMZ	9/19/2023	8:48	8.79	8.30	68.1	7.98	<1.0	1.02 J	0.0657	200
BDMZ-DUP	9/19/2023	8:48	9.13	8.50	69.3	7.98	<1.0	0.863 J	0.0422	157
BDMZ-FB	9/19/2023	8:48	N/A	0.400 J	N/A	N/A	<1.0	<0.433	<0.0130	<1
BDXX	9/19/2023	9:10	9.72	8.40	66.9	8.01	<1.0	0.600 J	0.0537	221
IDBW	9/19/2023	7:41	7.18	6.05	64.9	8.18	<1.0	0.540 J	0.0938	3.05
KMXX	9/19/2023	7:13	9.06	6.20	65.6	8.12	<1.0	0.678 J	0.104	12.3
MCXX	9/19/2023	6:51	7.44	4.50	64.4	8.10	<1.0	0.576 J	0.107	1.50
BDMZ	9/20/2023	7:15	10.7	8.00	69.2	7.84	<1.0	1.68 J	0.162	52.9
BDMZ-DUP	9/20/2023	7:15	10.8	8.60	69.2	7.85	<1.0	1.80 J	0.178	55.6
BDMZ-FB	9/20/2023	7:15	N/A	0.400 J	N/A	N/A	<1.0	0.515 J	<0.0130	<1
BDXX	9/20/2023	7:35	12.3	9.60	67.0	7.88	<1.0	0.556 J	0.0669	106
IDBW	9/20/2023	6:40	6.09	2.10	64.4	7.98	<1.0	<0.433	0.0907	5.60
KMXX	9/20/2023	6:14	4.49	2.00	65.4	7.85	<1.0	<0.433	0.0915	2.00
AMOG	9/25/2023	8:05	4.76	2.10	67.2	8.11	<1.0	<0.433	0.114	4.10
AWGB	9/25/2023	11:15	3.01	2.70	69.8	8.20	<1.0	0.684 J	0.131	13.4
BDMZ	9/25/2023	8:40	7.74	11.2	70.1	8.10	<1.0	0.711 J	0.0505	42.5
BDXX	9/25/2023	8:25	8.75	6.70	69.2	8.10	<1.0	0.529 J	0.0568	83.2
IBDW-FB	9/25/2023	7:35	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0268 J	<1
IDBW	9/25/2023	7:26	3.45	3.40	64.5	7.72	<1.0	<0.433	0.136	3.60
IDBW-DUP	9/25/2023	7:30	3.47	2.94	64.6	7.72	<1.0	0.469 J	0.133	2.55
KMXX	9/25/2023	7:00	2.87	3.80	64.0	7.62	<1.0	<0.433	0.145	1.50
MCXX	9/25/2023	6:35	3.64	5.39	63.1	7.50	<1.0	0.487 J	0.145	2.00
BDMZ	9/27/2023	7:49	10.8	9.10	66.7	7.80	<1.0	1.85 J	0.714	88.7
BDXX	9/27/2023	7:55	12.4	6.90	57.4	7.90	<1.0	0.645 J	0.0774	249
IDBW	9/27/2023	6:45	4.75	4.30	59.7	7.32	<1.0	<0.433	0.122	15.2
IDBW-DUP	9/27/2023	6:45	4.76	4.90	59.7	7.34	<1.0	0.434 J	0.129	20.6
IDBW-FB	9/27/2023	6:50	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0225 J	<1
KMXX	9/27/2023	6:15	4.44	4.60	57.2	7.31	<1.0	0.475 J	0.128	2.00
AMOG	10/18/2023	9:00	8.53	4.40	54.6	7.70	<1.0	0.507 J	0.115	15.2
AWGB	10/18/2023	8:20	11.8	5.50	54.6	7.31	<1.0	0.742 J	0.136	2.00
BDMZ	10/18/2023	9:28	27.1	13.4	57.0	7.62	<1.0	1.45 J	0.174	138
BDXX	10/18/2023	9:15	28.3	13.3	56.4	7.50	<1.0	1.39 J	0.0556	338
IDBW	10/18/2023	10:00	9.76	3.90	56.6	7.63	<1.0	0.667 J	0.147	6.20
KMXX	10/18/2023	10:50	14.0	8.30	56.6	7.92	<1.0	0.717 J	0.148	2.00
KMXX-DUP	10/18/2023	10:33	13.8	6.36	56.6	7.91	<1.0	0.763 J	0.138	2.50
KMXX-FB	10/18/2023	10:33	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0191 J	<1
MCXX	10/18/2023	11:00	9.77	5.40	57.9	7.87	<1.0	0.634 J	0.148	2.00

See last page for abbreviations and notes.

Table 1. 2023 Analytical Results

Sampling Location	Sampling Date	Sampling Time	Turbidity (NTU)	TSS (mg/L)	Temperature (°F)	pH (s.u.)	Cyanobacteria (ug/L)	Chromium (ug/L)	Hex. Chromium (ug/L)	*E. coli* (MPN/100 mL)
AMOG	11/14/2023	11:06	5.41	1.80 J	53.6	7.10	<1.0	0.587 J	0.153	<1
AWGL	11/14/2023	9:44	3.34	2.10	55.0	7.15	<1.0	<0.433	0.161	<1
BDMZ	11/14/2023	10:41	9.91	7.60	55.7	7.30	<1.0	0.732 J	0.0617	50.4
BDXX	11/14/2023	10:25	9.24	7.00	56.8	7.13	<1.0	0.531 J	0.0558	72.4
IDBW	11/14/2023	11:36	4.36	4.40	55.2	7.20	<1.0	2.890 J	0.132	<1
KMXX	11/14/2023	12:15	12.1	11.1	51.8	7.20	<1.0	0.548 J	0.160	3.00
MCXX	11/14/2023	12:52	10.9	5.40	55.2	7.30	<1.0	0.527 J	0.160	<1
MCXX-DUP	11/14/2023	12:57	10.9	6.80	55.1	7.30	<1.0	0.588 J	0.153	<1
MCXX-FB	11/14/2023	13:00	N/A	<0.300	N/A	N/A	<1.0	<0.433	0.0185 J	<1
AMOG	12/7/2023	8:02	3.61	1.80 J	38.6	6.82	<1.0	1.11 J	0.128	<1
AWGL	12/7/2023	9:40	4.94	1.60 J	41.3	7.63	<1.0	0.445 J	0.134	<1
AWGL-DUP	12/7/2023	9:41	5.07	1.50 J	41.4	7.68	<1.0	0.445 J	0.135	<1
AWGL-FB	12/7/2023	9:43	N/A	0.300 J	N/A	N/A	<1.0	<0.433	0.0143 J	<1
BDMZ	12/7/2023	7:40	14.7	13.0	43.7	7.23	<1.0	1.07 J	0.0475	128
BDXX	12/7/2023	7:28	16.4	11.4	42.6	6.96	<1.0	0.519 J	0.0419	134
IDBW	12/7/2023	8:37	5.10	3.23	39.2	6.84	<1.0	0.590 J	0.142	2.00
KMXX	12/7/2023	9:07	11.7	6.40	40.1	6.78	<1.0	0.592 J	0.129	<1
MCXX	12/7/2023	9:41	14.0	6.20	41.5	6.92	<1.0	0.618 J	0.144	1.00

Abbreviations:
TSS = total suspended solids
Hex. Chromium = hexavalent chromium
N/A = not applicable
<#.# = not detected at the limit of detection shown
J = the analyte was detected, but below the limit of quantitation
DUP = field duplicate
FB = field blank

Locations:
AWGL = American Water Intake - Gary Lakeside Pump House
AWGB = American Water Intake - Breakwall
AMOG = American Water Intake - Ogden
BDXX = Burns Ditch
BDMZ = Burns Ditch / Lake Michigan Mixing Zone
IDBW = Indiana Dunes Beach - Western Area
KMXX = Kemil Beach
MCXX = Michigan City

Units:
NTU = nephelometric turbidity unit
mg/L = milligrams per liter
°C = degrees Celcius
s.u. = standard units
µg/L = micrograms per liter
MPN = Most Probable Number per 100 milliliters

APPENDIX 2
SUMMARY OF EXPENDITURES AND EVIDENCE OF COMPLETION

Ramboll, PO No. 21222788							
Invoice	**Invoice Date**	**Amount**	**Due**	**Status**	**Payment Status**	**Due Date**	**Payment**
1940061059	11-Oct-24	$1,390.29	1,390.29	Approved	Not Paid	5-Feb-25	
1940058017	5-Sep-24	$1,052.69	1,052.69	Approved	Not Paid	6-Jan-25	
1940055482	6-Aug-24	$1,867.97	1,867.97	Approved	Not Paid	5-Dec-24	
1940052877	8-Jul-24	$1,418.39	0	Approved	Paid	5-Nov-24	1000256893
1940050456	7-Jun-24	$2,119.27	0	Approved	Paid	7-Oct-24	1000253494
1940045417	8-Apr-24	$1,995.37	0	Approved	Paid	5-Aug-24	1000246088
1940040800	12-Feb-24	$1,496.52	0	Approved	Paid	5-Jun-24	1000238958
1940038397	5-Jan-24	$1,421.88	0	Approved	Paid	6-May-24	1000235330
1940031395	16-Oct-23	$1,506.20	0	Approved	Paid	5-Feb-24	1000224500
1940029277	19-Sep-23	$1,761.15	0	Approved	Paid	5-Jan-24	1000221161
1690118849	3-Aug-23	$1,653.41	0	Approved	Paid	6-Nov-23	1000214069
1690117099	10-Jul-23	$1,874.17	0	Approved	Paid	6-Nov-23	1000214069
1690115460	13-Jun-23	$1,512.04	0	Approved	Paid	5-Oct-23	1000210313
1690113547	4-May-23	$934.74	0	Approved	Paid	7-Aug-23	1000203388
1690111890	10-Apr-23	$1,344.88	0	Approved	Paid	7-Aug-23	1000203388
1690107154	9-Jan-23	$609.50	0	Approved	Paid	5-May-23	1000192748
1690105206	8-Dec-22	$1,077.49	0	Approved	Paid	5-Apr-23	1000189067
1690103729	11-Nov-22	$333.99	0	Approved	Paid	6-Mar-23	1000185566
1690101984	13-Oct-22	$3,459.96	0	Approved	Paid	6-Feb-23	1000182342
1690100332	13-Sep-22	$1,598.48	0	Approved	Paid	5-Jan-23	1000178805
1690098407	4-Aug-22	$1,137.62	0	Approved	Paid	7-Nov-22	1000172141
1690096971	11-Jul-22	$1,604.27	0	Approved	Paid	7-Nov-22	1000172141
1690095301	9-Jun-22	$3,201.57	0	Approved	Paid	5-Oct-22	1000168468
1690093560	4-May-22	$927.50	0	Approved	Paid	5-Aug-22	1000161854
1690091920	7-Apr-22	$7,024.89	0	Approved	Paid	5-Aug-22	1000161854
1690090264	10-Mar-22	$4,712.48	0	Approved	Paid	5-Jul-22	1000158361
1690089616	28-Feb-22	$306.08	0	Approved	Paid	6-Jun-22	1000155184
1690087714	24-Jan-22	$752.60	0	Approved	Paid	5-May-22	1000151662
1690086306	23-Dec-21	$11,749.59	0	Approved	Paid	5-Apr-22	1000148354
1690084024	18-Nov-21	$36,072.75	0	Approved	Paid	7-Mar-22	1000145129

$97,917.74

ALS Group USA Corp., PO No. 20256969-469							
Invoice	**Invoice Date**	**Amount**	**Due**	**Status**	**Payment Status**	**Due Date**	**Payment**
4120-99406547	12-Nov-24	3,822.75	3,822.75	Approved	Not Paid	5-Feb-25	
4120-99406216	29-Oct-24	7,099.50	7,099.50	Approved	Not Paid	6-Jan-25	
4120-99405576	30-Sep-24	23,959.00	23,959.00	Approved	Not Paid	5-Dec-24	
4120-99404767	30-Aug-24	28,064.95	0	Approved	Paid	5-Nov-24	1341064757
4120-99403889	31-Jul-24	30,003.27	0	Approved	Paid	7-Oct-24	1341062616
4120-99402723	26-Jun-24	23,959.00	0	Approved	Paid	5-Sep-24	1341060235
4120-99401985	31-May-24	23,876.88	0	Approved	Paid	5-Aug-24	1341057964
4120-99398712	30-Apr-24	3,712.75	0	Approved	Paid	5-Jul-24	1341055704
4120-99397238	29-Mar-24	3,712.75	0	Approved	Paid	5-Jun-24	1341053582
4120-99395976	29-Feb-24	3,712.75	0	Approved	Paid	6-May-24	1341051383
4120-99394661	31-Jan-24	3,712.75	0	Approved	Paid	5-Apr-24	1341049155
4120-99393247	29-Dec-23	2,463.57	0	Approved	Paid	5-Mar-24	1341046845
4120-99391544	30-Nov-23	3,712.75	0	Approved	Paid	5-Feb-24	1341044620
4120-99389702	31-Oct-23	9,620.00	0	Approved	Paid	5-Jan-24	1341042393
4120-99387584	28-Sep-23	23,959.00	0	Approved	Paid	5-Dec-23	1341040136
4120-99384959	31-Aug-23	23,959.00	0	Approved	Paid	6-Nov-23	1341037998
4120-99382883	31-Jul-23	23,959.00	0	Approved	Paid	5-Oct-23	1341035515
4120-99380958	29-Jun-23	29,866.25	0	Approved	Paid	5-Sep-23	1341033151
4120-99379167	31-May-23	23,959.00	0	Approved	Paid	7-Aug-23	1341030909
4120-99377113	28-Apr-23	3,492.75	0	Approved	Paid	5-Jul-23	1341028380
4120-99373871	28-Feb-23	3,492.75	0	Approved	Paid	5-May-23	1341023692
4120-99372439	31-Jan-23	3,492.75	0	Approved	Paid	5-Apr-23	1341021111
4120-99370857	30-Dec-22	3,492.75	0	Approved	Paid	6-Mar-23	1341018549
4120-99369017	30-Nov-22	3,492.75	0	Approved	Paid	6-Feb-23	1341016116
4120-99367135	31-Oct-22	9,400.00	0	Approved	Paid	5-Jan-23	1341013527
4120-99365059	30-Sep-22	23,629.00	0	Approved	Paid	5-Dec-22	1341011035
4120-99363005	31-Aug-22	29,454.13	0	Approved	Paid	7-Nov-22	1341008708
4120-99360802	29-Jul-22	19,438.23	0	Approved	Paid	5-Oct-22	1341005906
4120-99358969	30-Jun-22	24,917.37	0	Approved	Paid	6-Sep-22	1341003502
4120-99357192	31-May-22	21,187.80	0	Approved	Paid	5-Aug-22	1341001096
4120-99354577	29-Apr-22	3,099.57	0	Approved	Paid	5-Jul-22	620375898
4120-99352743	31-Mar-22	5,644.22	0	Approved	Paid	6-Jun-22	620373455
4120-99348566	30-Dec-21	3,369.57	0	Approved	Paid	7-Mar-22	620365658
4120-99346848	30-Nov-21	2,158.57	0	Approved	Paid	7-Feb-22	620363255

$456,897.13

APPENDIX 3
SAMPLING AND ANALYSIS PLAN AND QUALITY ASSURANCE PROJECT PLAN

Intended for
U. S. Steel Midwest Plant

Document type
Sampling and Analysis Plan

Date
November 2021

STATE-ONLY ENVIRONMENTALLY BENEFICIAL PROJECT

LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING

RAMBOLL Bright ideas. Sustainable change.

STATE-ONLY ENVIRONMENTALLY BENEFICIAL PROJECT
LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING

Project name	**USS Midwest EBP**
Project no.	**1690023671**
Recipient	**U. S. Steel Midwest Plant**
Document type	**Sampling and Analysis Plan**
Version	**1**
Date	**November 10, 2021**
Prepared by	**Carrie Y. Cunnane**
Checked by	**Jackie Backus**
Approved by	**Robin Richards**

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1. INTRODUCTION

On August 30, 2021, the U.S. District Court for the Northern District of Indiana entered the revised Consent Decree[1] between the United States of America, on behalf of the United States Environmental Protection Agency (EPA), the National Park Service of the United States Department of the Interior and the National Oceanic and Atmospheric Administration of the United States Department of Commerce; and the State of Indiana, on behalf of the Indiana Department of Environmental Management (IDEM) and the Indiana Department of Natural Resources and United States Steel Corporation (U. S. Steel) for violations at its Midwest Plant Facility in Portage, Indiana (Midwest Plant).

The revised Consent Decree included a state-only environmentally beneficial project (EBP) that requires water quality testing and reporting from seven Indiana shore locations along Lake Michigan. The goal of the state EBP is to "contribute to significant public health benefits to the communities near the plant and to those who use the popular Indiana Dunes National Park for recreation" (USEPA, 2019[2]). Specifically, the objective of generating the Lake Michigan water quality data is to determine, at the locations sampled, whether the water quality is safe for recreational use.

This sampling and analysis plan (SAP) is designed to

- present U. S. Steel project contacts and those of its consultants,
- establish a communication plan,
- confirm distribution list of parties and contact information,
- assure consistent execution of sampling including identification of sample locations and establishment of sampling schedule,
- address health and safety during sampling,
- confirm laboratory methods, laboratory logistics for samples with short hold times, required detection limits, and electronic data transmittal format,
- identify specific in-situ methods and associated calibration requirements,
- document quality assurance/quality control (QA/QC) procedures to be followed,
- outline the data verification process and corrective actions, and
- present field note templates and data reporting templates.

This document will be updated yearly to document changes to the sampling program. These are anticipated to include site access, distribution list of parties, communications and contacts, or data quality updates.

2. PROJECT TEAM AND COMMUNICATIONS PLAN

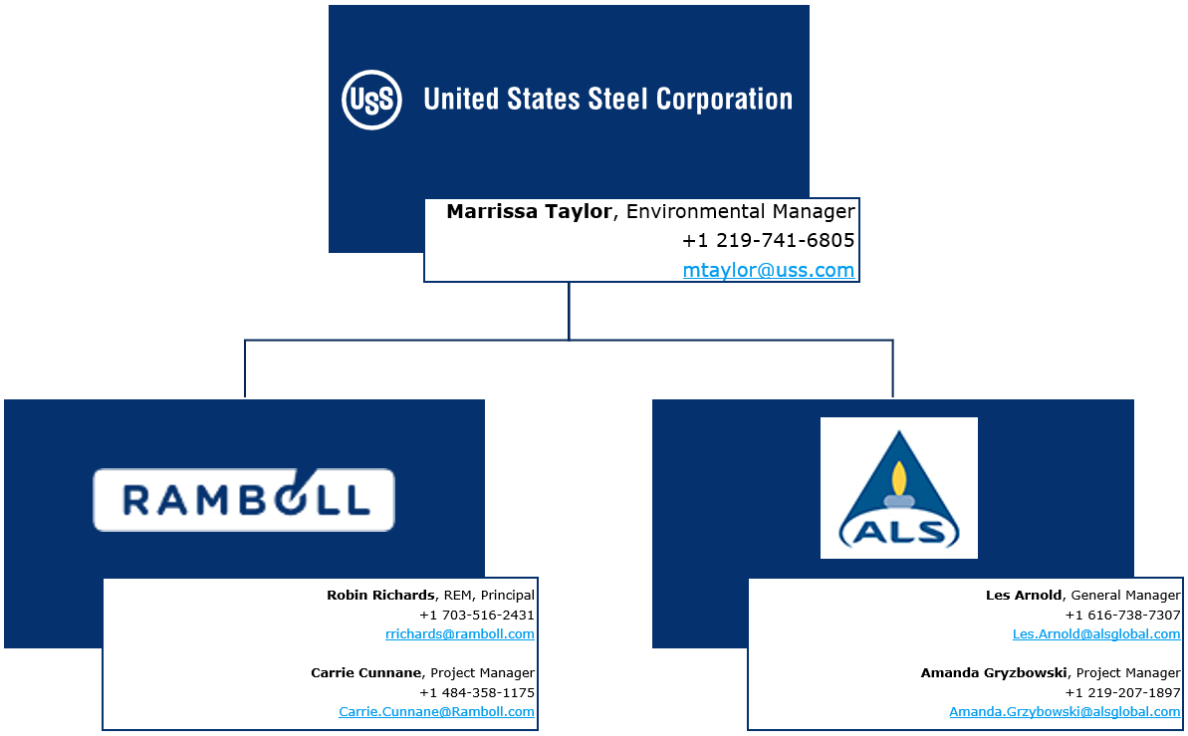
U. S. Steel has hired Ramboll and ALS to implement the EBP. U. S. Steel holds ultimate responsibility for the program and will be the main point of contact for USEPA and IDEM. Ramboll has prepared this Sampling and Analysis Plan and will provide QA/QC and project oversight. ALS

[1] USDC IN/ND case 2:18-cv-00127-TLS-JEM document 46-1 filed 11/20/19

[2] United States EPA (United States Environmental Protection Agency). 2019. U. S. Steel Corporation Consent Decree. Available on-line at: https://www.epa.gov/in/u-s-steel-corporation-consent-decree. Site last updated on October 20, 2020; site last accessed September 22, 2021.

personnel will collect field samples, perform laboratory analyses, validate data, and upload data to agency websites. Names and contact information for the project team are provided in the organization chart below (Figure 1).

Figure 1. Project Team Organization



To access the Town of Dune Acres, Burns Ditch, and American Water Intake at Ogden sampling locations, an email will be sent at least one day prior to sampling to the following recipients:

1. Jan Bapst, Town of Dune Acres (datownclerk@gmail.com)
2. Richard Hawksworth, Town of Dune Acres (richhawksworth@mac.com)
3. Pete Harretos, Indiana American Water (Pete.Harretos@amwater.com)
4. AJ Monroe, Portage Redevelopment Commission (ajmonroe@portage-in.com)

On the day of sampling, call Pete Harretos (219-384-9509) or Ryan Love (219-240-6239) at Indiana American Water to unchain the gate at Ogden.

Reports and data required to be submitted to IDEM and USEPA (see Section 6) will be sent to the following addresses.

USEPA by email:

R5weca@epa.gov
Re: U. S. Steel Midwest Plant

USEPA by mail:

Chief, Water Enforcement and Compliance Assurance Branch (WC-15J)
U.S. Environmental Protection Agency, Region 5
77 West Jackson Blvd.
Chicago, IL 60604
and
Office of Regional Counsel (C-14J)
U.S. Environmental Protection Agency
77 West Jackson Blvd.
Chicago, IL 60604

IDEM by email (upon receipt of notice from IDEM, per Paragraph 84 of the Consent Decree, that designated notice recipients have been changed):

BAdmire@idem.in.gov
AFarren@idem.in.gov
NReam@idem.in.gov

IDEM by mail:

Chief, Environmental Section
Office of the Attorney General
Indiana Government Center South, 5th Floor
402 West Washington Street
Indianapolis, IN 46204
and
Chief, Compliance Branch
Indiana Department of Environmental Management
Office of Water Quality, Mail Code 65-40
100 North Senate Avenue
Indianapolis, IN 46204-2251
and
General Counsel
Office of Legal Counsel
Mail Code 60-01
100 North Senate Street
Indianapolis, IN 46204-2251

3. FIELD SAMPLING

3.1 Locations

Water quality measurements and analytical laboratory samples will be collected from seven locations on along southern Lake Michigan in Indiana. The general locations were identified in the Consent Decree as:

a) Burns Ditch
b) Burns Ditch / Lake Michigan Mixing Zone
c) Kemil Beach
d) Indiana Dunes Beach – Western Area
e) Michigan City
f) Vicinity of American Water Intake – Gary
g) Vicinity of American Water Intake – Ogden

Site reconnaissance on September 14, 2021 identified more precise locations that could be safely accessed year-round. Table 1 lists the latitude-longitude locations of these proposed sampling areas and the Field QAPP provides aerial photographs. Exact sampling locations may change during the program due to weather, safety, or access concerns. Changes will be documented in the field notes and a GPS point will be recorded at the actual sampling location.

Table 1. Latitude and Longitude of Proposed Sampling Locations

Sampling Location (Abbreviation)	Latitude[1]	Longitude[1]
a. Burns Ditch (BDXX)	41°37'18.70"N	87°10'35.74"W
b. Burns Ditch / Lake Michigan Mixing Zone (BDMZ)	41°37'53.60"N	87°10'37.51"W
c. Kemil Beach (KMXX)	41°40'54.02"N	87°0'36.62"W
d. Indiana Dune Beach – Western Area (IDBW)	41°39'26.01"N	87°4'49.43"W
e. Michigan City (MCXX)	41°42'12.56"N	86°57'2.37"W
f. Vicinity of American Water Intake – Gary[2]		
Lakeside Pump House (Winter Sampling) (AWGL)	41°37'51.79"N	87°22'26.33"W
Breakwall (Summer Sampling) (AWGB)	41°37'29.87"N	87°19'37.87"W
g. Vicinity of American Water Intake – Ogden (AMOG)	41°37'37.87"N	87°12'9.57"W

Notes:
[1] Positions obtained using Google Earth Pro.
[2] Given that most of the coastline on the U. S. Steel Gary property is fortified with boulders, rip-rap, or concrete piles, there is very little safe access to the water. Samples are proposed to be collected from the Lakeside Pumphouse intake forebay when the intake is not being chlorinated (typically the months of October/November through April/May). To avoid interference with the analyses of *E. coli* and cyanobacteria during chlorination, samples are proposed to be collected from the west side of the slip breakwall when safe, during periods of intake chlorination (typically the months of May through October).

All surface water sample collection and water quality measurements will be collected via grab sampling from land; no boating is proposed. A sampling container attached to a pole will be dipped into the water to collect approximately one quart of surface water. Beach samples (locations c., d., e., and g.) and the U.S. Steel Gary breakwall sample (location f. during intake

chlorination timeframes) will be collected from the water's edge. Grab samples will be collected from platforms at locations a. and b. (historical outfall and fish pier, respectively) and from the intake forebay at the Lakeside Pump House (location f. when not being chlorinated). Further details of site access are provided in the Field QAPP.

3.2 Schedule

This EBP will continue for three years from the start of the first sampling event, which must occur within 90 days of issuance of the final Consent Decree. Based on August 30, 2021 as the day the Consent Decree was entered, sampling must begin by November 28, 2021.

Per the Consent Decree, all seven locations will be sampled once per month from October through April. From May 1 through September 30, locations e., f., and g. will be sampled weekly and locations a. through d. will be sampled twice weekly. The program will result in 291 samples per year for a total of 873 (Table 2).

Table 2. Proposed Number of Samples by Location and Year

Sampling Location	Number of Samples per Month (Oct – Apr)	Number of Samples per Week (May – Sep)	Number of Samples per Year	Total Number of Samples
a. BDXX	1	2	51	153
b. BDMZ	1	2	51	153
c. KMXX	1	2	51	153
d. IDBW	1	2	51	153
e. MCXX	1	1	29	87
f. AWGL / AWGB[(1)]	1	1	29	87
g. AMOG	1	1	29	87
Totals	7	11	291	873

Notes:
[(1)] The chlorination schedule depends on environmental factors and therefore the proportion of samples collected from the two locations cannot be determined at this time. Therefore, the total number of samples to be collected are shown regardless of which location in the Vicinity of American Water Intake – Gary they are collected.

A generalized calendar of the sampling program for the first year is shown in Figure 2. The proposed sampling dates included will be adjusted, as needed, based on weather, field team availability, and site accessibility. It is anticipated that weather will impact the sampling schedule, particularly during winter when ice accumulation may make sampling unsafe or impossible and during storm events when strong winds and large waves will preclude safe water work. Therefore, the following process will be used to obtain as many valid samples as possible during the three-year program.

October – April:

1. Schedule sampling during the first full week of the month to allow for delays.

2. If weather precludes sampling from all seven locations, schedule the event for the second week of the month.
3. If weather precludes sampling from all seven locations during the second week, schedule the event for the third week of the month.
4. Obtain as many samples as possible during the third week. If all seven locations cannot be obtained, schedule sampling of the remaining locations for the fourth week of the month.

Weather websites will be monitored, and on-the-ground conditions will be ascertained from plant personnel throughout the month. It is anticipated that all seven locations will be able to be sampled at some point during the month. However, if weather or other *force majeure* factors preclude sampling a location during a month in this period (October – April), the EBP will be extended by one month for that location.

May – September:

1. Schedule full sampling (seven locations) for Monday or Tuesday and supplemental sampling (four locations) for Wednesday or Thursday.
2. Field personnel will collect as many samples as safely possible on the scheduled event day. If storms, lightning, or other unsafe conditions preclude sampling from all planned locations, the remaining locations will be sampled the following day, if feasible.
3. Friday will be used as a back-up day to collect missed samples.

A reasonable attempt will be made to safely collect the 11 scheduled samples each week. The planned frequency of sampling will provide a robust data set; therefore, if 80% of the data for the week is available, it will be considered a comprehensive data set, and the EBP will not be extended.

Figure 2. General Sampling Schedule for Year 1*

* For illustrative purposes only.
Orange circles indicate sampling events where all seven locations will be visited.
Blue circles are events where only locations a. through d. will be sampled.

3.3 Health and Safety

A program-specific Health and Safety Plan will be prepared that addresses the risks associated with the field sampling and the laboratory analysis portions of the program. As described above, historical weather and marine information for southern Lake Michigan suggests that high winds, substantial waves, thunder/lightning storms, and icy conditions will be encountered during the three-year sampling program. The Health and Safety Plan will specifically address these risks:

- Work over water,
- Heat stress/cold stress,
- Thunderstorms,
- Biological hazards (poisonous plants, stinging insects, tick-borne disease, etc.),
- Slips, trips, falls (especially related to precipitation),
- Vehicle travel,
- COVID,
- Risks associated with an active industrial site (U. S. Steel Gary Works-specific precautions), and
- Chemical (e.g., preservatives, calibration solutions).

4. WATER QUALITY PARAMETERS

Surface water collected from the sampling locations identified in Section 3 will be measured or analyzed for the following parameters:

i. hexavalent chromium
ii. total chromium
iii. cyanobacteria
iv. *E. coli*
v. pH
vi. total suspended solids (TSS)
vii. temperature
viii. turbidity (as an indication of transparency)

4.1 In-situ Field Measurements

pH and temperature will be measured in the field using a Thermo Scientific Orion Star A121 pH meter with ATC electrode probe. A Thomas Scientific Traceable Temp Probe will be on location as a backup. Because transparency measured using a Secchi disk is best suited for deeper waterbodies and is not appropriate for shallow water beach settings, turbidity (as an indicator of transparency) will be measured using a Hach 2100p turbidity meter.

A grab sample of approximately 1 quart will be collected twice. For the first grab, the probe will be placed in the sampling container to measure pH and temperature. For the second grab, a portion of the sample will be transferred to a vial for turbidity measurement with a field meter (the remaining volume will be used to fill sample containers for lab analyses). Electronic readings will be recorded either in a dedicated field notebook or on field data sheets (see example provided in the Field QAPP).

4.2 Water Chemistry

Hexavalent chromium, total chromium, cyanobacteria, *E. coli*, and TSS will be analyzed in the analytical laboratory. A grab sample of surface water will be collected from each location and

transferred to laboratory-supplied bottleware. A portion of the sample will be filtered in the field for hexavalent chromium analysis. Labeled sample bottles will be placed in a cooler filled with wet ice for transport to the analytical laboratory. Given the relatively short hold-time for *E. coli* analysis, a courier will deliver water samples to the analytical laboratory mid-way through the sampling day during events when all seven locations will be sampled.

40 CFR Part 136-approved methods will be used for laboratory analysis, where applicable. ALS Valparaiso is accredited by The National Environmental Laboratories Accreditation Conference (NELAC) Institute and has certifications for hexavalent chromium by EPA 218.6, chromium by EPA 200.8, and totals suspended solids by Standard Methods 2540 D. *E. coli* analysis using SM 9223 B will be subcontracted by ALS to Utility Services.

There is no 40 CFR Part 136-approved method for cyanobacteria. ALS will perform lab analysis using the Abraxis Microcystin Test Strip 520022. Abraxis 520022 is a semi-quantitative Immunochromatographic Strip Test for the Detection of microcystins and nodularins in recreational water, which allows for the rapid screening of the presence of algal toxins down to a 1 part per billion level.

Table 3. Analytical Methods and Anticipated Detection Limits

Test Method	Analyte	Matrix	MDL[(1)]	PQL[(2)]	Units[(3)]
EPA 200.8	Chromium	Water	0.00011	0.002	mg/L
EPA 218.6	Chromium, hexavalent (dissolved)	Water	0.026	0.25	µg/L
SM 2540 D	TSS	Water	0.3	2	mg/L
SM 9223 B	*E. coli*	Water	1	1	mpn/100mL
Abraxis 520022	Cyanobacteria (microcystins/nodularins)	Water	1	1	µg/L

Notes:

[(1)] Laboratory limits subject to change as new method detection limit (MDL) studies are performed.

[(2)] PQL = practical quantitation limit

[(3)] Units: mg/L = milligrams per liter; µg/L = micrograms per liter; mpn/100 mL = most probable number per 100 milliliters

4.3 Field Blanks and Field Duplicate Samples

Field blanks for total and hexavalent chromium will be collected once per sample event. The field blank will consist of laboratory water processed in the same manner as samples (e.g., filtered for hexavalent chromium and chemical preservatives added) at one of the sample locations.

A field duplicate for all parameters (field and lab) will also be collected once per sample event (i.e., at one of the sample locations). The same sampling procedures as described above will be utilized. Duplicate temperature and pH will be taken using a third grab sample, with samples for turbidity and lab parameters coming from a fourth grab sample.

5. DATA QUALITY

The Consent Decree requires that data collected for the EBP fulfill the data quality assessment Level 3 criteria described in IDEM's (2015) *Technical Guidance for the Office of Water Quality External Data Framework*. The field and laboratory programs will follow the IDEM 2015 guidance, as described in the accompanying Field Quality Assurance Project Plan (QAPP) and Laboratory QAPP. The EBP-specific QAPPs were prepared by modifying the IDEM Nonpoint Source Program's (2011) QAPP template. Additionally, a quality audit will be performed monthly May through September and twice during the period of October through April using the *Certification Form for Submission of External Data for OWQ Tier 2 and Tier 3 Uses* presented as Appendix 1 of the IDEM (2015) Guidance.

6. REPORTING

There are five types of reporting associated with this project: public reporting, *E.coli* Beach*Alert*[3], IDEM External Data Framework, raw data, and completion. The frequency and submittal dates for each type of reporting follow the requirements outlined in the Consent Decree (Table 4). An example of the standardized reporting format for weekly/monthly public reporting is provided as Table 5. Concentrations below detection limits will be reported as "ND" with the practical quantitation limit or reporting limit provided afterwards in parenthesis.

[3] The Consent Decree requires data reporting to the "Beach Guard notification system." The web portal for this information is now called Beach*Alert*. The website in the Consent Decree (https://www.in.gov/idem/lakemichigan/pages/beachguard/) redirects the reader to https://portal.idem.in.gov/beachalert/.

Table 4. Summary of Reporting Requirements

Type	Frequency	Due Date	Data Type	Where / To Whom
Public Reporting	May – Sep: weekly Oct – Apr: monthly Annually	May – Sep: Wednesday of following week Oct – Apr: 5th business day of following month March 31 of following year	Dates and times of sampling events; Results of measurement of all parameters	IDEM(1); https://midwest.uss.com(2)
Beach*Alert*	May – Sep: twice weekly Oct – Apr: monthly	Within 8 hours of receiving results from the laboratory	*E. coli* concentrations	https://portal.idem.in.gov/beachalert/
External Data Framework	Annually	March 31 of following year	Data and data quality documentation	IDEM's Secondary Data portal
Raw Data	May – Sep: twice weekly Oct – Apr: monthly	Within 8 hours of receiving testing results from the laboratory	Raw laboratory data(3) for total chromium, hexavalent chromium, and *E. coli*	IDEM(1); USEPA(1)
Completion	Once	Within 30 days from the completion of the EBP	Project description; summary of expenditures; evidence of completion; corporate official certification	IDEM(1); USEPA(1)

Notes:

(1) Reports will be submitted electronically and in hard copy to the recipients listed in Section 2.

(2) The correct web address to reach the Midwest Plant page is https://midwest.uss.com. "www.midwest.uss.com" as stated in the Consent Decree leads to an error page.

(3) For the purposes of this requirement, a Level II ALS laboratory report with incorporation of the subcontractor *E. coli* results will be used for submission.

Table 5. Example Public Reporting Data Format*

Site ID	Site Name	Sample Number	Date Sampled	Time Sampled	Cr^{+6} (µg/L)	Cr (µg/L)	cyano (ppb)	E. coli (CFU)	pH	TSS (mg/L)	Temp (°C)	Temp (°F)	Turbidity (NTU)
BDXX	Burns Ditch	BDXX_MMDDYY	MM/DD/YYYY	HH:MM	ND (0.5)	ND (1.0)	3	18	7.8	1.2	15.7	60.3	0.55
BDMZ	Burns Ditch / Lake Michigan Mixing Zone	BDMZ_MMDDYY	MM/DD/YYYY	HH:MM									
KMXX	Kemil Beach	KMXX_MMDDYY	MM/DD/YYYY	HH:MM									
IDBW	Indiana Dune Beach – Western Area	IDBW_MMDDYY	MM/DD/YYYY	HH:MM									
MCXX	Michigan City	MCXX_MMDDYY	MM/DD/YYYY	HH:MM									
AWGL	American Water Intake – Gary; Lakeside Pump House	AWGL_MMDDYY	MM/DD/YYYY	HH:MM									
AWGB	American Water Intake – Gary; Breakwall	AWGB_MMDDYY	MM/DD/YYYY	HH:MM									
AMOG	American Water Intake – Ogden	AMOG_MMDDYY	MM/DD/YYYY	HH:MM									

ND ( ) = Not detected at the listed reporting limit.
NR = Sample was not collected because the location was not required to be sampled on that date.
NC = Sample was not collected due to severe weather, equipment failure, or other reason.

* For illustrative purposes only.

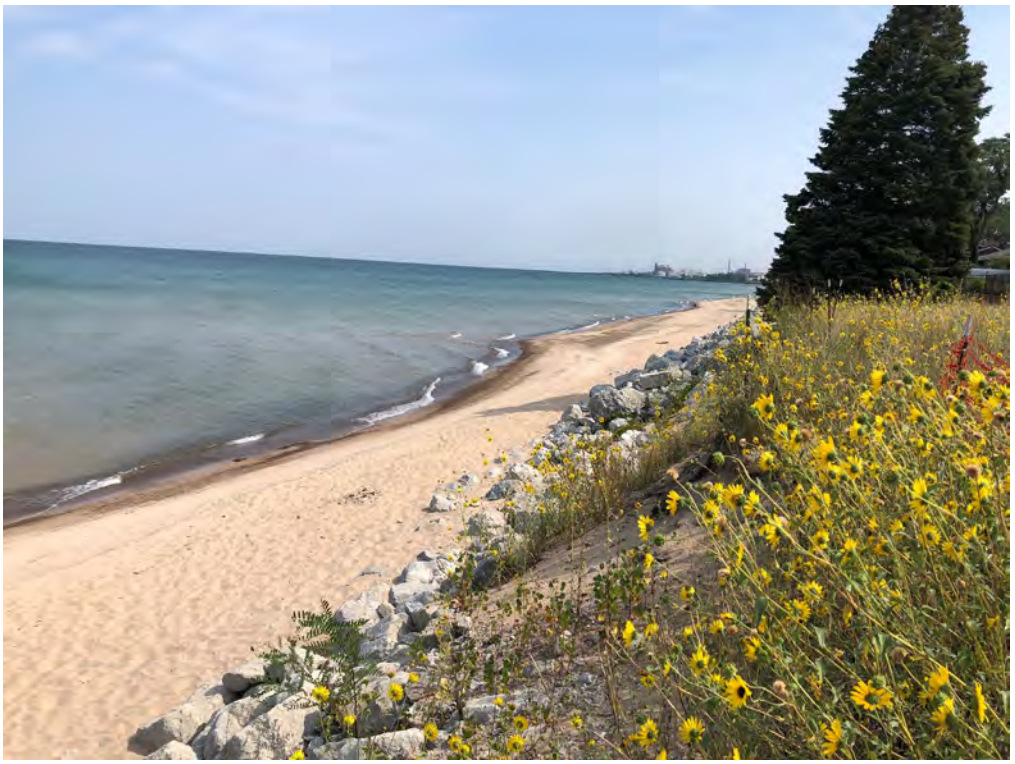
Intended for
U. S. Steel Midwest Plant

Document type
Quality Assurance Project Plan

Date
November 2021

STATE-ONLY ENVIRONMENTALLY BENEFICIAL PROJECT

LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING



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STATE-ONLY ENVIRONMENTALLY BENEFICIAL PROJECT
LAKE MICHIGAN'S INDIANA SHORELINE SAMPLING

Project name	**USS Midwest EBP**
Project no.	**1690023671**
Recipient	**U. S. Steel Midwest Plant**
Document type	**Quality Assurance Project Plan**
Version	**1**
Date	**November 10, 2021**
Prepared by	**Carrie Cunnane**
Checked by	**Jackie Backus**
Approved by	**Robin Richards**

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1. PROJECT ORGANIZATION

United States Steel Corporation (U. S. Steel) has hired Ramboll and ALS to implement the EBP. U. S. Steel holds ultimate responsibility for the program and will be the main point of contact for USEPA and IDEM. Ramboll has prepared this Sampling and Analysis Plan with input from ALS and will provide QA/QC and project oversight. ALS personnel will collect field samples, perform laboratory analyses, validate data, and upload data to agency websites.

1.1 Key Personnel

The following individuals will lead the EBP with support from additional, qualified staff in U. S. Steel, Ramboll, and ALS. Sampling events will be performed by two-person teams of field staff trained on the specific protocols and data quality measures needed for this EBP. It is anticipated that a courier will be needed during seven-location field events to transport samples to the analytical laboratory within the required hold time.

U. S. Steel

Project Manager: Marrissa Taylor, Environmental Manager
mtaylor@uss.com
219-741-6805
Ms. Taylor will be responsible for implementing the EBP and will be the direct contact with IDEM and USEPA.

Ramboll

Client Manager: Robin L. Richards, REM, Principal in Charge
rrichards@ramboll.com
703-516-2431
Ms. Richards will act as the client contact on behalf of Ramboll with U. S. Steel and will be responsible for project team-agency interactions.

Project Manager: Carrie Y. Cunnane, Permitting Specialist
Carrie.Cunnane@Ramboll.com
484-358-1175
Ms. Cunnane will manage Ramboll's portion of the project, including field work staffing, reporting, and coordination with ALS.

QA Manager: Jackie M. Backus, Project Quality Manager
jbackus@ramboll.com
703-516-2432
Ms. Backus will be responsible for in situ data quality and coordination with ALS for the laboratory QA/QC program.

Field Team Lead: Samuel Mallow, Consultant
smallow@ramboll.com
312-288-3853
As Field Team Lead, Mr. Mallow will oversee field sampling events on a regular basis for adherence to the sampling plan and quality objectives (likely once a month during May through September and twice during the period of October through April).

ALS

Client Manager: Les Arnold, General Manager
Les.Arnold@alsglobal.com
616-738-7307
Mr. Arnold will ensure that ALS has proper resources to complete the assigned field, laboratory, and reporting efforts for the project.

Project Manager: Amanda Grzybowski, Project Manager
Amanda.Grzybowski@alsglobal.com
219-207-1897
Ms. Grzybowski will oversee sample information entry into the ALS Laboratory Management Information System. She will be responsible for report generation and data management.

QA Manager: Chad Stoike
Chad.Stoike@alsglobal.com
616-399-6070
Mr. Stoike is responsible for laboratory Quality Systems and performance.

Field Team Lead: Brandon Frye, Environmental Project Manager.
Brandon.Frye@alsglobal.com
219-207-1896
Mr. Frye will coordinate field sampling events, obtain sampling and analysis equipment, and perform regularly attend sampling events.

1.2 Project Organization Chart

2. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

2.1 Goal Statements and Objective Statements

U. S. Steel is performing a three-year, state-only environmentally beneficial project (EBP) to monitor water quality at seven Indiana shore locations along Lake Michigan. The goal of the state-only EBP is to "contribute to significant public health benefits to the communities near the plant and to those who use the popular Indiana Dunes National Park for recreation" (USEPA, 2019[1]). Specifically, the objective of generating the Lake Michigan water quality data is to determine, at the locations sampled, whether the water quality is safe for recreational use.

U. S. Steel's contractors will collect surface water samples and measure water quality parameters in situ at recreational beach locations, along the U. S. Steel Gary Works property, and along Burns Ditch near the U. S. Steel Midwest Plant. Monthly (during the winter) and weekly or twice weekly sampling (depending on sampling location) will record pH, temperature, and turbidity (as a measure of transparency) and analyze the concentration of hexavalent chromium, total chromium, *E. coli*, cyanobacteria, and total suspended solids (TSS) in the water. Sampling details are provided in the sections below.

2.2 Study Site

Water quality measurements and analytical laboratory samples will be collected from seven locations on along southern Lake Michigan in Indiana (Figure 1 and Figure 2). The general locations were identified in the Consent Decree as:

a) Burns Ditch
b) Burns Ditch / Lake Michigan Mixing Zone
c) Kemil Beach
d) Indiana Dunes Beach – Western Area
e) Michigan City
f) Vicinity of American Water Intake – Gary
g) Vicinity of American Water Intake – Ogden

[1] United States EPA (United States Environmental Protection Agency). 2019. U. S. Steel Corporation Consent Decree. Available on-line at: https://www.epa.gov/in/u-s-steel-corporation-consent-decree. Site last updated on October 20, 2020; site last accessed September 22, 2021.

Figure 1. Burns Ditch Sampling Locations

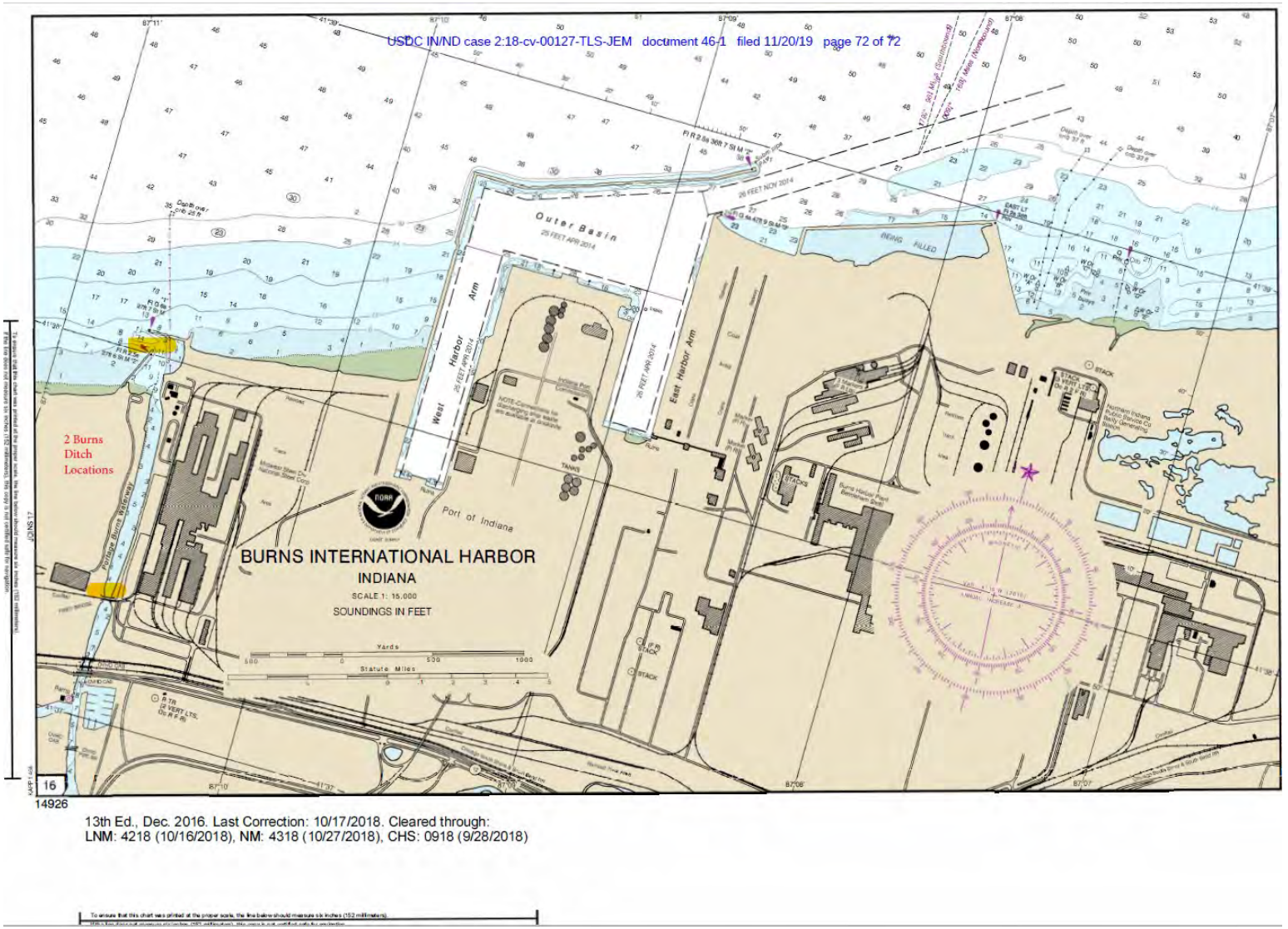
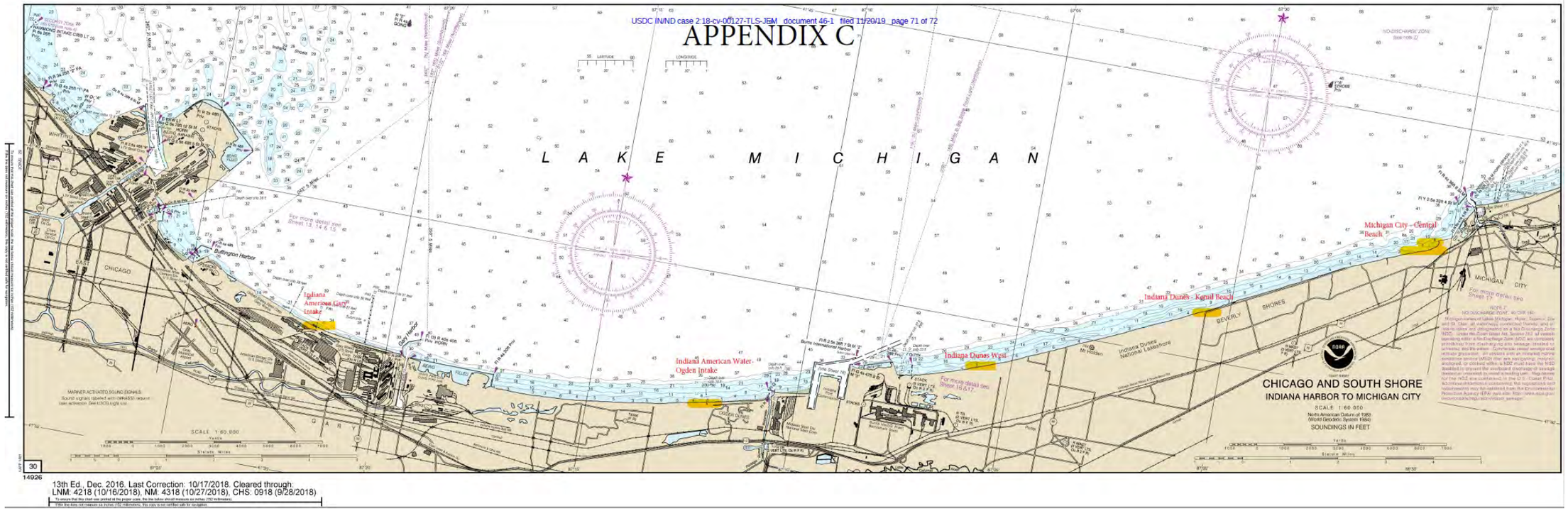


Figure 2. Lake Michigan Sampling Locations



Site reconnaissance on September 14, 2021 identified more precise locations that could be safely accessed year-round. Table 1 lists the latitude-longitude locations of these proposed sampling areas. Exact sampling locations may change during the program due to weather, safety, or access concerns. Changes will be documented in the field notes and a GPS point will be recorded at the actual sampling location.

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Sampling Location (Abbreviation)	Latitude[1]	Longitude[1]
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d. Indiana Dunes Beach – Western Area (IDBW)	41°39'26.01"N	87°4'49.43"W
e. Michigan City (MCXX)	41°42'12.56"N	86°57'2.37"W
f. Vicinity of American Water Intake – Gary[2]		
Lakeside Pump House (Winter Sampling) (AWGL)	41°37'51.79"N	87°22'26.33"W
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[1] Positions obtained using Google Earth Pro.
[2] Given that most of the coastline on the U. S. Steel Gary property is fortified with boulders, rip-rap, or concrete piles, there is very little safe access to the water. Samples are proposed to be collected from the Lakeside Pumphouse intake forebay when the intake is not being chlorinated (typically during the months of October/November through April/May). To avoid interference with the analyses of *E. coli* and cyanobacteria during chlorination, samples are proposed to be collected from the west side of the slip breakwall when safe, during periods of intake chlorination (typically the months of May through October).

All surface water sample collection and water quality measurements will be collected via grab sampling from land; no boating is proposed. A sampling container attached to a rope or pole will be dipped into the water to collect approximately one quart of surface water. Beach samples (locations c., d., e., and g.) and the U.S. Steel Gary breakwall sample (location f. during chlorination) will be collected from the water's edge. Grab samples will be collected from platforms at locations a. and b. (historical outfall and fish pier, respectively) and from the intake forebay at the Lakeside Pump House (location f. when not being chlorinated).

2.2.1 Burns Ditch (BDXX)

The Burns Ditch sampling location is located on the western bank of Burns Ditch (also called the East Arm Little Calumet River), on the south side of Train Cent Way (Figure 3). Samples will be collected from the metal platform previously used to access an outfall no longer in operation.

Figure 3. BDXX Sampling Location - Aerial View

Figure 4. BDXX Sampling Location – View from Riverwalk Drive

Figure 5. BDXX Sampling Location – Stairs to Sampling Platform

2.2.2 Burns Ditch / Lake Michigan Mixing Zone (BDMZ)

The Burns Ditch / Lake Michigan Mixing Zone sampling location is the Portage Lakefront and Riverwalk fishing pier. Samples will be collected from the northern end of the pier, which is approximately 400 feet away from the U. S. Steel Midwest Outfall 004, and therefore not affected by the outfall's effluent.

Figure 6. BDMZ Sampling Location – Aerial View

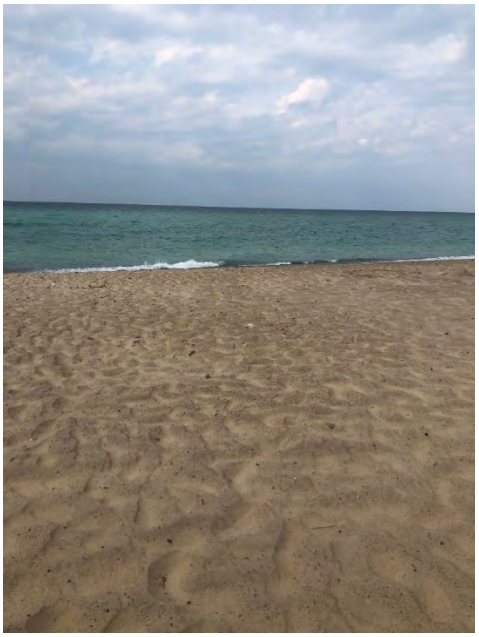
Figure 7. BDMZ Sampling Location – Fishing Pier Looking North

2.2.3 Kemil Beach (KMXX)

The Kemil Beach sampling location is in Beverly Shores, Pine Township, Porter County, Indiana. Public parking is available at the Kemil Beach and Dune Ridge Trail Parking Lot and the beach is publicly accessible from West Lake Front Dr.

Figure 8. KMXX Sampling Location – Aerial View

Figure 9. KMXX Sampling Location – Kemil Beach Under Calm Conditions



2.2.4 Indiana Dunes Beach – Western Area (IDWB)

The Indiana Dunes Beach West area is located in the Town of Dune Acres, a small, private community with 24-hour security that monitors traffic in and out of the town. The shoreline is narrow and steep, limiting access for sampling. The IDBW sampling location will be accessed from the eastern end of Beach Drive where parking and a gradual slope will allow entrance to the Town of Dune Acres beaches.

Figure 10. IDBW Sampling Location – Aerial View

2.2.5 Michigan City (MCXX)

The Michigan City Central Beach sampling location will be accessed via Central Ave., where public parking and beach access are available. Although most of the shoreline in this area is steep cliff, apparent beach replenishment allows access to the water.

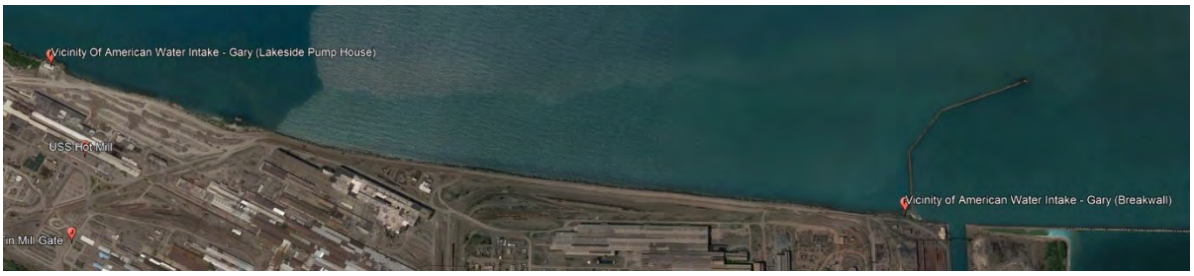
Figure 11. MCXX Sampling Location – Aerial View

Figure 12. MCXX Sampling Location – Beach Access through the Dunes

2.2.6 Vicinity of American Water Intake – Gary

The entire shoreline of the U. S. Steel Gary Works property near the Indiana American Water intake is highly modified, fortified with rip-rap, boulders, and concrete. With few safe locations to collect water samples, the safest and most accessible sampling location is from the Lakeside Pump House intake forebay. The sampled water would be from the offshore, submerged intake, similar to the American Water Intake. However, U. S. Steel Gary Works chlorinates the intake during summer to reduce biofouling, and chlorination would interfere with *E. coli* analyses. Therefore, surface water sampling and measurement will be performed on the western side of the Gary Harbor breakwall when the Lakeside Pump House intake is being chlorinated and conditions are deemed safe for access.

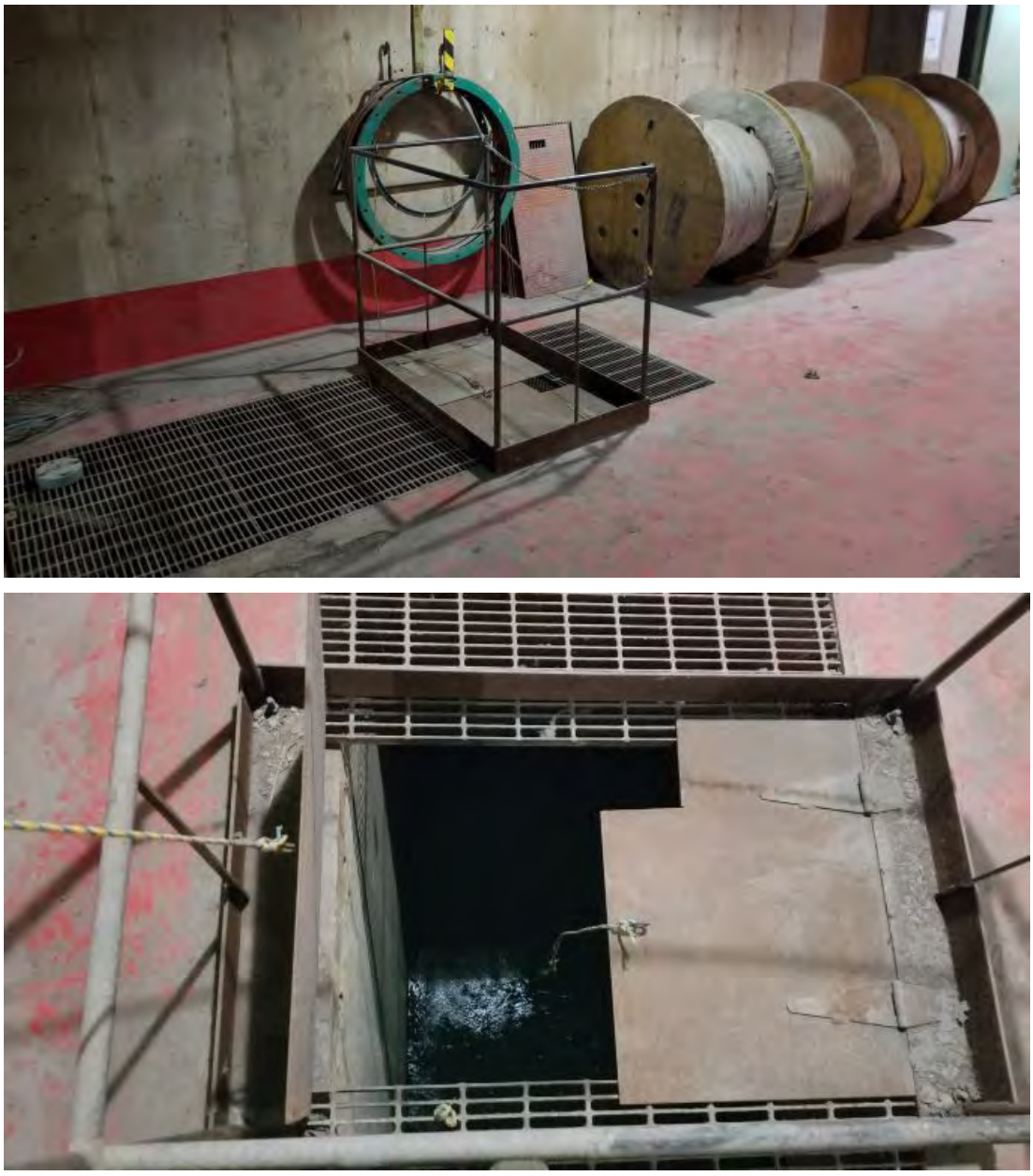
Figure 13. Vicinity of American Water Intake – Gary Sampling Location – Aerial View



2.2.6.1 Lakeside Pump House (AWGL)

The Lakeside Pump House's forebay provides a safe location to collect water samples, protected from severe weather.

Figure 14. AWGL Sampling Location – Intake Forebay



2.2.6.2 Breakwall (AWGB)

A staircase provides access to Gary Harbor breakwall and a small area of boulders and rip-rap is available for sampling to the west of the breakwall. Sampling on the eastern side of the breakwall would not be appropriate given the Gary Works outfall discharging in the vicinity.

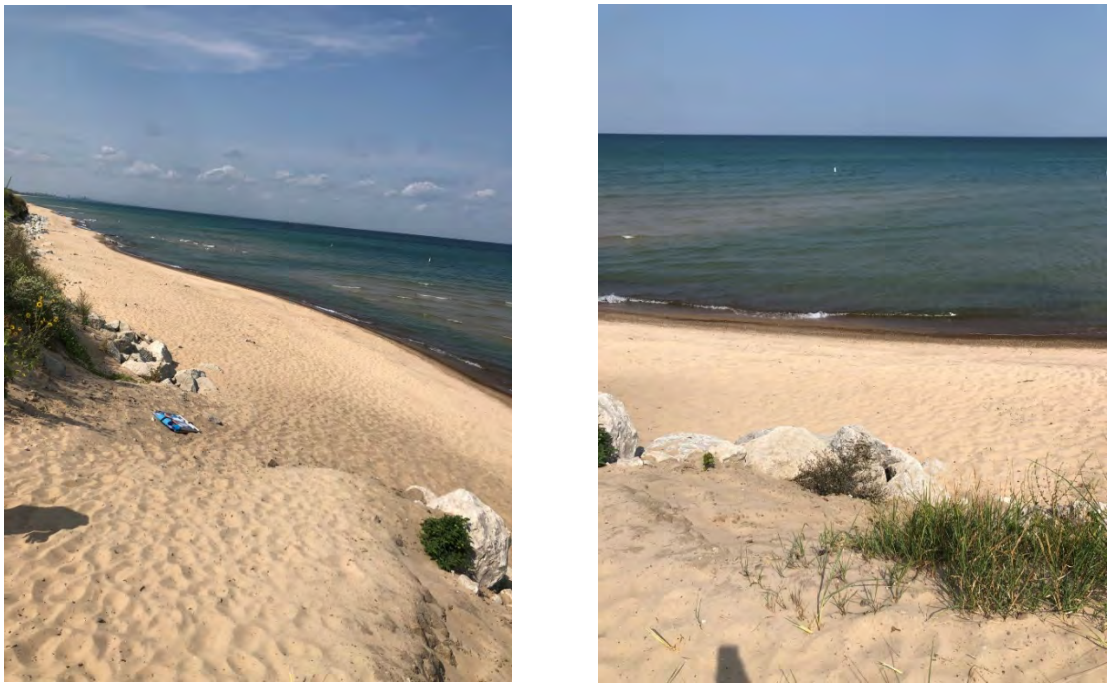
Figure 15. AWGB Sampling Location – Breakwall and Lake Access in Calm Conditions

2.2.7 Vicinity of American Water Intake – Ogden (AMOG)

The American Water Intake – Ogden sampling site is accessible from the Indiana American Water facility at the end of Shore Drive in Portage, IN or from Ogden Dunes Beach Accessway #18.

Figure 16. AMOG Sampling Location – Aerial View

Figure 17. AMOG Sampling Location – Beach Access through the Dunes



Photos taken from Ogden Dunes Beach Accessway #18. Sampling will occur approximately 1,000 feet west of this position (shown in the farfield in the left photo).

2.3 Sampling Design

The sampling approach for the EBP was defined in the Consent Decree, including the water quality parameters to be monitored and the frequency of sampling. Hexavalent chromium, total chromium, cyanobacteria, *E. coli*, and TSS concentrations in surface water will be analyzed in a laboratory; pH, temperature, and turbidity (as a measure of transparency) will be measured in the field. Field measurements may be performed by collecting a grab sample of approximately one quart followed by either transfer to a meter vial (for turbidity) or placing the probes into the sampling container (for pH and temperature). Analytical samples will be collected via grab sample and transferred to laboratory-supplied bottleware.

All eight parameters will be evaluated during each sampling event and at all accessible sampling locations. Additional analytical samples will be collected for QA/QC as outlined in the IDEM (2015) *Technical Guidance for the office of Water Quality External Data Framework* for lake samples. One field duplicate and one field blank will be collected during each sampling event given that there are less than 10 samples per event.

2.4 Study Timetable

Table 2. EBP Timetable

Activity	Start Date	End Date
Sample Collection(1): - October – April: monthly - May – September: twice weekly (locations a. through d.) - May – September: weekly (locations e. through g.)	November 2021	October 2024
Laboratory Analysis (ongoing)	December 2021	November 2024
Public Reporting and Raw Data Reporting: - October – April: monthly - May – September: weekly	December 2021	November 2024
Public Reporting (annual) and External Data Framework Reporting	March 2022	March 2025
Beach*Alert*[2] Reporting: - October – April: monthly - May – September: twice weekly	November 2021	October 2024
Completion Reporting	April 2025(2)	April 2025(2)

Notes:

(1) Cr^{6+}, Cr, cyanobacteria, *E. coli*, pH, TSS, temperature, turbidity

[b] Based on "completion" of the EBP by submitting the final annual reports in March 2025. The Consent Decree requires the Completion Report within 30 days "from the date of the EBP's completion."

[2] The Consent Decree requires data reporting to the "Beach Guard notification system." The web portal for this information is now called Beach*Alert*. The website in the Consent Decree (https://www.in.gov/idem/lakemichigan/pages/beachguard/) redirects the reader to https://portal.idem.in.gov/beachalert/.

Historical weather and marine information for southern Lake Michigan suggests that high winds, substantial waves, thunder/lightning storms, and icy conditions will be encountered during the three-year sampling program.

Therefore, the following process will be used to obtain as many valid samples as possible during the three-year program.

October – April:

1. Schedule sampling during the first week of the month to allow for delays.
2. If weather precludes sampling from all seven locations, schedule the event for the second week of the month.
3. If weather precludes sampling from all seven locations during the second week, schedule the event for the third week of the month.
4. Obtain as many samples as possible during the third week. If all seven cannot be obtained, schedule sampling of the remaining locations for the fourth week of the month.

Weather websites will be monitored, and on-the-ground conditions will be ascertained from plant personnel throughout the month. It is anticipated that all seven locations will be able to be sampled at some point during the month. However, if weather or other *force majeure* factors preclude sampling a location during a month in this period (October – April), the EBP will be extended by one month for that location.

May – September:

1. Schedule full sampling (seven locations) for Monday or Tuesday and supplemental sampling (four locations) for Wednesday or Thursday.
2. Field personnel will collect as many samples as safely possible on the scheduled event day. If storms, lightening, or other unsafe conditions preclude sampling from all planned locations, the remaining locations will be sampled the following day, if feasible.
3. Friday will be used as a back-up day to collect missed samples.

A reasonable attempt will be made to safely collect the 11 scheduled samples each week. The planned frequency of sampling will provide a robust data set; therefore, if 80% of the data for the week is available, it will be considered a comprehensive data set, and the EBP will not be extended.

3. DATA QUALITY INDICATORS

This sampling program will collect data conforming to IDEM's (2015) data quality assessment Level 3 criteria and data quality documentation will be provided to support IDEM's review. This section describes the field and laboratory activities that will be performed to document data quality; however, the sampling program does not include assessment of the data quality indicators.

3.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement (IDEM 2015). Relative percent difference (RPD) can be used to determine precision by comparing the results from an analytical sample to the field duplicate sample results. Because there will be

less than 10 samples per event, one field duplicate for all parameters will be collected per event (sample day) for this purpose. The RPD data will periodically be reviewed and if considered necessary, field sampling procedures will be reviewed and may be revised.

3.2 Accuracy and/or Bias

Accuracy is the degree to which an observed value and an accepted reference value agree (IDEM 2015). As appropriate and in accordance with contract laboratory Standard Operating Procedures (SOPs), percent recovery (%R) of reference standards can be calculated to measure accuracy of laboratory methods for chemical parameters.

For *E.coli* analyses, accuracy would be based on the sensitivity of the media as determined by media control tests.

To address bias from possible field contamination, one field blank will be collected for analysis of total chromium and hexavalent chromium during each sampling event and bias can be measured based on the standard deviation of the sample set.

Field instrumentation can be assessed for accuracy through the analysis of certified reference materials or calibration check standards, where applicable. The following schedule of checks will be completed:

Table 3. Frequency of Field Meter Quality Checks

Field Parameter	Calibration w/Reference Standards	Calibration Verification Standard
Temperature	Annual Calibration	Annual Verification(1)
pH	Day of Sampling Calibration	Minimum 1 per event (end of day)
Turbidity	Calibration at least once every three months	Minimum 2 per event (before sampling and end of day)

Notes:
(1) Temperature measurement devices will be verified for accuracy using a National Institute of Standards and Technology (NIST)-traceable reference thermometer on an annual basis.

3.3 Completeness

Completeness can be assessed against the Consent Decree's requirement to measure the eight water quality parameters in surface water collected from the seven identified locations at the frequency specified in Table 2.

As described above, weather conditions will likely prevent collection of some samples, even when the contingency times and locations are used. A reasonable attempt will be made to collect the proposed samples.

3.4 Representativeness

Representativeness is the degree to which the sample data represent an environmental condition. The general sampling vicinities along approximately 22 miles of southern Lake Michigan were identified within the Consent Decree. The proposed sampling locations herein are all within that same footprint and most are no more than one-half mile from the original vicinity. Given the

substantial mixing created by frequent windy conditions, water quality is not anticipated to vary considerably within the sample vicinities.

3.5 Comparability

Comparability is the confidence that one data set can be compared to another. Standard, reproducible techniques will be used throughout the program, and the same pool of trained field staff will be used to the extent practicable. Field notes and standardized field data sheets will be used to document sampling locations and methods that would need to be considered in subsequent monitoring programs for comparability.

3.6 Sensitivity

Sensitivity measures the ability of the method to quantify the test parameters at appropriately low concentrations. The Consent Decree specified that the analytical method used for hexavalent chromium must have a detection limit of 1 µg/L or less and the method used for total chromium's detection limit must be 2 µg/L or less. Table 4 below provides the anticipated detection limits for these parameters as well as the other analytes.

Table 4. Analytical Methods and Detection Limits

Test Method	Analyte	Matrix	MDL(1)	PQL(2)	Units(3)
EPA 200.8	Chromium	Water	0.00011	0.002	mg/L
EPA 218.6	Chromium, hexavalent (dissolved)	Water	0.026	0.25	µg/L
SM 2540 D	TSS	Water	0.3	2	mg/L
SM 9223 B	*E. coli*	Water	1	1	mpn/ 100mL
Abraxis 520022	Cyanobacteria (microcystins/nodularins)	Water	1	1	µg/L

Notes:

(1) Laboratory limits subject to change as new method detection limit (MDL) studies are performed.

(2) PQL = practical quantitation limit

(3) Units: mg/L = milligrams per liter; µg/L = micrograms per liter; mpn/100mL = most probable number per 100 milliliter

4. FIELD MONITORING REQUIREMENTS

4.1 Field Methods

All surface water samples will be collected by ALS and/or Ramboll staff and analyzed by ALS at their Valparaiso, IN facility, except for *E. coli* which will be analyzed by Utility Services in Valparaiso. Table 5 summarizes the collection methods and sample volumes needed to measure water quality parameters.

Table 5. Sampling Methods and Containers

Parameter	Sampling Method	Sample Container (minimum)	Sample Volume	Hold Time
Cr^{6+}	Grab Sample	125 mL plastic bottle	10 mL	6 months
Cr	Grab Sample	250 mL plastic bottle	50 mL	6 months
Cyanobacteria	Grab Sample	40 mL glass vial	2 mL	5 days
E. coli	Grab Sample	125 mL sterile bottle	100 mL	6 hours
pH	Field Meter (1)	In field	In field	In field
TSS	Grab Sample	1 L plastic bottle	1,000 mL	7 days
Temperature	Field Meter (1)	In field	In field	In field
Turbidity	Field Meter (1)	In field	15 mL	In field

Notes:
(1) pH and temperature will be measured using a Thermo Scientific Orion Star A121 pH meter with ATC electrode probe. A Thomas Scientific Traceable Temp Probe will be on location as a backup. Turbidity will be measured using a Hach 2100p turbidity meter.

See Appendix 1 for the following SOPs that will be used for field activities:

- HN-FIELD-001 Surface Water Sampling: collection of field samples
- HN-FIELD-007 pH for Field Determination: pH measurement in field samples
- HN-FIELD-008 Temperature Measurement: temperature measurement in field samples
- HN-WC-035 Turbidity: turbidity measurement in field samples

Surface water samples will be filtered in the field for hexavalent chromium laboratory analysis using a 60-ml syringe and 0.45-µm disc filters. Two syringe volumes per sample (120 ml total) will be filtered into a 125-mL plastic bottle.

Field measurements will be recorded in a dedicated field notebook or on a Field Data Sheet (see Appendix 2 for an example).

4.2 Field QC Activities

The Thermo Scientific Orion Star A121 pH meter with ATC electrode probe and Hach 2100p turbidity meter will be calibrated per manufacturer's instructions (see Appendix 3) following the schedule outlined in Table 3. Equipment information, date, and calibration readings will be recorded either in a dedicated field notebook or on a Calibration Record (see Appendix 2 for an example).

One field duplicate and one field blank will be collected per event (sample day) for laboratory analysis of TSS, hexavalent chromium, total chromium, *E. coli*, and cyanobacteria. One duplicate measurement will be recorded per event for temperature, pH, and turbidity.

Table 6 and Table 7 summarize specifications for the Thermo Scientific Orion Star A121 and the Hach 2100p meters, respectively.

Table 6. Thermo Scientific Orion Star A121 pH Meter with ATC Electrode Probe Specifications

	Temperature	pH
Range	5 to 105 ºC, 23 to 221 ºF	-2.00 to 16.00
Resolution	0.1 ºC, 0.1 ºF	0.1, 0.01
Repeatability	--	±0.05 pH
Accuracy	±0.1 ºC, ±0.1 ºF	±0.01
Other	Temperature offset calibration	Up to three-point calibration

Table 7. Hach 2100p Turbidity Meter Specifications

	Turbidity
Measurement method	Ratio Nephelometric signal (90°) scatter light ratio to transmitted light
Range	0–1000 FNU with automatic decimal point placement or manual range selection of 0–9.99, 0–99.9 and 0–1000 FNU
Resolution	0.01 FNU on lowest range
Accuracy	± 2% of reading plus stray light from 0–1000 FNU
Repeatability	± 1% of reading or 0.01 FNU, whichever is greater (with Gelex standards)

5. LABORATORY ANALYTICAL REQUIREMENTS

The ALS Environmental Quality Assurance Manual (Appendix 4) provides laboratory-specific QA/QC information.

Laboratory SOPs for the following analytical methods are outlined in Appendix 5:

- VAL-MET-001 Metals by ICP-MS
- VAL-MET-002 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP-MS Spectroscopy
- VAL-WC-013 Determination of Hexavalent Chromium by Ion Chromatography
- VAL-WC-001 Total Suspended Solids
- Eurofins Abraxis Microcystins Strip Test
- USC215 Total and Fecal Coliform Enumeration Using the Quanti-Tray Method

6. SAMPLE HANDLING AND CUSTODY REQUIREMENTS

An essential component of the sampling program is the integrity of the individual samples from the time of collection through analysis and final storage or destruction. Possession of the sample must be traceable, identifying all personnel handling the samples (e.g., sampler, transporter [if possible] and analyst). Chain-of-Custody (COC) procedures and records will be followed and maintained in order to document sample possession. See Appendix 6 for an example COC form.

The integrity of the samples will be maintained by adhering to the following COC protocol:

1. Properly sealed and labeled sample containers will be individually bagged in plastic, sealable bags and then collectively placed into a second, larger plastic bag. The bagged samples will be surrounded by ice in coolers. *E. coli* samples will be kept in a separate cooler to expedite transfer of the samples within the 6-hour hold time.
2. Once samples have been collected, COC forms will be completed, sealed in a ziplock bag, and placed in the respective coolers (*E. coli* samples will be recorded on a separate COC form).
3. The coolers will be taped around the top and bottom and both sides.
4. A custody seal will be signed and dated by a member of the field team and affixed to each cooler.
5. An ALS courier or field team member will transport the coolers to ALS Valparaiso and Utility Services (for *E. coli* samples).
6. ALS and Utility Services will receive the coolers and obtain custody following their internal procedures.

When transferring samples, the individuals relinquishing or receiving the samples will sign, date, and note the time of transfer on the COC record. COC records will be completed for each sampling event and will contain the minimum following information:

- Unique sample identification number,
- Signature of the collector,
- Date and time of collection,
- Sample type (e.g., grab),
- Sampling location,
- Shuttle (cooler) contents,
- Parameters requested for analysis (i.e., hexavalent chromium, total chromium, E. coli, cyanobacteria, and TSS),
- Signature of person(s) involved in the chain of possession,
- Inclusive dates of possession, and
- Pertinent comments or remarks (e.g., an alternate sampling location).

Laboratory analysis of hexavalent chromium, total chromium, cyanobacteria, and TSS will be performed at ALS Valparaiso and *E.coli* at Utility Services in Valparaiso, IN.

7. INSPECTION AND MAINTENANCE

The Thermo Scientific Orion Star A121 pH meter with ATC electrode probe and Hach 2100p turbidity meter will be visually inspected prior to each sampling event for obvious issues that could affect performance (e.g., a frayed cord or cracked screen). Regular maintenance will follow manufacturer's instructions (see Appendix 3).

Laboratory instrumentation testing and calibration information is outlined in Appendix 4, ALS Environmental's Quality Assurance Manual.

8. DATA QUALITY ASSESSMENT AND DECISION RULES

8.1 Data Quality Indicators

8.1.1 Precision

Per the IDEM 2015 Guidance, the acceptable level of precision (as measured with duplicates) for Tier 3 data quality for lakes is +/- 2 standard deviations (SD). All collected data will be submitted to IDEM and available for IDEM evaluation.

8.1.2 Accuracy / Bias

Per the IDEM 2015 Guidance, the upper and lower warning limits for bias in lake samples are +/- 2 SD and upper and lower control limits are +/- 3 SD. Detections above the upper warning limit are considered suspect but usable; detections above the control limit will be rejected. All collected data will be submitted to IDEM and available for IDEM evaluation.

For bacteriology, the following media controls must be obtained:

- A sterility control sample (*E. coli*, Fecal Coliform and Total Coliforms)
- *Pseudomonas aeruginosa* (PA) Negative (*E. coli*)
- *Klebsiella pneumoniae* (KP) Positive Culture (*E. coli*)
- *Escherichia coli* (EC) Positive Culture (*E. coli*)

8.1.3 Completeness

The data quality goal for completeness is 100%. Completeness for this project will be assessed by comparing the data obtained to those proposed. If specific parameters or entire samples cannot be collected/measured, the field team will confer with U. S. Steel, Ramboll, and ALS Quality Managers to determine the potential effect on data usability.

8.2 Analytical QC Criteria

Table 8 summarizes quality control sample information and criteria to assess measurement performance.

Table 8. ALS – Valparaiso, IN QC Criteria for Parameters to Be Analyzed in the Laboratory

Matrix	Water
Analytical Group	Chromium, Total Recoverable
Analytical Method / SOP Reference	EPA 200.8 VAL-MET-001
Analytical Organization	ALS – Valparaiso, IN

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike/Duplicate (MS/MSD)	One per matrix per analytical method for each batch of at most 20 samples per site.	Per Method and Lab SOP	Examine the project-specific DQOs. Notify lab QA Officer and Project Chemist of additional measures to be taken.	Analyst Project Chemist	Accuracy/ Precision	Recovery = 70-130% RPD < 20%
Laboratory Control Sample (LCS)	Each group of 20 or less prior to analysis of samples.	Per Method and Lab SOP	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Accuracy	Recovery = 85-115%
Method Blank	Each group of 20 or less prior to analysis of samples.	No analytes detected ≥ PQL	Correct problem, then re-prep and reanalyze the MB and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Sensitivity	No analytes detected ≥ PQL
Continuing Calibration Check	Prior to sample analysis, every 10 samples, end of sequence.	Per Method and Lab SOP	Rerun CCC. If it does not pass, correct problem, recalibrate, then reanalyze all samples bracketing the failure.	Analyst Project Chemist	Accuracy	Recovery = 90-110%
Serial Dilution	Each group of 20 or less samples.	Per Method and Lab SOP.	Correct problem, recalibrate, and reanalyze all samples in the associated batch for failed analytes in the associated batch	Analyst Project Chemist	Precision	RPD < 10% for analytes 25x Reporting Limit
Interference Check (ICSA)	Prior to sample analysis and end of sequence	Per Method and Lab SOP	Correct problem, recalibrate, and reanalyze all samples in the associated run for failed analytes	Analyst Project Chemist	Accuracy	Spiked Analytes: Recovery 80-120 Unspiked Analytes: Not detected ≥ PQL

Matrix	Water
Analytical Group	Hexavalent Chromium (Dissolved)
Analytical Method / SOP Reference	EPA 218.6 VAL-WC-013
Analytical Organization	ALS – Valparaiso, IN

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One per matrix per analytical method for each batch of at most 20 samples per site.	Per Method and Lab SOP	Examine the project-specific DQOs. Notify lab QA Officer and Project Chemist of additional measures to be taken.	Analyst Project Chemist	Accuracy/ Precision	Recovery = 90-110%
LCS	Each group of 20 or less prior or analysis of samples.	Per Method and Lab SOP	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Accuracy	Recovery = 90-110%
Method Blank	Each group of 20 or less prior or analysis of samples.	No analytes detected ≥ PQL	Correct problem, then re-prep and reanalyze the MB and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Sensitivity	No analytes detected ≥ PQL
Continuing Calibration Check	Prior to sample analysis, every 10 samples, end of sequence.	Per Method and Lab SOP	Rerun CCC. If it does not pass, correct problem, recalibrate, then reanalyze all samples bracketing the failure.	Analyst Project Chemist	Accuracy	Recovery = 95-105%

Matrix	Water
Analytical Group	Total Suspended Solids
Analytical Method / SOP Reference	SM 2540 D VAL-WC-001
Analytical Organization	ALS – Valparaiso, IN

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample Duplicate (DUP)	One per matrix per analytical method per every 10 samples per each batch of at most 20 samples per site.	Per Method and Lab SOP	Examine the project-specific DQOs. Notify lab QA Officer and Project Chemist of additional measures to be taken.	Analyst Project Chemist	Precision	RPD < 10%
LCS	Each group of 10 or less prior or analysis of samples.	Per Method and Lab SOP	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Accuracy	Recovery = 80-115%
Method Blank	Each group of 20 or less prior or analysis of samples.	No analytes detected ≥ PQL	Correct problem, then re-prep and reanalyze the MB and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Sensitivity	No analytes detected ≥ PQL
Calibration Verification	Daily, prior to analysis of samples	Per Method and Lab SOP	Correct problem, re-run calibration verification.	Analyst Project Chemist	Accuracy	Within tolerance as defined in SOP.

Matrix	Water
Analytical Group	E. coli
Analytical Method / SOP Reference	SM 9223 B USC-215
Analytical Organization	Utility Services – Valparaiso, IN

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample Duplicate (DUP)	One per matrix per analytical method per sampling event.	Per Method and Lab SOP	Examine the project-specific DQOs. Notify lab QA Officer and Project Chemist of additional measures to be taken.	Analyst Project Chemist	Precision	Report
Method Blank	Each group of 20 or less prior or analysis of samples.	No analytes detected ≥ PQL	Correct problem, then re-prep and reanalyze the MB and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Sensitivity	No analytes detected ≥ PQL
Media Controls (KP, PA, EC cultures)	Once per lot of media	Per Method and Lab SOP	Correct problem, obtain new media prior to use for sample analysis.	Analyst Project Chemist	Positive / Negative	KP = Positive PA = Negative EC = Positive

KP = *Klebsiella pneumoniae*, PA = *Pseudomonas aeru*ginosa, EC = *Escherichia coli*

Matrix	Water
Analytical Group	Cyanobacteria
Analytical Method / SOP Reference	Abraxis 520022
Analytical Organization	Utility Services – Valparaiso, IN

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample Duplicate (DUP)	One per matrix per analytical method per sampling event.	Per Method and Lab SOP	Examine the project-specific DQOs. Notify lab QA Officer and Project Chemist of additional measures to be taken.	Analyst Project Chemist	Precision	<20%
Method Blank	Once per day.	Per Method and Lab SOP	Correct problem, initiate resampling event.	Analyst Project Chemist	Sensitivity	Concentration < RL
LCS	Each group of 20 or less prior or analysis of samples.	Per Method and Lab SOP	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient material is available.	Analyst Project Chemist	Accuracy	Recovery = 75-125%

8.3 Corrective Action

Corrective actions taken in the field will be documented in writing in field logbooks and reported to the Project Manager. Corrective actions for laboratory analysis will follow ALS or Utility Services (*for E. coli*) protocols.

9. PERFORMANCE AND SYSTEM AUDITS

It is anticipated that IDEM may conduct an external performance and/or systems audit of the EBP. The Field Team Lead will also perform internal quality audits using the checklist attached to the IDEM (2015)[3] *Certification Form for Submission of External Data for OWQ Tier 2 and Tier 3 Uses* (Appendix 7) monthly May through September and twice during the period of October through April.

10. PREVENTIVE MAINTENANCE

General maintenance of the Thermo Scientific Orion Star A121 pH meter with ATC electrode probe and Hach 2100p turbidity meter will be performed according to manufacturer's instructions (Appendix 3).

Maintenance requirements of ALS laboratory equipment are described in Appendix 4.

11. DATA REVIEW, VERIFICATION, AND VALIDATION

SOP VAL-QS-009 (Appendix 8) provides the procedures for data reduction, review, and validation of analytical results produced by ALS Environmental. These procedures are performed either manually or through the use of computer programs associated with the analytical instruments and/or the LIMS system. Reviews are employed to ensure processes are carried out to meet project and/or method QC requirements and to ensure the validity of the data for its intended use.

The data validation process consists of the following reviews:

- Preliminary review of information regarding samples (e.g., sample collection, preservation, holding time requirements, condition upon arrival).
- Evaluation of QC analysis data (e.g., calibrations, blanks, spikes, replicates, etc.) against method and/or project specified QC limits
- Evaluation of compliance with project requirements.
- Comparison of sample results against raw data to identify and correct transcription errors.
- A peer review of all analytical data.
- A review for reasonableness as part of an independent assessment of the data prior to being reported to the client.

Data qualifiers will be applied in accordance with SOP VAL-ADM-005, Report Formatting (Appendix 8).

[3] Indiana Department of Environmental Management (IDEM). 2015. Technical Guidance for the Office of Water Quality External Data Framework. Prepared by Jody Arthur, Watershed Assessment and Planning Branch, Office of Water Quality September 23, 2015.

12. REPORTS, DOCUMENTATION, AND RECORDS

The field team will perform the first data check for missing information and recording errors in field-recorded data at the end of each sampling event. The field notebook and datasheets will be checked for completeness and obvious data issues (e.g., a temperature reading substantially higher or lower than the other readings for the event). Missing or incorrect information will be rectified at that time.

Analytical data will be reviewed in accordance with SOP VAL-QS-009, Data Reduction, Review, and Validation (Appendix 8).

12.1 Data Reporting

Concentrations and measurements of the eight surface water quality parameters will be reported weekly to IDEM from May through September (by Wednesday of the week following sample collection) and monthly from October through April (by the 5th business day of the month following sample collection), as required by the Consent Decree. The dates and times of the sampling events and results of measurement of the parameters will be submitted to IDEM and be posted at U. S. Steel's website (https://midwest.uss.com)[4].

A summary of the monitoring program and its results will be compiled in yearly reports submitted to IDEM by and published on the U. S. Steel website by March 31 of each subsequent year.

E. coli concentrations will also be entered into the Beach*Alert* notification system electronically at this website: https://portal.idem.in.gov/beachalert/. Concentrations will be entered within eight hours of receiving the results from the laboratory.

The raw *E. coli*, hexavalent chromium, and total chromium data will also be submitted to IDEM and USEPA within eight hours of receiving the results from the laboratory. These data will be submitted as a Level II ALS laboratory report with incorporation of the subcontractor *E. coli* results.

The monitoring results and data quality documentation (e.g., laboratory data validation reports) will be submitted to the External Data Framework through IDEM's Secondary Data Portal (https://www.hoosierriverwatch.com/portal/) at least annually.

12.2 Quality Assurance Reports

Quality audit information described in Section 9 will be kept on file until completion of the program. The IDEM (2015) checklist attached to the *Certification Form for Submission of External Data for OWQ Tier 2 and Tier 3 Uses* (Appendix 7) will be used for internal audits.

ALS Environmental laboratory audit reports are described in Appendix 4.

[4] The correct web address to reach the Midwest Plant page is https://midwest.uss.com. "www.midwest.uss.com" as stated in the Consent Decree leads to an error page.

APPENDIX 1
FIELD STANDARD OPERATING PROCEDURES

ALS Standard Operating Procedure

DOCUMENT TITLE:	SURFACE WATER SAMPLING
REFERENCED METHOD:	SW846 CHAPTER 1, SECTION 3
SOP ID:	HN-FIELD-001
REV. NUMBER:	R04
EFFECTIVE DATE:	01/31/2016

SURFACE WATER SAMPLING

SW846 CHAPTER 1, SECTION 3

SOPID:	HN-Field-001	Rev. Number:	R04	Effective Date:	01/31/2016

Approved By: _____ Date: 1/27/16
Department Supervisor

Approved By: _____ Date: 1/28/16
Operations Manager

Approved By: _____ Date: 1/28/16
Quality Assurance

Approved By: _____ Date: 1/28/16
Laboratory Director

Archival Date:	_____	Doc Control ID#:	_____	Editor:	_____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWENTY FOUR ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
	QA Manager	5/22/2018

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SURFACE WATER SAMPLING

1) Scope and Applicability

1.1 This SOP describes the requirements and procedures for collection/containment of surface water samples.

2) Summary of Procedure

2.1 Surface water samples are collected according to defined procedure, or alternatively, project specified work plans, to ascertain the types and/or levels of potential contamination.

2.2 The procedures described herein are applicable to a variety of aqueous matrices including potable water, fresh water, and saline water (fresh and saline).

3) Definitions

3.1 Surface Water: Water that flows over or is retained on the land and which is open to the atmosphere.

3.2 Shallow Surface Water: Water depth that is within 1 – 3.3 feet of the body's surface.

3.3 Deep Surface Water: Water depth that is > 3.3 feet from the body's surface.

3.4 Grab Sample: A sample aliquot taken from a discrete point at a specified period of time.

3.5 Composite Sample: Two or more sub-samples taken from specified points over a period of time and combined into a single sample.

3.6 De-ionized Water: Distilled or de-ionized water achieving quality criteria of Type 2 or better water criteria.

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.

4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.

4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.
4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Peristaltic pumps cannot generally lift water over distances greater than 20 feet. Alternative pump types should be used for the sampling of deep surface water exceeding a depth of > 20 feet.

5.2 When sampling for volatile analytes or other compounds subject to atmospheric degradation, exercise all possible caution towards minimizing sample disturbance.

6) Interferences

6.1 N/A

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This SOP is restricted to use by or under the supervision of experienced field personnel.

7.2 Field Personnel – It is the responsibility of field personnel to:

7.2.1 Perform surface water sampling according to the procedures outlined in this SOP or as defined in a project specific work plan,
7.2.2 Maintain complete and legible documentation as to sampling parameters and/or sample characteristics.

7.3 Field Service Supervisor – It is the responsibility of the section supervisor to:

7.3.1 Ensure that all field personnel have both the technical ability and necessary training to perform this procedure,
7.3.2 Ensure that all field personnel perform surface water sampling according to the defined procedures of this document, and
7.3.3 Ensure that all required documentation is maintained for the traceability and verification of sample integrity.

8) Sample Collection, Handling, and Preservation

8.1 See Section 8 of the applicable analytical SOP.

9) Equipment and Supplies

9.1 Field Log

9.2 Waterproof pens and markers

9.3 Appropriate sample containers w/labels

9.4 Plastic zip-lock bags

9.5 Ice-bags or blue-ice

9.6 Shipping coolers w/tape

9.7 Custody seals

9.8 Shipping labels and documentation

9.9 Protective clothing and equipment

9.10 De-ionized water

9.11 Plastic sheeting

9.12 Project Specific material:

9.12.1 Sampler with 1 L Teflon container, clamp, and telescoping pole
9.12.2 Weighted 1 L Teflon bottle with ½ in braided nylon rope
9.12.3 Peristaltic (or equivalent) pump w/clean Teflon tubing
9.12.4 Low flow meter capable of simultaneous measurement of temperature, pH, conductivity, and dissolved oxygen
9.12.5 Boat with depth finder
9.12.6 Global positioning system (GPS)
9.12.7 Spare parts for Sections 9.12.1 thru 9.12.6.

10) Standards and Reagents

10.1 Reference the project specific work plan or analytical SOP for required sample preservatives.

11) Method Calibration

11.1 All metering equipment must be calibrated according to individual equipment technical specifications.

12) Sample Preparation/Analysis

12.1 Preparation

12.1.1 Utilize appropriate protective equipment as dictated by the work plan, site health & safety plan, and/or site-specific hazards.
12.1.2 Select sampling locations representative of sample homogeneity. Avoid areas that may present bias to the sampling event (i.e., constrictions, bends, falls, stratification, etc).
12.1.3 Place clean plastic sheeting on an available flat surface and lay out required sampling equipment.

12.1.3.1 Verify cleanliness of equipment prior to use
12.1.3.2 Install clean Teflon tubing on any pumps to be utilized
12.1.3.3 Prepare any other needed equipment prior to use

12.1.4 Conduct and record project specific field measurements relative to water characteristics (temperature, pH, conductivity, etc)
12.1.5 Locate sampling points on the site map and describe in the field log. Utilize

GPS if possible to detail location.

12.1.5.1 If known, sampling points should be located from the least to the most contaminated area.
12.1.5.2 When collecting multiple samples from moving water, collect from downstream to upstream.

12.1.6 Prepare labels for each sample container with the appropriate information. Secure with waterproof tape after each individual sampling event is completed.
12.1.7 Document sampling events and record all information in the field log. Document all deviations and/or modifications from this SOP and/or site work plan in the field log.

12.2 Shallow Surface Water Sample Collection

12.2.1 Collection of Volatile Organic Compounds

12.2.1.1 Non-Preserved Samples

12.2.1.1.1 Approach sample location from downstream.
12.2.1.1.2 Gently submerge a VOA vial with opening facing upstream
12.2.1.1.3 Allow vial to fill completely.
12.2.1.1.4 Cap the vial while underwater. Dislodge air bubbles from the cap prior to sealing vial.
12.2.1.1.5 Check the sealed VOA vial for air bubbles by inverting. If any bubbles are present greater than pea-sized, discard and resample.
12.2.1.1.6 Proceed to Section 12.2.1.2.5.

12.2.1.2 Preserved Samples

12.2.1.2.1 Approach sample location from downstream.
12.2.1.2.2 Gently submerge a clean glass jar and fill
12.2.1.2.3 Immediately decant into a clean, preserved VOA vial with minimal agitation. Fill the vial completely.
12.2.1.2.4 Cap each vial. Invert the vial and check for air bubbles. If any bubbles are present greater than pea-sized, discard and resample.
12.2.1.2.5 Dry the outside of the vial with a clean paper towel and affix the completed sample label.
12.2.1.2.6 Place the sample(s) in a zip-lock bag.
12.2.1.2.7 Immediately pack all samples into a chilled cooler.

12.2.2 Collection of Non-Volatile and Inorganic Compounds

12.2.2.1 Dip the sample container into the water with the opening facing upstream and fill.

12.2.2.2 If compositing or filtering is required, the sample(s) may be collected in a Teflon container and then filtered or decanted into the sample container.
12.2.2.3 Add required preservation as documented in the work plan or applicable analytical protocol.
12.2.2.4 Cap the container and wipe dry with a clean paper towel. Affix the completed sample label.
12.2.2.5 Place the sample(s) in a zip-lock bag.
12.2.2.6 Immediately pack all samples into a chilled cooler.

12.3 Deep Surface Water Sample Collection

12.3.1 Weighted Bottle Sampling

12.3.1.1 Using a previously marked rope, lower a weighted bottle sample to the specified sampling depth.
12.3.1.2 Open the bottle sampler by tightening the sampler line and allow the bottle to fill.
12.3.1.3 Release the sampler line to close the bottle.
12.3.1.4 Return the bottle to the surface and wipe dry with a clean paper towel.
12.3.1.5 Decant sample into the appropriate sample container.

12.3.1.5.1 Complete VOA sampling first according to the details outlined in Section 12.2.1.2.3 thru 12.2.1.2.7.
12.3.1.5.2 For non-volatile and inorganic compounds, complete sampling according to the details outlined in Section 12.2.2.2 thru 12.2.2.6.

12.3.2 Peristaltic Pump

12.3.2.1 Install clean unused Teflon tubing on the pump head discharge. Install sufficient tubing for ease of dispensing into sample containers.
12.3.2.2 Attach previously measured and clean unused Teflon tubing to the intake. (Previously measured to specified sampling depth.)
12.3.2.3 Lower the intake tubing to the specified sampling depth and start the pump.
12.3.2.4 Allow approximately 1.0 L of water to flow through and rinse the system prior to sample collection. To minimize sampling disturbances in the water body, collect purged water in a separate container and return to the water body after sampling is completed.
12.3.2.5 Pump sample from the discharge tube into the appropriate sample container.

12.3.2.5.1 Complete VOA sampling first according to the details outlined in Section 12.2.1.2.3 thru 12.2.1.2.7.
12.3.2.5.2 For non-volatile and inorganic compounds, complete sampling according to the details outlined in Section 12.2.2.2 thru 12.2.2.6.

12.3.2.6 Drain the pump, rinse with DI water, and wipe dry.
12.3.2.7 Replace all tubing with clean unused tubing prior to the next sampling event. Place used tubing in an appropriately marked bag for subsequent disposal or decontamination.

13) Troubleshooting

13.1 Refer to individual equipment technical manuals.

14) Data Acquisition

14.1 Record all applicable information in the appropriate field log.

15) Calculation, and Data Reduction Requirements

15.1 N/A

16) Quality Control, Data Assessment and Corrective Action

16.1 Equipment blanks, equipment rinses, trip blanks, and duplicate sampling must be completed for each sampling event.

17) Data Records Management

17.1 All data and field records must be maintained in the designated field log.

17.2 All field log entries must be complete, legible, dated, and initialed.

17.3 All field records shall be maintained for a period of no less than ten (10) years.

18) Quality Assurance and Quality Control

18.1 Field logs must be reviewed monthly by the department supervisor.

18.2 Field logs must be reviewed quarterly by the QA staff.

19) Contingencies for Handling Out of Control Data

19.1 Report any deviations from acceptable criteria via the NC/CA database.

20) Method Performance

20.1 N/A

21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R03	7/1/12	CES	Formatting
R04	01/31/16	CES	Removed sec 7.2.3; added new sec 9.7; revised sec 17.3 and secs 22.1 and 22.2.

22) References and Related Documents

22.1 ALS Environmental Quality Assurance Manual, Revision (most current)

22.2 Standard Methods for the Examination of Water and Wastewater, on-line.

22.3 US EPA SW846, Chapter 1, Section 3, 1997

22.4 Camp, Dresser, and McKee, Sampling Procedure

ALS Standard Operating Procedure

DOCUMENT TITLE:	PH DETERMINATION FOR FIELD ANALYSIS
REFERENCED METHOD:	SM 4500-H^+ B
SOP ID:	HN-FIELD-007
REV. NUMBER:	R01
EFFECTIVE DATE:	01/31/2016

PH DETERMINATION FOR FIELD ANALYSIS

SM 4500-H+ B

SOPID:	HN-Field-007	Rev. Number:	R01	Effective Date:	01/31/2016

Approved By: _____ Date: 2/1/16
Department Supervisor

Approved By: _____ Date: 1/28/16
Operations Manager

Approved By: _____ Date: 1/28/16
QA Manager

Approved By: _____ Date: 1/28/16
Laboratory Director

Archival Date:	_____	Doc Control ID#:	_____	Editor:	_____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
	QA Manager	5/22/2018

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PH DETERMINATION FOR FIELD ANALYSIS

1) Scope and Applicability

1.1 This method is applicable to determining the pH of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain (atmospheric deposition).

1.2 This method is adapted from the Standard Methods for the Examination of Waste and Wastewater, Method 4500-H^+ B 2011, "pH Value (Electrometric)."

1.3 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current standards in effect for the National Environmental Laboratory Accreditation Program (NELAP).

1.4 Individual project requirements may override criteria listed in this SOP.

2) Summary of Procedure

2.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

3) Definitions

3.1 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 ALS maintains Material Safety Data Sheets (MSDSs) on all chemicals used in this procedure. ALS recommends that all individuals performing this SOP familiarize themselves with the MSDSs associated with the procedure prior to SOP performance. MSDSs are available to all staff and are located in hard copy in the MSDS reference library.

5.2 All possible steps shall be taken to limit the analyst contact with chemicals and samples. In field sampling, the minimum personal protective equipment (PPE) requirements are appropriate chemical resistant gloves and safety glasses. This PPE shall reduce the possibility of contact to a safe level, but the analyst shall not limit themselves to these PPE minimums.

5.3 In addition to the PPE minimums required above, a face shield shall be worn at all times while dispensing, diluting or handling any quantity of concentrated acid.

5.4 Analysts should always exercise caution when handling samples since the chemical and biological composition of the samples is unknown.

5.5 The health hazards of each substance used in this method may not have been fully established. Each substance shall be regarded as a potential health hazard and exposure shall be as low as reasonably achievable.

5.6 Care shall be used in handling the pH buffer solutions, the use of gloves and protective eyewear is required.

6) Interferences

6.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

6.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a “low sodium error” electrode.

6.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by deionized water rinsing. An additional treatment with HCl (1 + 9) may be necessary to remove any remaining film.

6.4 Temperature effects of the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled; it shall therefore be noted by reporting both the pH and the temperature at the time of analysis.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.

7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).
7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances
7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Sample collection, preservation, and handling shall be performed according to SOP HN-Field-005.

8.2 Samples for field pH measurement shall be collected in a clean glass or plastic container. A minimum amount of 50 mL (or enough sample to ensure the electrode is immersed) is required.

8.3 A field pH reading must be obtained within 15 minutes of sample collection.

8.4 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers shall be filled completely and kept sealed prior to analysis.

9) Equipment and Supplies

9.1 pH meter with means for temperature compensation accurate and reproducible to 0.01 pH units with a range of 0 to 14; ALS Field Services currently uses the following pH meter, though any equivalent meter may be used:

9.1.1 Eutech Instrument pHTestr 30 (equivalent to Oakton pHTestr 3+)
9.1.2 Orion Star A121

10) Standards and Reagents

10.1 Primary Standard Buffers - NIST traceable, purchased from VWR. Stored at room temperature and labeled with an expiration date of 2 years from receipt. Manufacturer's labeled expiration dates, when provided, take precedent over all other expiration dates.

10.1.1 pH 4.01 – catalog #34170-154, or equivalent.

10.1.2 pH 7.00 – catalog #34170-157, or equivalent.

10.1.3 pH 10.01 – catalog #34170-160, or equivalent.

10.2 ASTM Type II Reagent Water (DI water)

11) Method Calibration

NOTE: Each instrument/electrode system shall be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Each instrument is calibrated prior to each day of use. Readings must fall within ± 0.05 pH units of true value during the initial calibration. After a successful initial calibration, a Check Standard must be read. The Check standard must be within ± 0.1 pH units accuracy of the standard used. If this is not obtained, the initial calibration must be repeated.

11.1 Calibration of the Eutech Instruments pHTestr 30 (or Oakton pHTestr 3+)

11.1.1 The hand-held tester is calibrated in "USA" mode using buffers pH 4.01, pH 7.00, and pH 10.01.

11.1.1.1 While pressing the HOLD/ENT button, switch on the Testr by pressing the ON/OFF button.
11.1.1.2 Release the HOLD/ENT button. The display will flash either USA or NIST. Press CAL button to toggle between modes.
11.1.1.3 Press HOLD/ENT button to confirm the selection.

11.1.2 Press ON/OFF button to switch unit on.
11.1.3 Dip electrode about 2-3 cm into the pH 4.01 standard buffer solution.
11.1.4 Press the CAL button to enter calibration mode. The CAL indicator will be shown. The upper display will show the measured reading based on the last calibration while the lower display will indicate the pH buffer solution.
11.1.5 Allow about 2 minutes for the Testr reading to stabilize before pressing the HOLD/ENT button to confirm the 1st calibration point. The upper display will be calibrated to the pH standard buffer solution and the lower display will then search for the next pH standard buffer solution.
11.1.6 Rinse electrode with DI before proceeding.
11.1.7 Repeat 11.1.3 through 11.1.6 for the pH 10.01 buffer.
11.1.8 After the 2nd buffer is read, press CAL to return to measurement mode.
11.1.9 Read the pH 7.00 buffer for verification of the calibration. Buffer must read within ± 0.1 pH units.

11.2 Calibration of the Orion Star A121 meter

11.2.1 Press mode/enter to display pH for pH measurement mode.

11.2.2 Select fresh pH buffers 4.01 and 10.01.
11.2.3 Press CAL. Rinse electrode with DI water, blot dry, and place into buffer solution.
11.2.4 Wait for "READY" to appear.

11.2.4.1 With automatic buffer recognition (AUTO CAL appears at the top of the display)
11.2.4.2 Repeat steps 11.2.3 and 11.2.4 with pH 10.01 buffer solution.

11.2.5 When finished, press mode/enter to save and end calibration.
11.2.6 The slope will be displayed and the meter will automatically proceed to measurement mode. Ensure slope is within probe specifications prior to proceeding with analysis.
11.2.7 Verify the calibration with the measurement of the pH 7.00 buffer solution. Buffer must read within ± 0.1 pH units.

12) Sample Preparation/Analysis

12.1 Field Measurements with pH Testr meter

12.1.1 Collect enough sample to cover the end of the probe.
12.1.2 Turn on the instrument (meter automatically goes to the 'measure' mode). Remove electrode from its storage solution (normally 4 Buffer) and rinse thoroughly with deionized water and then rinse with the sample. Gently shake off excess water.
12.1.3 Place electrode into the container containing the sample. Swirl gently for a few seconds to provide homogeneity and suspension of solids and then allow the probes to stand in the sample until the reading stabilizes. The constant rate of swirling shall minimize the air transfer rate at the air water interface of the sample.

12.1.3.1 If the sample temperature differs by more than 2 ºC from the buffer solution the measured pH values must be corrected. Instruments are equipped with automatic compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

12.1.4 Record the pH and temperature in the field log when readings have stabilized.
12.1.5 When complete, remove the electrode and rinse thoroughly with deionized water. Gently shake off excess water.
12.1.6 Place electrode back into its storage solution and turn off instrument.

12.2 Field Measurements with Orion Star A121 meter

12.2.1 Press mode/enter to display "pH" for pH measurement mode.
12.2.2 Rinse the electrode with DI water, blot dry and place into sample, covering the end of the probe.
12.2.3 If in "AUTO-READ" mode press "measure". If the meter is in continuous read mode, the meter will immediately start taking readings.
12.2.4 Record the pH and temperature of the sample in the field log when "READY" is displayed and "pH" stops blinking.
12.2.5 When complete, remove the electrode and rinse thoroughly with deionized water. Gently shake off excess water.

12.2.6 Place electrode back into its storage solution and turn off instrument.

12.3 Electrode Care

12.3.1 The pH electrode during non-use periods shall be immersed in a pH 4 buffer solution. NEVER store the electrode in DI water, as it will foul the probe.

13) Troubleshooting

13.1 Refer to pH meter instructions manuals for further guidance.

14) Data Acquisition

14.1 Report field pH to the nearest 0.01 unit and temperature to the nearest 0.1ºC.

14.2 Report results on the chain of custody and also document in the field log. Logbook entries shall contain time, date, and the sampler's initials.

14.3 Results that fall outside of know guidelines (i.e. a pH reading *below* 6.00 or *above* 9.00 at a NPDES outfall at a facility that has permitted discharge limits of a pH *between* 6.00 and 9.00) shall be reported immediately to the established contact at the facility. It is the responsibility of the individual technician to know when this action is appropriate and who the contact is.

15) Calculation, and Data Reduction Requirements

15.1 QC Calculations:

15.1.1 Relative Standard Deviation (RSD) for duplicate measurements.

$$\%RSD = \frac{Sample\ Standard\ Deviation\ (S)}{Mean\ Recovered\ Concentration}$$

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Demonstration of Capability (DOC)

16.1.1 Each technician shall complete a successful Initial Demonstration of Capability (IDOC) to become qualified to work independently to conduct this method. Each qualified technician shall perform an annual DOC for ongoing proficiency as specified in the QA Manual, Technical Training or when significant changes in instrumentation are made.

16.1.2 Analyze four replicates of the pH standard according to the analysis procedure. Calculate the relative standard deviation (RSD).

16.1.3 Acceptance Criterion:

16.1.3.1 Accuracy: All four results shall be within 10% (± 0.1 standard pH units) of the true value

16.1.3.2 Precision: RSD shall be <10%

16.1.3.3 If this acceptance criterion is met, performance is judged acceptable and independent sample (preparation) analysis may begin. If data is not acceptable, find and correct the source of the problem, then repeat the analysis. The DOC must be acceptable before independent analysis begins.

16.2 Following initial calibration, after every 10 field samples, a continuing calibration standard will be read at 7.00 pH standard units. This reading must be within ± 0.1 pH units of the true value. If the reading is out of range, a recalibration must be done.

16.2.1 Continuing calibration checks may not be possible in all field situations (i.e. low flow sampling using a flow cell and taking readings every five minutes since it would impede stabilization). In low flow and certain other well sampling situations, it may only be practical to calibrate initially in the field and then do an 'end of event' calibration check. Therefore it is imperative that equipment used on these events be serviced thoroughly preceding the scheduled event.

16.2.2 When sampling several different types of wastes streams or sampling at several unrelated different locations, it may be necessary to exceed the frequency of checks samples listed in section 16.2. It is up to the individual Field Services Technician to use his or hers Best Professional Judgment when this may be necessary.

16.3 Documentation of initial and continuing calibrations will be documented in the field services logbook and in the appropriate maintenance or calibration logbook.

16.4 Limits for initial calibration standards are ± 0.05 standard pH units. If the reading is greater than ± 0.05 pH standard units from true value, re-calibrate the meter using procedures listed in Section 11. Limits for a check standard (or continuing standard) are ± 0.1 standard pH units. If this is not obtained, re-calibrate the meter using procedures listed in Section 11.

16.5 All containers used to transport calibration buffers to the sampling site for calibrations in the field shall be properly labeled with the value of the buffer (i.e. pH 7.00), the lot number (listed on the container the portion was taken from), the expiration date (listed on the container the portion was taken from), and the date the portion was filled into the individual field container.

16.6 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for no less than 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis can not be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 10% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) are not applicable to this procedure.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R01	1/31/16	CES	New SOP

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, EPA/600/4-79-020, Method 150.1.

21.2 Standard Methods for Examination of Water and Wastewater 4500-H^+ B, online Edition.

21.3 Test Methods for Evaluating Solid Waste Physical/Chemical Methods SW-846, Method 9040C and 9045D.

21.4 ALS Environmental Quality Assurance Manual Revision (current version)

TEMPERATURE MEASUREMENT

METHOD: SM 2550B-2010

DOCUMENT I.D. HN-FIELD-008-R00

Approved By: _____ Date: 8/23/17
Client Services Supervisor, Keith Wierenga

Approved By: _____ Date: 8/23/17
Laboratory Director, Jeff Glaser

Prepared By: _____ Date: 8/23/17
Quality Assurance Manager, Chad Stoike

Periodic Review:

Reviewed By: _____ Date: 8/20/19

Reviewed By: _____ Date: _____

Reviewed By: _____ Date: _____

Reviewed By: _____ Date: _____

Doc Control ID:	_____	Archived Date:	_____

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1) Scope & Applicability

1.1 This SOP establishes the minimum requirements for checking and recording temperature of environmental samples in the field.

1.2 This method is adopted from "Standard Methods for the Examination of Water and Wastewater" Method 2550B 2010, "Temperature".

2) Summary of Procedure

2.1 The temperature of a sample is determined in the field at the time of sampling using a field meter equipped with a temperature probe.

3) Definitions

3.1 PPE – Personal Protective Equipment

4) Responsibilities

4.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

4.2 Analyst - It is the responsibility of the analyst(s) to:

4.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.
4.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).
4.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
4.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
4.2.5 The analysts must submit data for peer or supervisor review.

4.3 Section Supervisor - It is the responsibility of the section supervisor to:

4.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
4.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
4.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
4.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

4.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

4.5 QA Manager: The QA Manager is responsible for

4.5.1 Approving deviations and non-conformances
4.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
4.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

5) Interferences

5.1 Temperature readings are taken immediately at the time of collection. Once a sample is stored for transportation back to the laboratory, field sample temperature is no longer obtainable. Interferences are not relevant as long as the sample is handled appropriately, which would include protecting the sample from prolonged exposure to direct sunlight in summer months and exposure to cold wind in winter months, while the reading is being obtained.

6) Safety

6.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

6.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses

6.3 This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Equipment Calibration	**Hazards**	**Preventative Measures**
Calibration at elevated operating temperature	Burns	Wear heat resistant gloves. Avoid contact with heat source.
Thermometer handling – excessive heating	Bursting glassware and shrapnel	Do not heat above intended range of use. Wear cut resistant gloves and eye protection if working with device past range of use.
Thermometer handling – broken glassware	Lacerations	Wear cut resistant gloves when handling broken glassware.

6.4 Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Samples are collected and temperatures are read immediately (within 15 minutes) upon collection. Once a sample has been stored in a cooler with other samples, temperature (at time of sampling) cannot be determined.

7.2 Collect a sufficient aliquot of sample to immerse the end of the electrode.

8) Apparatus and Equipment

8.1 Field Services uses a variety of field meters that measure temperature. pH meters, dissolved oxygen meters and conductivity meters have temperature probes included within the electrode of the individual instrument. Only meters that have been calibrated by the QA Department and have a valid correction factor listed on the back of the individual meter can be used for temperature in the field. Refer to SOP HN-Field-007, for meter information.

8.2 Glass bulb thermometer, capable of reading within the desired range of use.

8.3 The device shall be able to distinguish temperature readings of 0.1°C or less.

8.4 Sample Collection vessel, minimum volume of 50 ml.

9) Standards, Reagents, and Consumable Materials

9.1 None

10) Calibration

10.1 Refer to SOP HN-EQ-002, Thermometer Calibration for specific calibration procedures.

10.2 The thermometer calibration utilized for field determinations must be traceable to N.I.S.T.

10.3 Electronic temperature devices must be calibrated quarterly. Glass bulb thermometers must be calibrated annually.

11) Procedure

11.1 An aliquot of sample shall be collected for temperature determination. Ensure there is sufficient sample to immerse the temperature device to the appropriate level. Make the determination within 15 minutes of collection.

11.2 Record the date and time of the temperature reading.

11.3 Place the temperature probe directly into the sample. Wait briefly for stabilization. Read results directly from device.

11.4 Use the correction factor listed on the device to obtain the accurate temperature. (See Sec. 13.1.1 for calculation.)

11.5 Record the temperature in the designated logbook.

12) Quality Assurance/Quality Control Requirements

12.1 A valid calibration is the only requirement for the performance of this method.

13) Data Reduction, Reporting, Documentation, and Records

13.1 Correction Factor

13.1.1 A correction factor is applied at (or near) the corresponding temperature reading for that particular device. For example, A 0 ˚C correction at 10 ˚C means that a 0 ˚C correction is applied at (or near) a 10 ˚C reading in order to record the true temperature, which would be 10 ˚C. A +1 ˚C correction at 10 ˚C means that a +1 ˚C correction is applied at (or near) the 10 ˚C reading in order to record the true temperature, which would be 11 ˚C. A -2 ˚C correction at 10 ˚C means that a -2 ˚C correction is applied at (or near) the 10 ˚C graduation in order to record the true temperature, which would be 8 ˚C.

13.2 All data is stored both electronically and hard copy for no less than 10 years.

13.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

13.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

13.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

14) Method Performance

14.1 The device shall be able to distinguish temperature readings of 0.1˚C or less.

14.2 Temperature measurement devices shall not exceed a correction factor of +/- 3˚C.

15) Pollution Prevention

15.1 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

16) Waste Management

16.1 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP HN-SAF-001, Waste Disposal Procedures, must be followed.

17) Corrective Actions for Out-of-Control Data

17.1 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

18) Contingencies for Handling Out-of-Control or Unacceptable Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory records for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Training

19.1 Demonstration of Capability (DOC): an analyst may be deemed qualified for this SOP based on prior experience and a demonstrated understanding of the procedure. SOP review documentation will serve as the employee's qualification statement. This procedure will only require recertification if the procedure is modified due to regulatory changes.

20) Summary of Changes

Table 20.1 - Summary of Revision Changes

Revision Number	Effective Date	Document Editor	Description of Changes
0.0	09.01.2017	C. Stoike	New SOP

21) References and Related Documents

21.1 "Standard Methods for the Examination of Water and Wastewater", Method 2550 B, 2010.

21.2 SOP HN-Field-007, pH for Field Determination, (current version).

21.3 SOP HN-EQ-002, Thermometer Calibration, (current version).

21.4 ALS Environmental Quality Assurance Manual, (current version).

22) Attachments/Appendices

22.1 None

TURBIDITY

SM 2130B-11

SOPID: HN-WC-035 Rev. Number: R05 Effective Date: 03/01/2021

Approved By: _____ Date: 3/6/21
Department Supervisor

Approved By: _____ Date: 3/1/2021
Technical Director

Prepared By: _____ Date: 03/01/21
QA Manager

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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TURBIDITY

1) Scope and Applicability

1.1 This procedure is used in the determination of the Nephelometric Turbidity Units (NTU) of water samples by the use of a Hach Model 2100P ratio Nephelometric turbidity meter.

1.2 This procedure is based upon and compliant with SM 2130B-11.

2) Summary of Procedure

2.1 The determination of turbidity is through a ratio of Nephelometric signal at 90° scatter light to transmitted light.

2.2 This method is applicable for a range of 0.01 – 800 NTU

2.3 Method detection limits (MDL) and practical quantitation limits (PQL) are documented in the applicable LIMS test code

3) Definitions

3.1 DI: De-ionized reagent water meeting purity characteristics of ASTM Type II or better

3.2 Laboratory Control Sample (LCS): A clean, analyte-free matrix spiked with compound(s) representative of the target analyte(s) and carried throughout the complete preparation/analytical procedure

3.3 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes and/or proportions as in sample processing. The method blank is carried throughout the complete sample preparation/analytical procedure

3.4 Matrix: The component or substrate (e.g. surface water, groundwater, or soil) containing the analyte of interest

3.5 Sample Duplicate: A duplicate field sample treated in the same manner as the sample

3.6 Turbidity: A measure of the degree to which water loses transparency due to the presence of suspended particulate matter

3.7 Gelex™ Secondary Standards: Particulate suspensions similar to the formazin primary standards in light scattering characteristics

3.8 StablCal Stabilized Formazin Standards: A series of standards purchased from Hach comprised of the formazin standard, which is the primary standard for turbidity.

4) Health and Safety Warnings

4.1 Lab Safety

4.1.1 Due to various hazards in the laboratory, safety glasses, disposable gloves, and laboratory coats or aprons must be worn when working with unknown samples. In addition, heavy-duty gloves and a face shield are recommended when dealing with toxic, caustic, and/or flammable chemicals.

4.1.2 The toxicity or carcinogenicity of each reagent used has not been precisely defined. However, each chemical used must be treated as a potential health hazard and exposure reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.2 Waste Disposal

4.2.1 Procedures for sample disposal are documented in SOP HN-SAF-001, *Waste Disposal Procedures*.
4.2.2 Samples must be disposed according to Federal, State, and local regulations.

4.3 Pollution Prevention

4.3.1 The quantities of chemicals purchased, when possible, must be based on the expected usage during its shelf life.
4.3.2 Standards and reagents must be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 N/A

6) Interferences

6.1 Interference due to condensation on the sample cell from cold samples in a warm environment is a chief concern. Allow samples to warm to room temperature before reading.

6.2 Glass sample cells can be easily scratched. Applying silicone oil to the outside of the sample cell can mask minor imperfections.

6.3 Extremely turbid samples may cause a negative interference where the measured turbidity is lower than the actual turbidity.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities – This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst – It is the responsibility of the analyst(s) to:

7.2.1 Read, understand, and follow this SOP as written.
7.2.2 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).
7.2.3 Complete the required demonstration of proficiency before performing this procedure without supervision.
7.2.4 Create and populate a data entry batch in LIMS for review by the Supervisor.

7.3 Section Supervisor – It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
7.3.2 Ensure analysts have completed the required demonstration of proficiency before performing this procedure without supervision.
7.3.3 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.4 QA Manager – It is the responsibility of the QA Manager to:

7.4.1 Ensure this method and SOP are followed as written through internal and system audits

7.5 Project Manager – It is the responsibility of the Project Manager to:

7.5.1 Ensure that all contractual requirements for a client requesting this procedure are understood prior to initiating this procedure for a given set of samples.

8) Sample Collection, Handling, and Preservation

8.1 Samples are collected in glass or plastic containers. Containers are purchased by the laboratory and meet all EPA specifications

8.2 Preserve samples with refrigeration at 4 ± 2°C from the time of collection until analysis

8.3 Analysis must be completed within 48 hours of sample collection

9) Equipment and Supplies

9.1 Hach 2100P Portable Turbidimeter

9.2 StablCal Formazin Standards – purchased from Hach in concentrations of <0.1 NTU, 20 NTU, 100 NTU and 800 NTU

9.3 Glass sample cells – purchased from Hach

9.4 Silicone oil – purchased from Hach

9.5 Velvet wiping cloth – purchased from Hach

9.6 Various class A volumetric flasks

10) Standards and Reagents

10.1 DI water meeting purity characteristics for ASTM Type II or better.

10.2 StablCal Stabilized Formazin Standards

10.2.1 These standards are purchased from Hach.
10.2.2 The standards are purchased in the following concentrations: <0.1 NTU, 20 NTU, 100 NTU, and 800 NTU.
10.2.3 Do not dilute any of these standards.

10.3 StablCal Standard - <1.0 NTU

10.3.1 Wash the vial and cap with liquid detergent and water.
10.3.2 Immediately follow with at least 10 rinses with DI water.
10.3.3 Do not allow the inside of the vial to become dry.
10.3.4 Do not invert this standard before using it!
10.3.5 Immediately rinse the vial and cap 3 times with the <1.0 NTU StablCal standard.
10.3.6 Fill the vial to the index band with the standard and immediately cap the vial.
10.3.7 Wipe the outside of the vial with a kimwipe.
10.3.8 Label the vial with the standard value.
10.3.9 Store away from light and at 20-25°C.
10.3.10 This standard will remain stable for up to 1 year.

10.4 StablCal Standard – 20 NTU

10.4.1 Wash the vial and cap with liquid detergent and water.
10.4.2 Immediately follow with at least 10 rinses with DI water.
10.4.3 Do not allow the inside of the vial to become dry.
10.4.4 Gently invert the purchased standard a minimum of 20 times to completely mix the standard.

10.4.5 Immediately rinse the vial and cap 3 times with the 20 NTU StablCal standard.
10.4.6 Fill the vial to the index band with the standard and immediately cap the vial.
10.4.7 Wipe the outside of the vial with a kimwipe.
10.4.8 Label the vial with the standard value.
10.4.9 Store away from light and at 20-25°C.
10.4.10 This standard will remain stable for up to 1 year.

10.5 StablCal Standard – 100 NTU

10.5.1 Wash the vial and cap with liquid detergent and water
10.5.2 Immediately follow with at least 10 rinses with DI water
10.5.3 Do not allow the inside of the vial to become dry
10.5.4 Gently invert the purchased standard a minimum of 20 times to completely mix
10.5.5 Immediately rinse the vial and cap 3 times with the 100 NTU StablCal standard
10.5.6 Fill the vial to the index mark with the standard and immediately cap the vial
10.5.7 Wipe the outside of the vial with a kimwipe
10.5.8 Label the vial with the standard value
10.5.9 Store away from light and at 20-25°C
10.5.10 This standard will remain stable for up to 1 year

10.6 StablCal Standard – 800 NTU

10.6.1 Wash the vial and cap with liquid detergent and water.
10.6.2 Immediately follow with at least 10 rinses with DI water.
10.6.3 Do not allow the inside of the vial to become dry.
10.6.4 Gently invert the purchased standard a minimum of 20 times to completely mix
10.6.5 Immediately rinse the vial and cap 3 times with the 800 NTU StablCal standard
10.6.6 Fill the vial to the index mark with the standard and immediately cap the vial
10.6.7 Wipe the outside of the vial with a kimwipe
10.6.8 Label the vial with the standard value
10.6.9 Store away from light and at 20-25°C
10.6.10 This standard will remain stable for up to 1 year

11) Method Calibration

11.1 Prepare the calibration standards for use.

11.1.1 Gently invert the vials of standards (<1.0 NTU, 20 NTU, 100 NTU and 800 NTU) approximately 5 times to re-suspend the particles in suspension.
11.1.2 Allow the standards to sit undisturbed for 1-2 minutes.

11.2 Utilizing the prepared standards from Section 11.1, calibrate the turbidimeter.

11.2.1 Press the I/O button to turn the instrument on.
11.2.2 Apply a small bead of silicone oil to the outside of the sample cell and wipe with the velvet cloth to ensure a uniform layer on the sample cell.
11.2.3 Insert the sample cell into the sample compartment with the orientation mark of the sample cell aligned with the orientation mark on the instrument and close the lid.
11.2.4 Press the CAL button – note that the CAL and S0 icons will appear on the screen with the 0 flashing.

11.2.4.1 NOTE: Do not use a blank of DI water as the lowest calibration point

if using the <1.0 NTU standard.

11.2.4.2 NOTE: If using standards other than those purchased from Hach, the source water used in the dilution of those standards must be used for the lowest calibration point.

11.2.5 Press READ – The instrument will count down calibration time (60 to 0), store the NTU reading, and automatically switch to S1 with the 1 flashing.

11.2.6 Prepare the next standard (20 NTU – the instrument will prompt user for the 20 NTU standard), insert the standard into the turbidimeter and press READ. Again the meter will count down, store the reading and switch to S2.

11.2.7 Insert the 100 NTU and 800 NTU standards respectively, repeating the steps above.

11.2.8 After reading the last standard, press CAL. The instrument will store the calibration until the next calibration is run.

11.2.8.1 If any errors occurred during calibration, an error message will appear after the CAL is pressed for the final time.

11.2.8.2 If E1 or E2 appears, check the standard preparation and repeat the calibration if necessary.

11.2.8.3 If CAL? appears, an error may have occurred during calibration. If CAL? appears and is flashing, the instrument has reverted to using the default calibration.

12) Sample Preparation/Analysis

12.1 Allow samples to come to room temperature before running them.

12.2 Sample cells are prepared as previously discussed – rinse the sample cell with sample and then apply a small bead of silicone oil to the outside of the cell. Wipe the cell with the velvet cloth to ensure a uniform layer on the sample cell.

12.3 QC Samples

12.3.1 Method Blank

12.3.1.1 Process DI water as the sample matrix

12.3.1.2 A method blank must be processed with each batch of 20 or less samples

12.3.2 LCS/LCS2

12.3.2.1 Check 1 (LCS) = the 20 NTU check standard or alternatively, a purchased low value Gelex™ secondary check standard.

12.3.2.2 Check 2 (LCS2) = the 100 NTU check standard or alternatively, a higher value Gelex™ secondary check standard.

12.3.2.3 A LCS1/LCS2 must be processed with each batch of 20 or less samples

12.3.3 Duplicate sample

12.3.3.1 A sample duplicate must be analyzed with each batch of 10 or less samples.

12.4 Place the sample cell in the cell compartment and close the lid.

12.5 Press RANGE until AUTORNG is displayed.

12.6 Record the meter reading when the lamp icon turns off and a stable reading is displayed (the reading will flash until it is done)

12.7 Repeat the process for each sample. If the meter should happen to turn off at any point during the reading of samples, simply press the I/O button to turn the meter back on (there is no need to recalibrate).

13) Troubleshooting

13.1 N/A

14) Data Acquisition

14.1 All data must be recorded in the Turbidity logbook

14.2 The sample concentration is read directly from the turbidimeter in NTU (Nephelometric Turbidity Units)

14.3 With extremely turbid waters, dilution may be necessary. Account for the dilution factor by multiplying the result from the meter by the dilution factor

14.4 A peer analyst must review all data prior to entering into LIMS. The reviewer must initial and date each batch following review.

15) Calculation, and Data Reduction Requirements

15.1 LIMS calculates the percent recovery for various QC samples (LCS) according to the following calculations:

15.1.1 % Recovery, %R (for standards and LCS)

$$\%R = \frac{SSR}{SA} \times 100$$

Where:

SSR = spiked sample result (mg/L or mg/kg)
SA = spike amount added (mg/L or mg/kg)

15.1.2 % RPD (for duplicate samples, precision or replication evaluation)

$$\%\ RPD = \frac{[SR1 - SR2]}{½\ (SR1 + SR2)} \times 100$$

Where:

SR1 = sample result for replicate 1
SR2 = sample result for replicate 2

16) Quality Control, Data Assessment and Corrective Action

16.1 Initial Calibration

16.1.1 Frequency

16.1.1.1 An initial calibration must be completed once every 3 months or per the manufacturer's recommendations.

16.1.2 Criteria

16.1.2.1 The initial calibration curve must be verified with a mid-level second source standard.

16.1.3 Corrective Action

16.1.3.1 All samples associated with a failed initial calibration or second source verification must be re-processed.
16.1.3.2 If samples cannot be re-processed, all associated results must be flagged and reported as unusable.

16.2 Continuing Calibration Verification (CCV)

16.2.1 Frequency

16.2.1.1 Calibration verification must be completed prior to analysis, after every 10 samples, and at the end of the analytical run.

16.2.2 Criteria

16.2.2.1 The calibration verification concentration must fall near or below the mid-point of the calibration curve.
16.2.2.2 The CCV must meet accuracy performance criteria as outlined in the applicable LIMS test code.
16.2.2.3 The CCV and the LCS may be used interchangeably, but must meet the most stringent recovery criteria.

16.2.3 Corrective Action

16.2.3.1 If the CCV falls outside of the acceptable criteria, perform any needed corrective action and re-calibrate the instrument.
16.2.3.2 All samples since the last acceptable CCV must be re-analyzed. (If the CCV fails high and all associated samples are non-detect, the results may be reported.)

16.3 Method Blank

16.3.1 Frequency

16.3.1.1 A method blank must be processed with each batch of 20 or fewer samples.

16.3.2 Criteria

16.3.2.1 < ½ the PQL, or
16.3.2.2 < 5% of the sample concentration, or
16.3.2.3 < 5% of the regulatory limit.

16.3.3 Corrective Action

16.3.3.1 All samples associated with a failed method blank must be re-processed.
16.3.3.2 If the samples cannot be re-processed, all associated analytical results must be flagged and narrated as to possible bias.

16.4 Laboratory Control Sample (LCS)

16.4.1 Frequency

16.4.1.1 A LCS1/LCS2 must be processed with each analytical batch of 20 or fewer samples.

16.4.2 Criteria

16.4.2.1 Must meet accuracy performance criteria as outlined in the applicable LIMS test code.

16.4.3 Corrective Action

16.4.3.1 All samples associated with a failed LCS must be re-processed.
16.4.3.2 If samples cannot be re-processed, all associated samples must be flagged and narrated as to possible bias.

16.5 Duplicate Sample

16.5.1 Frequency

16.5.1.1 A sample duplicate must be analyzed on a frequency of one duplicate for every 10 samples.

16.5.2 Criteria

16.5.2.1 Must meet precision performance criteria as outlined in the applicable LIMS test code.

16.5.3 Corrective Action

16.5.3.1 All samples associated with duplicate precision failure must be re-processed.
16.5.3.2 If samples cannot be re-processed, all associated samples must be flagged and narrated as to possible bias.

17) Data Records Management

17.1 Logbooks must be maintained for a period of no less than 10 years

17.2 Electronic records must be maintained for a period of no less than 10 years.

18) Quality Assurance and Quality Control

18.1 The QA Staff must conduct periodic audits to evaluate compliance with standard operating procedures.

19) Contingencies for Handling Out of Control Data

19.1 When method required QC failures occur, the source of the QC failure must be determined, corrected, and sample re-analysis carried out when possible.

19.2 When sample analysis cannot be repeated due to limitations on sample availability, the reporting of data associated with failed QC data must be appropriately flagged and narrated to define the error effect upon the data quality.

19.3 All analysts must report sufficient comments in LIMS so that project management can sufficiently narrate failed QC and ensure data qualifiers (flags) are properly assigned to the reported data.

19.4 Non-conformances must be documented on the associated analytical data checklist.

19.5 If the non-conformances are indicative of systemic or procedural errors, a corrective action must be documented and issued via the NC/CA database and submitted to the QA Staff for further evaluation.

20) Method Performance

20.1 N/A

21) Summary of Changes

Table 21.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R02	7/1/12	CES	Formatting
R03	12/15/14	CES	Formatting
R04	10/15/17	LC	Removed cover page graphics.
R04	10/15/17	LC	Update method reference.
R05	03/01/21	CES	Formatting

22) References and Related Documents

22.1 Standard Methods for the Examination of Water and Wastewater, Online Edition, Method 2130B, 2011.

22.2 ALS Environmental Quality Assurance Manual, Revision (most current).

22.3 Portable Turbidimeter, Model 2100P, Instrument and Procedure Manual, Hach Company

APPENDIX 2
EXAMPLE FIELD DATA SHEET AND CALIBRATION RECORD

Project Information

Client: U. S. Steel

Name: State-Only Environmentally Beneficial Project, Lake Michigan's Indiana Shoreline Sampling

Number: 1690023671

Event Information

Date:

Weather:

Air Temperature:

Field Team:

Probe/Meter and Analytical Method: HACH Turbidometer Model 2100P; Thermo Scientific Orion Star A121

Detection Limits: pH: -2.00 to 16.00 Temperature: 23 to 221 ºF Turbidity: 0 to 1,000 FNU

Field Calibration Form Completed? Yes / No

Location: BDXX
Time:
pH:
Temperature:
Turbidity:

Location: MCXX
Time:
pH:
Temperature:
Turbidity:

Location: BDMZ
Time:
pH:
Temperature:
Turbidity:

Location: AWGL / AWGB
Time:
pH:
Temperature:
Turbidity:

Location: KMXX
Time:
pH:
Temperature:
Turbidity:

Location: AMOG
Time:
pH:
Temperature:
Turbidity:

Location: IDBW
Time:
pH:
Temperature:
Turbidity:

Field Duplicate Location:
Time:
pH:
Temperature:
Turbidity:

Location Codes:

BDXX	Burns Ditch
BDMZ	Burns Ditch / Lake Michigan Mixing Zone
KMXX	Kemil Beach
IDBW	Indiana Dune Beach – Western Area
MCXX	Michigan City
	Vicinity of American Water Intake – Gary
AWGL	Lakeside Pump House (Winter Sampling)
AWGB	Breakwall (Summer Sampling)
AMOG	Vicinity of American Water Intake – Ogden

pH: field duplicates = 5% of all measurements

RAMBOLL

Calibration Record

Instrument Type:	**Orion Star A121 pH Portable Meter**
Manufacturer:	**Thermo Scientific**
Instrument Serial No.:	
Calibration Equipment: (make & model)	
Calibration Standard Expiration Date:	
Additional Comments & Notes:	

Monitoring and sampling instruments will be calibrated per 2021 Quality Assurance Project Plan instructions.

Keep 1 copy on-site for the project duration. Place copies in the Project File for retention.

Location Code	Date & Time Calibrated	pH 7	pH 10	Performed By (initial)
BDXX				
BDMZ				
KMXX				
IDBW				
MCXX				
AWGL / AWGB				
AMOG				

Per IDEM (2015) Technical Guidance for the Office of Water Quality External Data Framework for Tier 3 use:

- Field instrumentation must be calibrated once per sampling location per event.
- Thermometers must be calibrated annually.

Location Codes:

BDXX	Burns Ditch
BDMZ	Burns Ditch / Lake Michigan Mixing Zone
KMXX	Kemil Beach
IDBW	Indiana Dune Beach – Western Area
MCXX	Michigan City
	Vicinity of American Water Intake – Gary
AWGL	Lakeside Pump House (Winter Sampling)
AWGB	Breakwall (Summer Sampling)
AMOG	Vicinity of American Water Intake – Ogden

RAMBOLL

Calibration Record

Instrument Type:	**Portable Turbidimeter Model 2100P ISO**
Manufacturer:	HACH
Instrument Serial No.:	
Calibration Equipment: (make & model)	
Calibration Standard Expiration Date:	
Additional Comments & Notes:	

Monitoring and sampling instruments will be calibrated per 2021 Quality Assurance Project Plan instructions.

Keep 1 copy on-site for the project duration. Place copies in the Project File for retention.

Location Code	Date & Time Calibrated	Calibrated to:	Initial Reading	Adjusted Reading	Performed By (initial)
BDXX					
BDMZ					
KMXX					
IDBW					
MCXX					
AWGL / AWGB					
AMOG					

Per IDEM (2015) Technical Guidance for the Office of Water Quality External Data Framework for Tier 3 use:

- Field instrumentation must be calibrated once per sampling location per event.
- Thermometers must be calibrated annually.

Location Codes:

BDXX	Burns Ditch
BDMZ	Burns Ditch / Lake Michigan Mixing Zone
KMXX	Kemil Beach
IDBW	Indiana Dune Beach – Western Area
MCXX	Michigan City
	Vicinity of American Water Intake – Gary
AWGL	Lakeside Pump House (Winter Sampling)
AWGB	Breakwall (Summer Sampling)
AMOG	Vicinity of American Water Intake – Ogden

APPENDIX 3
FIELD INSTRUMENT MANUALS

CAT. NO. 44740-88

PORTABLE TURBIDIMETER

Model 2100P ISO

Instrument and Procedure Manual

4/08 3ed

TABLE OF CONTENTS

TABLE OF CONTENTS, continued

CERTIFICATION

Hach Company certifies this instrument was tested thoroughly, inspected and found to meet its published specifications when it was shipped from the factory.

The Model 2100P ISO Portable Turbidimeter has been tested and is certified as indicated to the following instrumentation standards:

Product Safety

External Power Supplies Only:
115 V ac Supply, UL Listed & CSA Certified, or
230 V ac Supply, CE Marked per 73/23/EEC, VDE Listed

EMI Immunity:

Instrument Tested with external 230 V, 50 Hz Power Supply:
Per **89/336/EEC** EMC: **EN 61326:1998** (Electrical Equipment for measurement, control and laboratory use—EMC requirements) Supporting test records by Hach Company, certified compliance by Hach Company.

Standards include:
IEC 1000-4-2:1995 (EN 61000-4-2:1995) Electro-Static Discharge Immunity (Criteria B)

IEC 1000-4-3:1995 (EN 61000-4-3:1996) Radiated RF Electro-Magnetic Field Immunity (Criteria B)

IEC 1000-4-4:1995 (EN 61000-4-5:1995) Electrical Fast Transients/Burst (Criteria B)

IEC 1000-4-5:1995 (EN 61000-4-5:1995) Surge (Criteria B)

IEC 1000-4-6:1996 (EN 61000-4-6:1996) Conducted Disturbances Induced by RF Fields (Criteria A)

IEC 1000-4-11:1994 (EN 61000-4-11:1994) Voltage Dip/Short Interruptions (Criteria B)

Emissions

Instrument Tested with the external 230 V, 50 Hz Power Supply:
Per **89/336/EEC** EMC: **EN 61326:1998** (Electrical Equipment for measurement, control and laboratory use—EMC requirements) Class “B” emission limits. Supporting test records by Hewlett Packard, Fort Collins, Colorado Hardware Test Center (A2LA # 0905-01) certified compliance by Hach Company.

CERTIFICATION, continued

Standards include:
EN 61000-3-2 Harmonic Disturbances Caused by Electrical Equipment

EN 61000-3-3 Voltage Fluctuation (Flicker) Disturbances Caused by Electrical Equipment

Additional Emissions Standard/s include:
EN 55011 (CISPR 11) Class “B” emission limits

Canadian Interference-causing Equipment Regulation, IECS-003: Class “A” emission limits. Supporting test records by Hewlett Packard, Fort Collins, Colorado Hardware Test Center (A2LA # 0905-01), certified compliance by Hach Company.

This Class A digital apparatus meets all requirements of the Canadian Interference-causing Equipment Regulations.

Cet appareil numérique de la classe A respecte toutes les exigences du Règlement sur le matériel brouilleur du Canada.

FCC Part 15: Supporting test records by Hewlett Packard, Fort Collins, Colorado Hardware Test Center (A2LA # 0905-01), certified compliance by Hach Company.

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

(1) this device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.

Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be

required to correct the interference at his own expense. The following techniques of reducing the interference problems are applied easily.

1. Disconnect the external power supply or remove the batteries from the 2100P ISO Portable Turbidimeter to determine whether it is the source of the interference.
2. Move the 2100P ISO Portable Turbidimeter and its power supply away from the device receiving the interference.
3. Move the device receiving the interference to a different location.
4. Try combinations of the above.

SAFETY PRECAUTIONS

Please read this entire manual before unpacking, setting up, or operating this instrument. Pay particular attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that which is specified in this manual.

Use of Hazard Information

If multiple hazards exist, this manual will use the signal word (Danger, Caution, Note) corresponding to the greatest hazard.

DANGER
Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION
Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTE
Information that requires special emphasis.

Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

 This symbol, if noted on the instrument, references the instruction manual for operational and/or safety information.

SPECIFICATIONS

Specifications are subject to change without notice.
Operating specifications applicable at 25 °C unless noted.

Measurement Method: Ratio Nephelometric signal (90°) scatter light ratio to transmitted light

Range: 0–1000 FNU with automatic decimal point placement or manual range selection of 0–9.99, 0–99.9 and 0–1000 FNU

Accuracy: ± 2% of reading plus stray light from 0–1000 FNU

Resolution: 0.01 FNU on lowest range

Repeatability: ± 1% of reading or 0.01 FNU, whichever is greater (with Gelex standards)

Response Time: 6 seconds for full step change without signal averaging in constant reading mode

Stray Light: <0.04 FNU

Standardization: StablCal® Stabilized Formazin primary standards or Formazin primary standards

Secondary Standards: Gelex® Secondary Standards

Display: Four-digit liquid crystal; 10.16 mm (0.4 in.) high digits with custom icons

Light Source: T860 nm LED lamp

Detectors: Silicon photovoltaic

Signal Averaging: Operator selectable on or off

Sample Cells: (Height X width) 60.0 X 25 mm (2.36 X 1 in.) Borosilicate glass with screw caps, marking band and fill line

Sample Required: 15 mL (0.5 oz)

Storage Temperature: –40 to 60 °C (–40 to 140 °F) (instrument only)

SPECIFICATIONS, continued

Operating Temperature: 0 to 50 °C (32 to 122 °F) (instrument only)

Operating Humidity Range: 0 to 90% RH noncondensing at 30 °C;
0 to 80% RH noncondensing at 40 °C;
0 to 70% RH noncondensing at 50 °C

Power Requirements: Four AA Alkaline cells or optional battery eliminator

Battery Life: Typically 300 tests with signal average mode off; 180 tests with signal average mode on

Battery Eliminator (optional):

For 120 V eliminator: CSA and UL approved for 120 V ac ± 10%, 60 Hz, 6 V at 800 mA dc output

For 230 V eliminator: CE (VDE) approval pending for 230 V ac ± 10%, 50 Hz, 6 V at 900 mA dc output

Enclosure: High impact ABS plastic

Dimensions: 22.2 X 9.5 X 7.9 cm (8.75 X 3.75 X 3.12 in.)

Instrument Weight: 520 kg (1 lb 2.5 oz)

Shipping Weight: 3.1 kg (6 lb 8.5 oz)

OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Das Arbeiten mit chemischen Proben, Standards und Reagenzien ist mit Gefahren verbunden. Es wird dem Benutzer dieser Produkte empfohlen, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

PERICOLO

La manipolazione di campioni, standard e reattivi chimici può essere pericolosa. La preghiamo di prendere conoscenza delle Schede Techniche necessarie legate alla Sicurezza dei Materiali e di abituarsi con tutte le procedure di sicurezza prima di manipolare ogni prodotto chimico.

SECTION 1 DESCRIPTION

1.1 General Description

The Hach Model 2100P ISO Portable Turbidimeter (*Figure 1*) measures turbidity from 0.01 to 1000 FNU in automatic range mode with automatic decimal point placement. The manual range mode measures turbidity in three ranges: 0.01–9.99, 10–99.9, and 100–1000 FNU. The instrument operates on four AA alkaline batteries or with an optional battery eliminator. Rechargeable nickel-cadmium cells may be used, but cannot be recharged in the instrument. The carrying case holds teh instrument, all standard accessories, and the optional battery eliminator.

Figure 1 2100P ISO Turbidimeter and Accessories

Note: *Avoid prolonged exposure to ultraviolet light and sunlight.*

Note: *When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.*

1.2 Accessories

Accessories supplied with the turbidimeter include nine sample cells; three Gelex® Secondary Standards (included with 4474000 only); one sealed vial each of:
<0.1-FNU, 20-FNU, 100-FNU, and 800-FNU StablCal® Stabilized

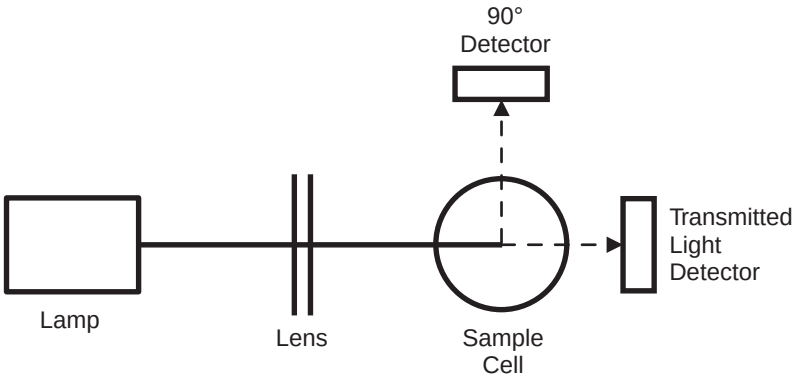
Formazin Standards; 4 AA alkaline batteries; 15 mL of silicone oil; oiling cloth; carrying case; instrument manual; and quick reference card.

1.3 Principle of Operation

The Model 2100P ISO Portable Turbidimeter operates on the nephelometric principle of turbidity measurement. This instrument meets the design criteria specified by ISO 7027.

The ratio optical system* (*Figure 2*) includes an LED lamp, a 90° detector to monitor scattered light, and a transmitted light detector. The microprocessor calculates the ratio of the signals from the 90° and transmitted light detector. This ratio technique corrects for interferences from color and/or light absorbing materials (such as activated carbon) and compensates for fluctuations in lamp intensity, providing long-term calibration stability. The optical design also minimizes stray light, increasing measurement accuracy.

Figure 2 Ratio Optical System



1.4 Preparation for Use

1.4.1 Unpacking

Remove the instrument and accessories from the shipping box and inspect for damage that may have occurred due to rough handling or

* Patent number 4,198,161; other patents pending.

extreme weather conditions. Verify the following are present (*Figure 1*):

- Model 2100P ISO Portable Turbidimeter
- Instrument Manual (with quick reference card)
- Set of StablCal Primary Standards in sealed vials, one each of:
  <0.1 FNU*
  20 FNU
  100 FNU
  800 FNU
- Standardization Kit containing Gelex Secondary Standards (included with 4474000 only), 0-10, 0-100 and 0-1000 ranges plus nine sample cells with caps.
- Silicone Oil, 15-mL (0.5 oz) dropping bottle
- Oiling Cloth
- Carrying Case
- Four AA alkaline batteries

If any of the items are missing or damaged, please contact the Customer Service Department, Hach Company, Loveland, Colorado. The toll-free number in the United States is 800-227-4224. International customers should contact the Hach office or authorized distributor serving your area. Refer to *REPAIR SERVICE* on page *63*. **Please do not return the instrument without prior authorization from Hach.**

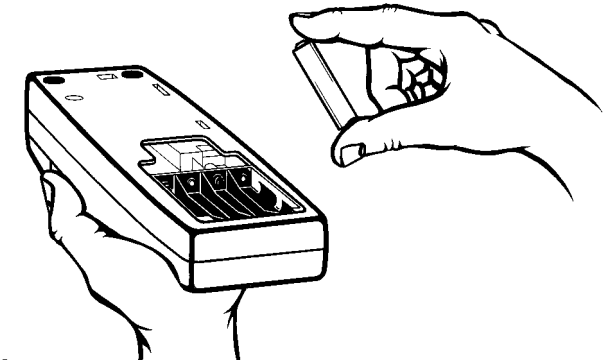
1.4.2 Battery Installation

The instrument is shipped without the batteries installed. Before use, install the four AA alkaline batteries (*Figure 3*) or connect the battery eliminator. For battery operation:

1. Remove the battery compartment cover on the instrument bottom.
2. Install the batteries.
3. Correct battery polarity is shown on the battery holder. Reinstall the battery compartment cover.

* Used in place of the dilution water standard when performing a calibration.

Figure 3 Battery Installation



1.4.3 Using the Battery Eliminator and Rechargeable Batteries

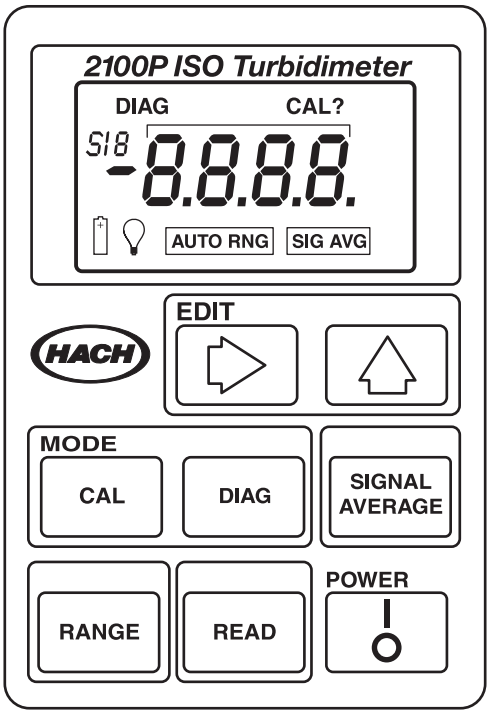
Plug the eliminator jack into the connector on the turbidimeter side. The optional battery eliminator may be used with or without the batteries installed. **The eliminator will not charge batteries.** Rechargeable batteries may be used in the instrument, but must be removed for recharging. See *HOW TO ORDER* on page *62* for ordering information. To prolong battery life, the instrument lamp turns on temporarily when the **READ** key is pressed.

1.4.4 Calibration

The 2100P ISO Portable Turbidimeter is calibrated with Formazin Primary Standard at the factory and **does not require recalibration before use.** See *Section 2.4* on page *22* for calibration instructions.

SECTION 2 OPERATION

2.1 Operational Controls and Indicators



Key	Description
I O	Power key turns instrument on and off. If no keys are pressed for 5.5 minutes, the instrument turns off automatically.
READ	Performs a measurement. To conserve battery power, the lamp turns on only when **READ** is pressed. A reading is displayed about 12 seconds after the key is pressed. After the reading is displayed, the lamp turns off and the reading continues to be displayed. After a measurement is taken, there is a 4-second recovery time before another can be started. If **READ** is pressed during the recovery time, the display will flash but the lamp will not turn on until the recovery time is complete. If no key strokes occur for 5.5 minutes, the meter turns off. Hold this key for continuous reading if the meter is **not** in the Signal Averaging mode. After the initial delay, the reading is updated every 1.2 seconds.

Key	Description
CAL	Performs a calibration or review calibration data. Also terminates a calibration or calibration review and returns to the 2100P ISO measurement mode.
	Edits a flashing digit in the calibration mode, or scrolls through the calibration standards (S0,S1, S2, S3) or diagnostic menu.
	Moves the editing cursor to the digits being edited in the calibration mode or initiate editing of a standard value.
SIGNAL AVERAGE	Turns the signal averaging function on or off.
DIAG	Selects the diagnostic mode.
RANGE	Scrolls between Auto Range, Manual Range, 0.00–9.99 FNU range, 10–99.9 FNU range, and 100–1000 FNU range. The range selection cannot be changed when a reading or calibration is in progress. When the meter is turned on it defaults to the last used range mode and measurement range.

Display Icon	Description
DIAG	Turns on after **DIAG** is pressed. A number displayed under the DIAG icon (1–9) indicates which diagnostic function is active. See Section 5.1 on page 55 for more information on diagnostic codes.
CAL	Turns on after **CAL** is pressed and remains on during the calibration.
CAL?	Appears after calibration if a value entered during calibration is outside an acceptable range. May indicate an operator error or possible instrument malfunction. Flashing **CAL?** indicates the default calibration coefficients are being used (even after a user-calibration has been done) or that no calibration data is currently stored.
S__	Appears during calibration. The S is followed by a number to indicate which standard value is currently being edited or displayed. The flashing number is prompting user for measurement of **S0**, **S1**, **S2** or **S3** to establish a calibration. Steady number identifies which standard value is being displayed.
	Flashes when the battery voltage drops to 4.4 volts indicating to change batteries. At <4.0 volts, the instrument automatically shuts off.

Display Icon	Description
	Appears constantly when the lamp is on and flashes after a reading if a marginal light level reaches the transmitted light detector. When flashing, it indicates the sample may be too turbid (not within measurement range) and needs dilution or the lamp needs replacing.
SIGNAL AVERAGE	Indicates the signal averaging mode is on. The icon turns off if signal averaging is not selected.
AUTO RNG	Indicates instrument is in automatic range mode. The icon turns off when manual range mode is selected.
8888	The 4-digit display is active when the instrument is on (measurements are displayed to three digits). After the **READ** key is pressed **- - - -** is displayed during wait periods.

2.2 Signal Averaging

Signal averaging measures and averages ten measurements while displaying intermediate results. The initial value is displayed after about 11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 22 seconds). Afterwards, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

When signal averaging is off, the instrument takes three measurements, the microprocessor averages them, then displays the average. If the **READ** key is held during measurement, the initial value is displayed in 12 seconds and is updated every 1.2 seconds as long as the **READ** key is held.

When the instrument is turned on, the instrument defaults to the signal averaging mode which was used during the last measurement.

2.3 Restoring the Default Calibration

To restore and use the default calibration, turn the instrument off. Press and hold **DIAG**, then press and release **I/O**. Release **DIAG** when the software version number disappears from the display. (For models with serial number less than 920300000800, **2100** disappears). This clears any user-entered calibration from memory; the 2100P ISO will use the default calibration for measurement. **CAL?** will appear and continue to flash until a user-entered calibration is successfully completed.

For best results, a user-entered calibration should be done every three months.

2.4 Calibration

Calibration of the 2100P ISO Turbidimeter is based on formazin, the primary standard for turbidity. The electronic and optical design provide long-term stability and minimize the need for frequent calibration. The two-detector ratioing system compensates for most fluctuations in lamp output. A formazin recalibration should be performed at least once every three months, more often if experience dictates. When calibration is necessary, use a primary standard such as StablCal™ Stabilized Standards or formazin standards.

For Hach turbidimeters, use only StablCal® Stabilized Formazin or formazin standards. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.

DO NOT calibrate with Gelex® Secondary Standards. Gelex standards are designed for instrument verification, not calibration.

2.4.1 StablCal Stabilized Formazin Standards*

Most consistent results will be achieved with the use of StablCal® Stabilized Formazin Standards for calibration. Refer to *Section 2.4.1.2* and *Section 2.4.1.3* for information on preparing the standards for use.

Hach StablCal Stabilized Formazin in 20-, 100-, and 800-FNU values is packaged in sets for calibration of the 2100P ISO Turbidimeter. (See *OPTIONAL ACCESSORIES AND REAGENTS* on page *59*.)

2.4.1.1 Storing and Handling StablCal Stabilized Standards

For optimum results when using StablCal Stabilized Standards, adhere to the following recommendations:

- Do not transfer the standard to another container for storage.

* StablCal Stabilized Formazin is cited as a primary standard in Hach Method 8195, an acceptable version of USEPA Method 180.1.

- Do not return standard from the sample cell back into the its original container. Standard contamination will result.
- Store standards between 0 and 25 °C.
- For long-term storage, refrigeration at 5 °C is recommended. Do not store above 25 °C.
- Allow the standard to acclimate to ambient instrument conditions before use (not to exceed 40 °C).
- Store away from direct sunlight. Store vials in their respective kit or shipping box with the cover in place.

2.4.1.2 Preparing Bulk StablCal Stabilized Standards

Bulk standards that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*.

When using <0.1-FNU StablCal Standards, do not shake or invert.

1. Shake the standard vigorously for 2–3 minutes to suspend particles.
2. Allow the standard to stand undisturbed for 5 minutes.
3. Gently invert the bottle of StablCal 5 to 7 times.
4. Prepare the sample cell for measurement using traditional preparation techniques. This usually consists of oiling the sample cell (see Section 3.1.1 on page 35) and marking the cell to maintain the same orientation in the sample cell compartment (see Section 3.2.2 on page 39). This step will eliminate any optical variations in the sample cell.
5. Rinse the sample cell at least once with the standard and discard the rinse.
6. Immediately fill the sample cell with the standard. Cap the sample cell and let it stand for one minute. The standard is now ready for use in the calibration procedure, Section 2.4.3 on page 27.

2.4.1.3 Preparing StablCal Stabilized Standards in Sealed Vials

Sealed vials that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*.

When using <0.1-FNU* StablCal Standards do not shake or invert.

1. Shake the standard vigorously for 2–3 minutes to suspend particles.
2. Allow the standard to stand undisturbed for 5 minutes.
3. Gently invert the vial of StablCal 5 to 7 times.
4. Prepare the vial for measurement using traditional preparation techniques. This usually consists of oiling the vial (see Section 3.1.1 on page 35) and marking the vial to maintain the same orientation in the sample cell compartment (see Section 3.2.2 on page 39). This step will eliminate any optical variations in the sample vial.
5. Let the vial stand for one minute. The standard is now ready for use in the calibration procedure, Section 2.4.3 on page 27.

2.4.2 Formazin Primary Standards

Perform the procedure in Section 2.4.2.1 on page 24 to prepare a 4000-FNU standard, or order a 4000-FNU stock solution (Cat. No. 2461-49). Prepare the dilutions from the 4000-FNU stock solution by following the instructions in Section 2.4.2.4 on page 26.

2.4.2.1 Preparing Formazin Stock Solution

Dilute formazin standard solutions from a 4000 FNU stock solution equivalent to Hach Cat. No. 2461-49. The prepared stock solution is stable for up to one year when properly prepared. An alternative to purchasing the 4000 FNU stock solution is preparing a stock solution as follows:

1. Dissolve 5.000 grams of reagent grade hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) in 400 mL of distilled water.
2. Dissolve 50.000 grams of pure hexamethylenetetramine in 400 mL of distilled water.

3. Pour the two solutions into a 1000-mL volumetric flask and dilute to the mark with distilled water.

4. Let the solution stand for 48 hours at 25 °C (77 °F) to develop the 4000-FNU stock suspension. The standing temperature is critical for correct formation of formazin polymers.

5. Mix the 4000 FNU suspension for at least ten minutes before use. Then it can be diluted with distilled or demineralized water to achieve a solution of the desired FNU value.

Instead of diluting a formazin stock solution, StablCal Stabilized Formazin Standards may be used. (See *OPTIONAL ACCESSORIES AND REAGENTS* on page *59*.)

2.4.2.2 Correcting for Turbidity of Dilution Water

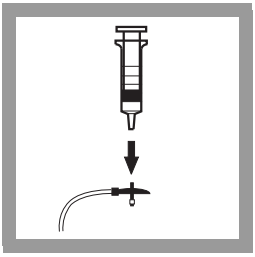
The 2100P ISO Turbidimeter automatically compensates for turbidity contributed by dilution water when calculating the true value of the lowest formazin standard. Use high quality distilled or deionized water less than 0.5 FNU. The instrument will display “E 1” after calibration if the dilution water turbidity is greater than 0.5 FNU. In this case, prepare the water as directed below.

The value of the dilution water can be arbitrarily forced to zero using the calibration procedure (see step 4 on page 31). This is not recommended for most applications and, if done, should be done only if the dilution water turbidity is less than 0.2 FNU.

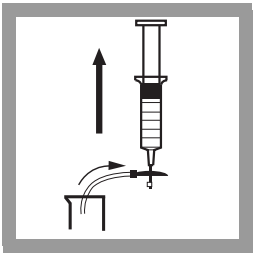
2.4.2.3 Preparing Dilution Water

Note: *Use the same dilution water for all dilutions and the sample blank.*

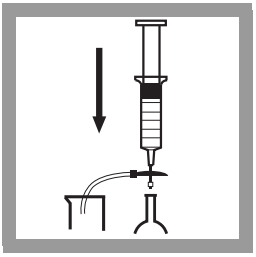
Collect at least 1000 mL of high quality dilution water (distilled or deionized water). The 2100P ISO Turbidimeter, as received from the factory, is precalibrated and may be used to check the dilution water turbidity. If the turbidity is greater than 0.5 FNU, filter the water with the Sample Filtration and Degassing Kit (Cat. No. 43975-10) or equivalent. When measuring low range turbidity, clean all glassware with 1:1 hydrochloric acid and rinse several times with dilution water. If the glassware is not used immediately, use stoppers to prevent contamination from small particles.



1. Attach the syringe to the 3-way valve by gently twisting the square end into the syringe tip. Attach the connector, tubing and a 0.2 micron filter (clear part faces syringe) as shown. Be sure the connections are tight.



2. Fill a beaker or container with the water to be filtered. Insert the tubing into the container. Slowly draw the 50 mL of water into the syringe by pulling up on the syringe plunger.



3. Slowly push on the plunger to force the water through the filter and into a graduated cylinder or volumetric flask. Repeat Steps 1–3 until the desired amount of water is obtained.

Note: *As the filter clogs, it gets more difficult to push water through it. Discard the filter and attach a new filter.*

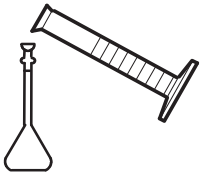
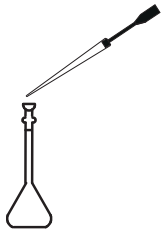
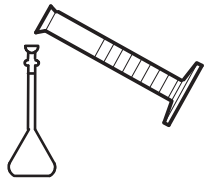
2.4.2.4 Preparing Formazin Dilutions (Factory recommended)

Use 20, 100, and 800 FNU formazin standards for calibrating the 2100P ISO Turbidimeter. Dilutions with other FNU values can be prepared and used (see Section 2.4.3.1 on page 30). If problems occur when using alternate solutions, use the dilutions specified here.

Prepare all formazin dilutions immediately before use and discard after calibration. The 4000 FNU solution is stable for up to a year, but dilutions deteriorate more rapidly. Use the same high quality water (turbidity <0.5 FNU) for the dilutions and the blank.

Preparing the 20, 100 and 800 FNU standards

Table 1 Formazin Standard Preparation

	Step 1	Step 2	Step 3
Standards			
20 FNU	Add 100 mL of dilution water to a clean **200-mL** Class A volumetric flask.	With a TenSette1 pipet, add 1.00 mL of well-mixed 4000 FNU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
100 FNU	Add 100 mL of dilution water to a clean **200-mL** Class A volumetric flask.	With a TenSette pipet, add 5.00 mL of well-mixed 4000 FNU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
800 FNU	Add 50 mL of dilution water to a clean **100-mL** Class A volumetric flask.	With a TenSette pipet, add 20.00 mL of well-mixed 4000 FNU Formazin stock solution to the 100-mL flask.	Dilute to the mark with dilution water. Stopper and mix.

1 A Class A volumetric pipet may be used in place of a TenSette Pipet.

2.4.3 Calibrating the Turbidimeter

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ**, **I/O**, **UP**, and **RIGHT** keys function. Signal averaging and range mode must be selected before entering the calibration mode.

- If "E1" or "E2" are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. Press **DIAG** to cancel the error message (E1 or E2). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If "CAL?" is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.
- To review a calibration, press **CAL** and then **UP** to view the calibration standard values. As long as **READ** is never pressed and CAL is not flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

Note: *For accuracy, use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the cell so the orientation mark placed on the cell during the matching procedure is correctly aligned. (See Section 3.2.3 on page 41 for matching sample cells).*

Use only StablCal® Stabilized Formazin or formazin standards for calibration. Turbidimeter performance is not guaranteed if calibrated with co-polymer styrene divinylbenzene beads or other suspensions. **DO NOT** calibrate with Gelex® Secondary Standards.

1. Rinse a clean sample cell several times with dilution water. Fill the cell to the line (about 15 mL) with dilution water or use StablCal <0.1 FNU standard.

   Note: *The same dilution water used for preparing the standards must be used in this step.*

2. Insert the sample cell in the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid. Press **I/O**.

3. Choose signal average mode option (on or off) before pressing **CAL**. The **SIGNAL AVERAGE** key is not functional in calibration mode.

4. Press **CAL**. The CAL and S0 icons will appear (the 0 will flash). The 4-digit display will show the value of the S0 standard for the previous calibration. If the blank value was forced to 0.0, the display will be blank. Press the **RIGHT** key to get a numerical display.

5. Press **READ**. The meter will count from 60 to 0, (67 to 0 if signal average is on), read the blank, and use it to calculate a correction factor for the 20 FNU standard measurement. If the dilution water is ³ 0.5 FNU, E1 will appear when the calibration is calculated (See Section 2.4.2.3 on page 25 for more dilution water information). The display will automatically increment to the next standard.

6. Remove the sample cell from the cell compartment.

The turbidity of the dilution water can be "forced" to zero by pressing the **RIGHT** key rather than reading the dilution water. The display will show "S0 FNU". Press the **UP** key to continue with the next standard.

7. The display will show "S1" (with the 1 flashing) and 20 FNU or the value of the S1 standard for the previous calibration. If the value is incorrect, edit using the arrow keys.

8. Fill a clean sample cell to the line with well mixed 20 FNU StablCal Standard or 20 FNU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

9. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard.

10. Remove the sample cell from the cell compartment.

11. The display will show "S2" (with the 2 flashing) and "100 FNU" or the value of the S2 standard for the previous calibration. If the value is incorrect, edit using the arrow keys.

12. Fill a clean sample cell to the line with well mixed 100 FNU StablCal Standard or 100 FNU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

13. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity, and store the value. The display will automatically increment to the next standard.

14. Remove the sample cell from the cell compartment.

15. The display will show “S3” (with the 3 flashing) and “800 FNU” or the value of the S3 standard for the previous calibration. If the value is incorrect, edit using the arrow keys.

16. Fill a clean sample cell to the line with well mixed 800 FNU StablCal Standard or 800 FNU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

17. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will increment back to the S0 display. Remove the sample cell from the cell compartment.

18. Press **CAL** to accept the calibration. The instrument will return to measurement mode automatically.

Note: *If calibration errors occurred during calibration, error messages will appear after CAL is pressed (See Section 5.4 on page 57).*

2.4.3.1 Preparing User-selected Formazin Dilutions

The formazin solutions should span the entire range of the instrument. Hach recommends preparing three standards:

- 10–30 FNU
- 90–110 FNU
- 700–900 FNU

The standards must have a difference of at least 60 FNU.
In addition, a blank made from the dilution water should be prepared.

Prepare the formazin standard solutions from the well mixed 4000 FNU stock solution as specified in Section 2.4.2.4 on page 26 and dilution water as specified in *Section 2.4.2.2* and Section 2.4.2.3 on page 25. Make the standards **immediately** before use and discard them after calibration is done.

2.4.3.2 Calibrating with User-selected Standards

For accuracy, use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the sample cell with the same orientation.

1. Fill a clean sample cell to the line (about 15 mL) with dilution water.

Note: *The same dilution water used for preparing the standards must be used in this step.*

2. Insert the sample cell into the cell compartment and close the lid. Press **I/O**.

3. Choose signal average mode option (on or off) before pressing **CAL**. The **SIGNAL AVERAGE** key is not functional in calibration mode.

4. Press **CAL**. The CAL and S0 icons will appear (the "0" will flash). The 4-digit display will show the value of the S0 standard for the previous calibration.

5. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the blank and use it to calculate a correction factor for the lowest standard. If the dilution water is ³ 0.5 FNU, "E1" will appear (see Section 2.4.2.3 on page 26 for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

6. Thoroughly mix the 10 to 30 FNU range standard, then fill a clean sample cell to the line with the standard. Insert the sample cell into the cell compartment.

7. The display will show the S1 icon (with the "1" flashing) and 20 FNU or the value of the S1 standard for the previous calibration.

8. Edit the standard concentration using the arrow keys.

9. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard.

10. Remove the sample cell from the cell compartment.

11. Thoroughly mix the 90 to 110 FNU standard, then fill a clean sample cell to the line with the standard.

12. Insert the cell into the cell compartment.

13. The display will show the S2 icon (with the 2 flashing) and 100 FNU or the value of the S2 standard for the previous calibration.

14. Edit the standard concentration using the arrow keys.

15. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Remove the sample cell from the cell compartment.

16. Thoroughly mix the 700 to 900 FNU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.

17. The display will show the S3 icon (with the "3" flashing) and 800 FNU or the value of the S3 standard for the previous calibration.

18. Edit the standard concentration using the arrow keys.

19. Press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The instrument will increment back to S0. Remove the sample cell from the cell compartment.

20. Press **CAL**. The instrument will store the new calibration data and return the instrument to the measurement mode.

Note: *If calibration errors occurred during calibration, error messages will appear after CAL is pressed.*

2.4.4 Using Gelex® Secondary Turbidity Standards

Note: *Store Gelex standards at room temperature. Do not allow to freeze or exceed 50 °C.*

Gelex Secondary Standards (included with 4474000 only) are particulate suspensions similar to formazin primary standards in light scattering characteristics. FNU values on the Gelex standards indicate the range for which they should be used. Due to minor variations in glass and individual instrument optical systems, the true value of the Gelex standards must be determined against formazin in the same instrument they will be used with for later calibration checks.

2.4.4.1 Assigning Values to Gelex Standards

Note: *Correct cell orientation is essential to obtain accurate Gelex values. Always orient the cell so the diamond mark aligns with the orientation mark on the instrument.*

1. Calibrate the instrument with formazin.
2. Select automatic range mode using the **RANGE** key.
3. Thoroughly clean the outside of the Gelex vials and apply a thin coat of silicone oil.
4. Place the 0–10 FNU Gelex standard in the cell compartment so the diamond on the vial aligns with the orientation mark on the instrument. Close the lid.
5. Press **READ**. Record the value, remove the vial from the instrument and mark the value on the band near the top of the vial.
6. Repeat steps 3–5 for other Gelex standards. Reassign values tot he Gelex standards each time the meter is calibrated with formazin.

2.4.4.2 Routine Calibration Check With Gelex® Standards

The 2100P ISO Turbidimeter does not require standardization before every measurement as some turbidimeters do. Periodically, as experience dictates, check the instrument calibration using the appropriate Gelex Secondary Standard. Be sure the Gelex standards are aligned correctly when inserting them (diamond aligns with orientation mark). If the reading is not within 5% of the previously established value, recalibrate the instrument with StablCal Stabilized Formazin Primary Standard or formazin primary standard (Section 2.4.3 on page 27).

DO NOT calibrate with Gelex Secondary Standards. Gelex standards are designed for instrument verification, not calibration.

SECTION 3 TURBIDITY MEASUREMENT

3.1 Turbidity Measurement

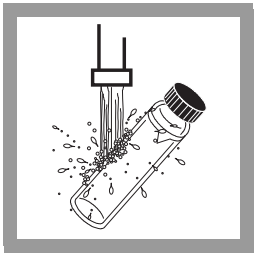
Measurements may be made with the signal average mode on or off and in manual or automatic range selection mode (Section 2.2 on page 21).

With signal average mode off, the final value is displayed after approximately 12 seconds.

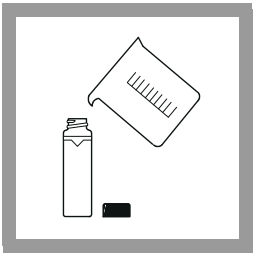
Accurate turbidity measurement depends on good measurement technique by the analyst, such as using clean sample cells in good condition and removing air bubbles (degassing). Refer to Section 3.2 on page 38 for a detailed discussion of measurement techniques.

Place the instrument on a flat, sturdy surface during measurements.

3.1.1 Turbidity Measurement Procedure

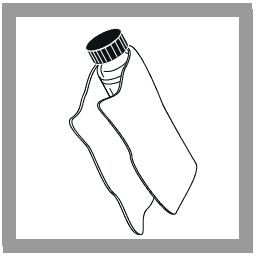


1. Thoroughly clean the sample cell.

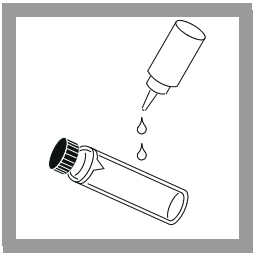


2. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. (See Section 3.2 on page 38 for more information about collecting a representative sample).

Cap the cell.

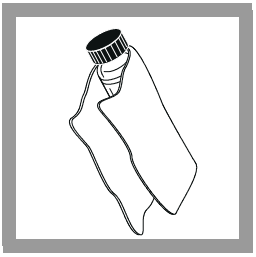


3. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.



4. Apply a thin film of silicone oil. Wipe with a soft, lint-free cloth to obtain an even film over the entire surface.

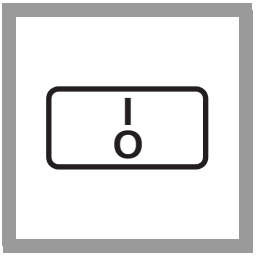
Note: *Applying a thin coat of silicone oil will mask minor imperfections and scratches which may contribute to turbidity or stray light. Use silicone oil equivalent to Hach Cat. No. 1269-36, which has the same refractive index as glass.*



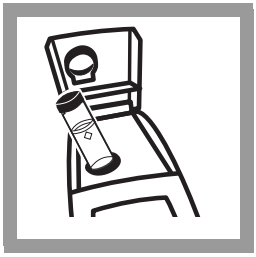
5. Wipe off the excess so that only a thin coat of oil is left. Excess oil may retain dirt and contaminate the instrument cell compartment.

The cell should appear nearly dry with little or no visible oil.

Note: *Soft, lint-free cloth (velvet) works well for oiling. Store the oiling cloth with the sample cells and keep it free of dirt. After a few applications of oil, the cloth will contain enough residual oil that simply wiping the cell with the oiled cloth will provide a sufficient oil coat on the sample cell. Periodically, add a small amount of oil to the sample cell surface to replenish the oil in the cloth.*

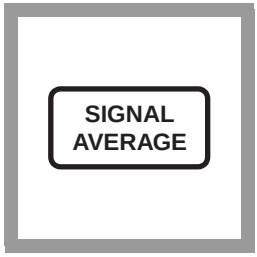


6. Press: **I/O**.

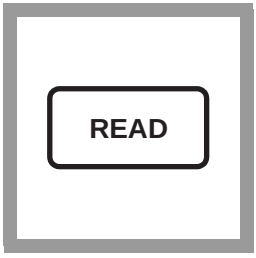


7. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment.
Close the lid.

8. Press: **RANGE**. Select mannual or automatic range mode.



9. Press: **SIGNAL AVERAGE**.



10. Press: **READ**

The display will show "- - - -", then the turbidity in FNU. Record the turbidity after the lamp symbol turns off.

Note: *The instrument defaults to the last operating mode selected. If automatic range mode and signal averaging were used on the previous measurements, these options will automatically be selected for subsequent samples.*

3.1.2 Measurement Notes

- Always cap the sample cell to prevent spillage of sample into the instrument.
- When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.
- Always close the sample compartment lid during measurement and storage.
- Always use clean sample cells in good condition. Dirty, scratched, or damaged cells can cause inaccurate readings.
- Do not leave a sample cell in the cell compartment for extended periods of time. This may compress the spring in the cell holder.
- Remove sample cell and batteries from instrument if the instrument is stored for extended time period (more than a month).
- Avoid operating in direct sunlight.
- Make certain cold samples do not “fog” the sample cell.
- Avoid settling of sample prior to measurement.
- Keep sample compartment lid closed to prevent dust and dirt from entering.

3.2 Measurement Techniques

Proper measurement techniques are important to minimize the effects of instrument variation, stray light, and air bubbles. Regardless of the instrument used, measurements are more accurate, precise, and repeatable if the analyst pays close attention to proper measurement techniques.

Measure samples immediately to prevent temperature changes and settling. Avoid sample dilution when possible. Particles suspended in the original sample may dissolve or otherwise change characteristics when the sample temperature changes or when the sample is diluted, resulting in a non-representative sample measurement.

3.2.1 Cleaning Sample Cells

Cells must be extremely clean and free from significant scratches. The glass used to make cells is easily scratched – manufacturing cells free of minor scratches and other imperfections is difficult. However, minor imperfections are effectively masked by applying silicone oil as outlined in Section 3.1.1 on page 35.

Clean the inside and outside of the cells by washing with laboratory detergent. Follow with multiple rinses of distilled or deionized water. Allow cells to air dry. Handle cells only by the top to minimize dirt, scratches, and fingerprints in the light path.

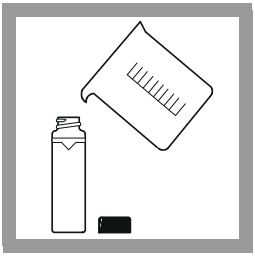
3.2.2 Orienting Sample Cells

Note: *When orienting and matching cells, it may be more efficient to use the continuous reading mode. The instrument performs continuous readings if the* **READ** *key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1.2 seconds. The instrument cannot be used in continuous read mode if the signal averaging mode is on.*

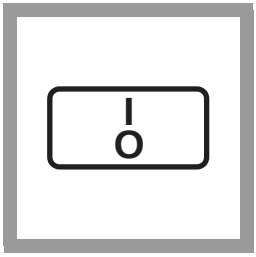
Precise measurements for very low turbidity samples require using a single cell for all measurements or optically matching the cells. Using one cell provides the best precision and repeatability. When one cell is used, an orientation mark (other than the factory-placed diamond) can be placed on the cell so that it is inserted into the instrument with the same orientation each time.

3.2.2.1 Orienting a Single Cell

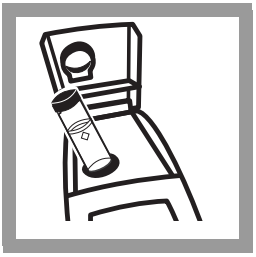
When using a single cell, make an index or orientation mark on the cell as follows:



1. Fill the clean sample cell to the line with high quality water (< 0.5 FNU). Cap and wipe with lint-free cloth. Apply silicone oil. See Section 2.4.2.2 on page 25 for more information about high quality water.



2. Press: **I/O**.



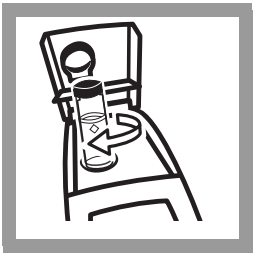
3. Insert the sample cell into the sample compartment. Close the cover.

4. Press: **READ**

Record the cell position in the compartment and the displayed reading.

Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*

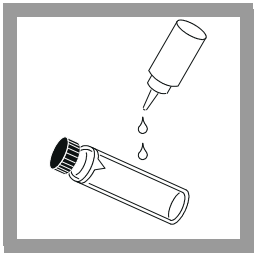
5. Remove the cell, rotate it slightly and reinsert it into the cell compartment. Close the cover, then press **READ**. Record the cell position and the displayed reading.



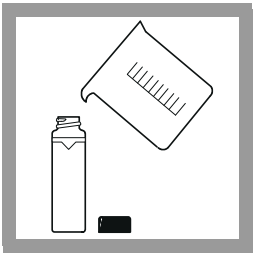
6. Repeat *step 5* until the lowest reading is displayed. Place an orientation mark on the cell marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading. When using the cell, always place it in the instrument so the orientation mark aligns with the raised mark on the instrument.

3.2.3 Matching Multiple Sample Cells

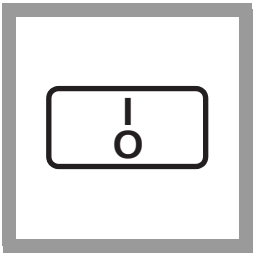
Precise measurements of very low turbidity samples require the cells be optically matched or a single cell be used for all measurements. If more than one cell is used, follow this procedure to match (index) the cells:



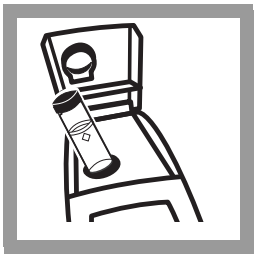
1. Clean and oil the sample cells as instructed in Section 3.1.1 on page 35.



2. Fill the clean sample cells to the line with the same sample.



3. Press: **I/O** to turn the instrument on.



4. Insert the **first** sample cell into the sample compartment and close the cover.

5. Press: **READ**

Record the cell position in the compartment and the displayed reading. Place an orientation mark on the cell marking band.

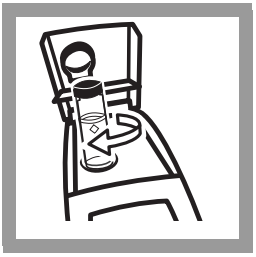
Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*

6. Insert the **second** sample cell into the cell compartment and close the cover.

7. Press: **READ**

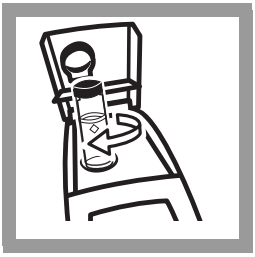
Record the cell position in the compartment and the displayed reading.

8. Remove the cell, rotate it slightly and reinsert into the cell compartment. Close the cover, then press **READ** again. Record the cell position and the displayed reading.



9. Repeat *step 8* until the value displayed for the second cell is within 0.01 FNU (or 1%) of the value obtained for the first cell. Place an orientation mark on the second cell marking band so it is consistently inserted in this position.

Note: *Due to variability in glass, it may not be possible to match all cells.*



10. Repeat *step 6* through *step 9* if matching other sample cells.

3.2.4 Removing Bubbles (Degassing)

Before measurement, removing air and other trapped gasses from the sample is strongly recommended, even if bubbles are not visible. Four degassing methods are commonly used:

- Applying a partial vacuum.
- Adding a surfactant.
- Using an ultrasonic bath.
- Heating the sample.

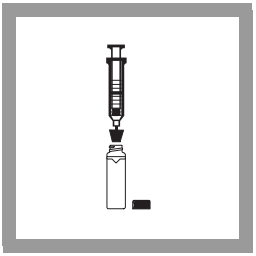
In some cases, more than one method may be necessary for effective bubble removal. For example, use of both a surfactant and ultrasonic bath may be necessary for some severe conditions. Use care with these techniques. If misused, sample turbidity can be altered.

Letting the sample stand for a period of time to remove air bubbles is not recommended. Particulates that cause turbidity may settle and the sample temperature may change, resulting in measurements not representative of the original turbidity.

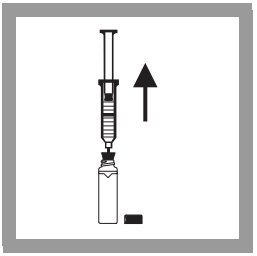
3.2.4.1 Application of a Partial Vacuum

The vacuum lowers the atmospheric pressure, allowing trapped bubbles to escape into the air above the sample. Vacuum works well with non-viscous samples (such as water) that do not contain volatile components. Applying vacuum to viscous, volatile samples (paint resins) may cause the volatile components to come out of solution and aggravate the bubble problem.

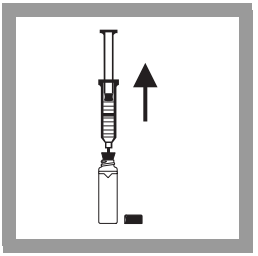
To apply a vacuum, use a sample degassing kit equivalent to Cat No. 43975-00 (Degassing Kit) or 43975-10 (Degassing and Filtration Kit). These kits contain a syringe and rubber stopper for vacuum degassing. An electric or hand-operated pump equivalent to Cat. No. 14283-00 may also be used.



1. Fill a sample cell to the mark with sample. Insert a #2 single-hole rubber stopper and syringe into the cell. If using a pump, insert a piece of glass tubing into the stopper.



2. **Slowly** apply the vacuum by carefully pulling the plunger upward, then holding it. If using a hand or electric pump, connect the tubing to the vacuum pump with vacuum hose. Apply vacuum until visible gas bubbles disappear.



3. **Slowly** release the vacuum. Remove the vacuum apparatus and cap the cell.

3.2.4.2 Adding a Surfactant

Surfactants should be limited to severe problems when other degassing methods are ineffective. This technique is very effective when the water is super-saturated with air. Surfactants change the surface tension of the water, which releases trapped gases.

1. Add one drop of a surfactant (Triton X-100, Cat. No. 14096-32 or equivalent) to the sample cell.

2. Add sample.

Note: *Any turbidity contributed by surfactant addition is negligible.*

3. Mix the sample gently but thoroughly, and analyze as soon as possible after adding the surfactant.

4. Rinse the sample cells thoroughly between samples to prevent surfactant accumulation.

3.2.4.3 Using an Ultrasonic Bath

Ultrasonic baths effectively remove gas bubbles from most samples, especially viscous liquids. However, the ultrasonic waves which cause degassing may also alter the characteristics of the particles causing the turbidity. Turbidity depends on the size, shape, composition and

refractive index of the suspended particles. Excessive ultrasound application may alter particle size and shape, thus changing sample turbidity. In some cases, ultrasound may impede air bubble removal by fracturing the bubbles, making degassing more difficult.

The basic procedure for an ultrasonic bath is:

1. Fill a clean sample cell to the line with sample. Leave uncapped.
2. Immerse the cell ($^1/_2$ to $^2/_3$ immersed) in an ultrasonic bath and allow it to stand until visible bubbles are expelled.
3. Remove the cell and replace the cap. Thoroughly dry the cell. Apply silicone oil as directed in Section 3.1.1 on page 35.

The time necessary to expel bubbles may vary from a few seconds to a minute or more. To avoid excessive application of ultrasound:

1. Apply ultrasound until all visible bubbles are absent.
2. Measure the sample turbidity.
3. Apply ultrasound for a short time period and again measure turbidity.
4. Continue for several repetitions, noting the treatment time and turbidity readings.

If turbidity begins to increase instead of decrease, the ultrasound waves have probably started to alter the suspended particles. Note the time it takes for this to occur and record it as the maximum time limit for ultrasonic treatment.

3.2.4.4 Application of Heat

Gentle heating may be helpful for degassing some very viscous samples when combined with application of vacuum or ultrasound. Heat may change the characteristics of the suspended particles and cause volatile components to come out of solution. If heat is necessary, heat the sample only until degassing occurs.

1. Prepare a warm water bath and partially immerse the filled sample cell.
2. Use the shortest time necessary for expelling visible bubbles.
3. Cool sample to original sample temperature before taking measurements.

3.2.5 Measuring Overrange Samples

Nephelometric turbidity measurement depends on detection of light scattered from particles suspended in the liquid. If the turbidity is very high, a significant amount of light is blocked or absorbed by the particles and only a small amount of light reaches the detector. The measured turbidity is then lower than the actual turbidity (negative interference). This condition is called “going blind”. The 2100P ISO Turbidimeter minimizes this effect and extends the instrument range. Dilution of highly turbid samples should be avoided since it may alter the characteristics of the suspended particles and produce erroneous results.

Light-absorbing particles such as activated carbon and highly colored samples may also cause an instrument to “go blind”. Dilution may not correct for these interferences. The 2100P ISO will correct for the presence of light absorbing particles and color.

3.2.6 Condensation (Fogging)

Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so wipe moisture off of the sample cell before measurement. If fogging recurs, let the sample warm slightly at room temperature, or immerse in a warm bath for a short period. After warming, mix the sample thoroughly before measurement. Warming samples can alter sample turbidity, so avoid warming before measurement if possible.

3.2.7 Representative Sampling

A representative sample accurately reflects the condition of the water source from which the sample was taken. To ensure a representative sample, gently but thoroughly mix every sample before aliquots are taken. Do not allow the sample to settle.

When sampling from a tap in a distribution system or treatment plant, allow the water to run for at least five minutes before sampling. When sampling from a stream, reservoir, clarifier, or storage tank, collect at least one liter (l quart) and thoroughly mix before measurement. If the water source is not uniform, it may be necessary to sample several locations at varying depths and combine the samples into a single, well-mixed composite sample before measurement.

MAINTENANCE

Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the maintenance tasks described in this portion of the manual.

Certains des chapitres suivants de ce mode d'emploi contiennent des informations sous la forme d'avertissements, messages de prudence et notes qui demandent une attention particulière. Lire et suivre ces instructions attentivement pour éviter les risques de blessures des personnes et de détoriation de l'appareil. Les tâches d'entretien décrites dans cette partie du mode d'emploi doivent être seulement effectuées par le personnel qualifié pour le faire.

Algunos de los capítulos del manual que presentamos contienen muy importante información en forma de alertas, notas y precauciones a tomar. Lea y siga cuidadosamente estas instrucciones a fin de evitar accidentes personales y daños al instrumento. Las tareas de mantenimiento descritas en la presente sección deberán ser efectuadas únicamente por personas debidamente cualificadas.

Einige der folgenden Abschnitte dieses Handbuchs enthalten Informationen in Form von Warnungen, Vorsichtsmaßnahmen oder Anmerkungen, die besonders beachtet werden müssen. Lesen und befolgen Sie diese Instruktionen aufmerksam, um Verletzungen von Personen oder Schäden am Gerät zu vyyyermeiden. In diesem Abschnitt beschriebene Wartungsaufgaben dürfen nur von qualifiziertem Personal durchgeführt werden.

Algumas das seguintes secções do manual contêm informações em forma de advertências, precauções e notas que requerem especial atenção. Leia e siga atentamente as presentes instruções para evitar ferimentos pessoais e não danificar o instrumento. As tarefas de manutenção descritas nesta parte do manual só poderão ser executadas por pessoal qualificado.

SECTION 4 MAINTENANCE

4.1 Cleaning

Keep the turbidimeter and accessories as clean as possible and store the instrument in the carrying case when not in use. Avoid prolonged exposure to sunlight and ultraviolet light. Wipe spills promptly. Wash sample cells with non-abrasive laboratory detergent, rinse with distilled or demineralized water, and air dry. Avoid scratching the cells and wipe all moisture and fingerprints off the cells before inserting them into the instrument. Failure to do so can give inaccurate readings.
See Section 3.2.1 on page 39 for more information about sample cell care.

4.2 Battery Replacement

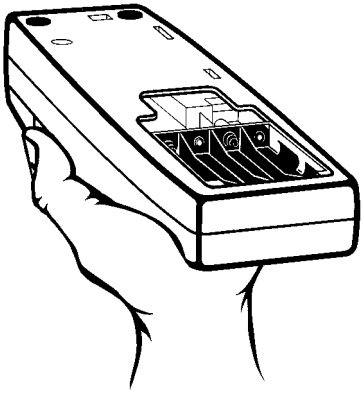
AA alkaline cells typically last for about 300 tests with the signal averaging mode off, about 180 tests if signal averaging is used.
The “battery” icon flashes when battery replacement is needed. Refer to Section 1.4.2 on page 17 for battery installation instructions. If the batteries are changed within 30 seconds, the instrument retains the latest range and signal average selections. If it takes more than 30 seconds, the instrument uses the default settings.

If the instrument will not turn off or on after changing batteries, and the batteries are good, remove the batteries and reinstall them. If the meter still does not function, contact Hach Service or the nearest authorized dealer.

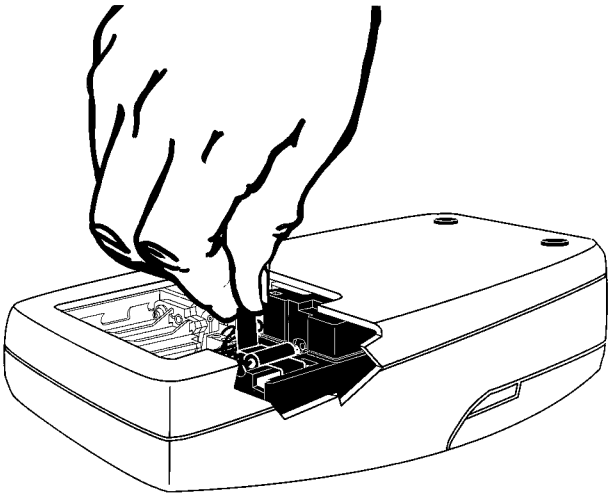
4.3 Lamp Replacement

The procedure below explains lamp installation and electrical connections. Use a small screwdriver to remove and install the lamp leads in the terminal block. The instrument requires calibration after lamp replacement.

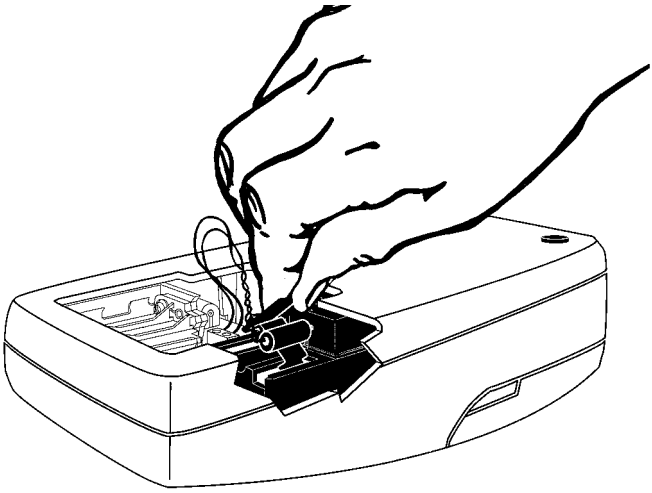
1. Orient the instrument so it is upside down and the top faces away from you. Remove the battery cover and at least one battery.



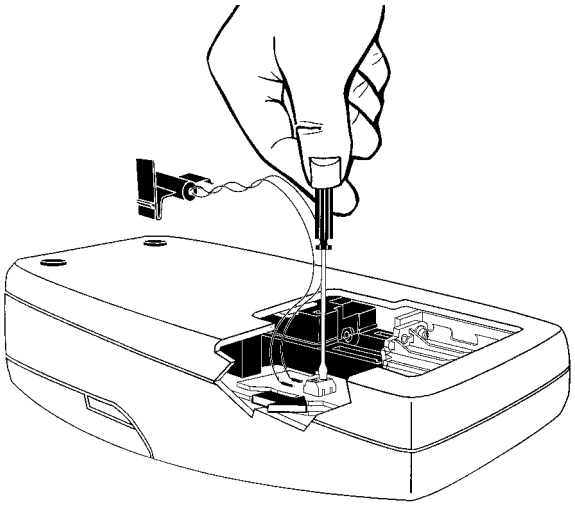
2. Remove the lamp assembly by grasping the tab on the left side of the assembly. Firmly, but gently, slide the assembly towards the rear of the instrument.



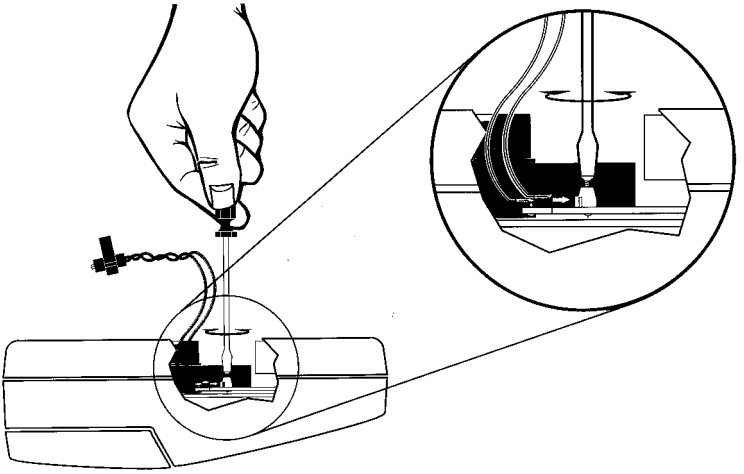
3. Rotate the tab towards the nearest outside edge. The assembly should release and slip out easily.



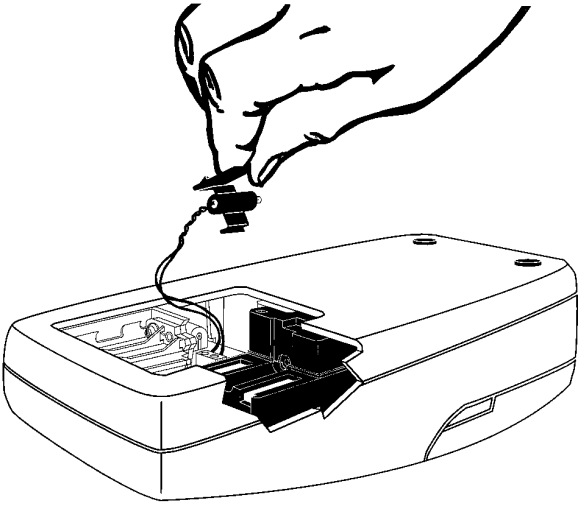
4. Back the terminal block screws **partially** out (1 to 2 turns) and remove the old lamp leads.



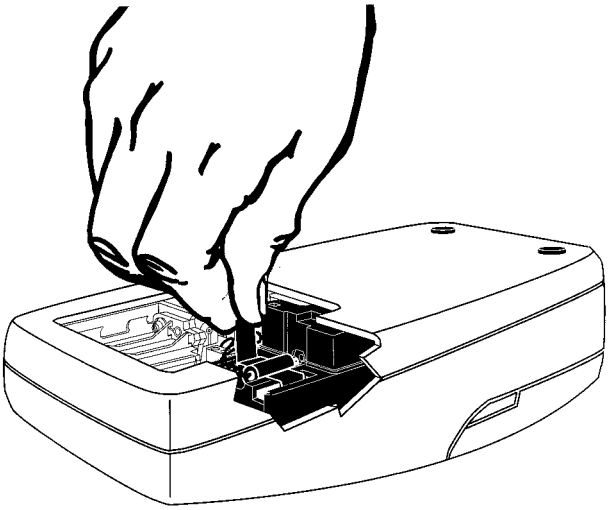
5. Gently bend the wires of the new lamp assembly into an “L” shape so they fit easily into the housing. Insert the leads into the terminal screws and tighten with clockwise turns. Gently tug on the wires to make sure they are connected to the terminal block.



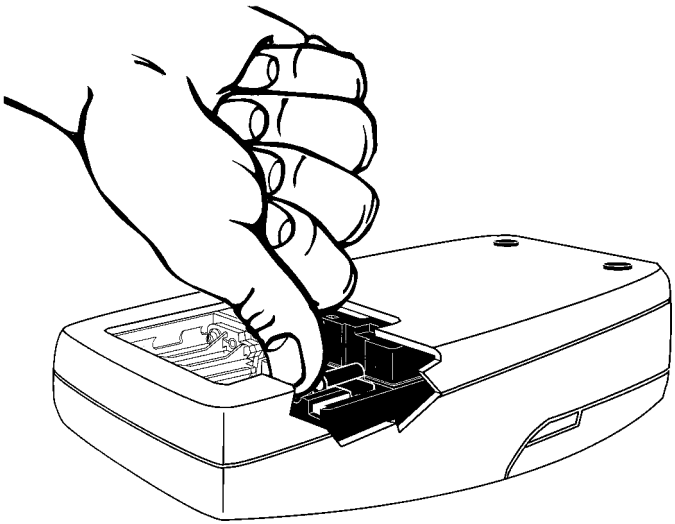
6. Hold the new lamp assembly by the tab with the lamp facing the tope (keyboard) of the instrument. Slide the small catch on the other side of the assembly into the black plastic slot (towards the nearest edge of the instrument).



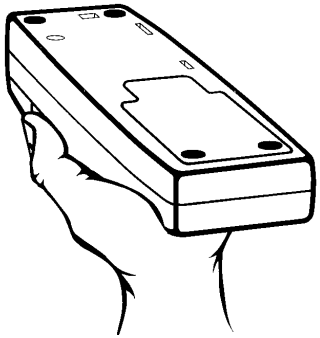
7. Snap the U-shaped bottom of the tab into the slot on the left side of the black plastic that holds the lamp assembly.



8. With your thumb, firmly slide the assembly forward until it stops. Again, push firmly against the tab to make sure the lamp is seated correctly.

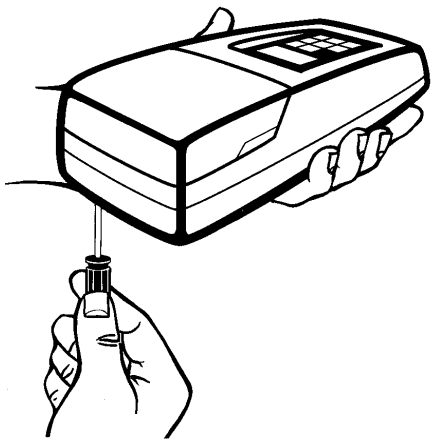


9. Replace the batteries and battery cover.



10. Insert the 800 FNU formazin standard into the sample cell. Press and hold **READ**. Then press **I/O**. Release the **READ** key after the software version number disappears from the display (for models with serial numbers less than 920300000800, **2100** disappears).

11. Adjust the scattered light amplifier output by inserting a small flat-blade screwdriver into the trimpot hole (located on bottom). Adjust the display to read 2.5 ± 0.3 volts (2.0 volts for models that display **2100** when turned on).



12. Press **I/O** to exit gain adjust mode.

13. Perform a formazin calibration according to Section 2.4.2 on page 24 or Section 2.4.2.4 on page 26.

SECTION 5 TROUBLESHOOTING

5.1 Using the Diagnostic Functions Key

Enter the diagnostic mode by pressing the **DIAG** key. Exit this mode at any time by pressing the key again. The diagnostic mode allows access to information about instrument function which may be useful for servicing and troubleshooting.

5.1.1 Basic Diagnostic Codes

Diagnostic Codes

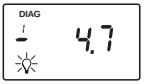
Code	Description
1	Checks the battery voltage with the lamp on, then with the lamp off. This is a dual diagnostic code.
2	Displays calibration coefficient a_o
3	Displays calibration coefficient a_1
4	Displays calibration coefficient b_o
5	Displays calibration coefficient b_1
6	Displays the lamp voltage (about 3 volts)
7	Displays the dark voltage of the transmitted light detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.
8	Displays the high gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.1
9	Displays the low gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.

1 Samples with turbidity >10 FNU may display "- - -" for the lamp-on amplifier voltage.

5.2 The Diagnostic Procedure

1. Fill a clean sample cell to the line with clear water, cap the cell and place it in the cell compartment. Press **READ** and wait until the reading is finished.

2. Press: **DIAG**



The **DIAG** icon will turn on and **1** will be displayed below the icon. The instrument will measure the battery voltage with the lamp off and display the result in volts (V). Then the lamp icon will turn on and the instrument will measure the voltage with the lamp on. The value is briefly displayed before defaulting to the lamp-off reading. To repeat the measurement, press **READ**.

3. To continuously display the lamp-on voltage, press the **RIGHT** key. The lamp icon will flash. Press the **RIGHT** key to turn the lamp icon off (the lamp is not on during this display).

4. Press the **UP** key to scroll through the other diagnostics. Each press of the key increments the digit in the small numerical display below the **DIAG** icon and the result of the diagnostic measurement is then displayed. Each press of **READ** updates the value. For measurements made with the lamp off and again with the lamp on, the measurement with the lamp off is displayed when the diagnostic is entered. To see the second measurement with the lamp on, press the **RIGHT** key (only works with diagnostic codes 1, 7, 8, & 9). The lamp icon will flash and the lamp-on measurement will be displayed in volts. Press the **RIGHT** key to turn the lamp icon off.

Note: *DIAG 8 will display "----" for the lamp-on voltage if a of >10 FNU is placed in the cell compartment.*

5.3 Other Instrument Diagnostics

5.3.1 Display Test

Pressing and holding the **I/O** key turns on all the display icons and elements so you can determine if all the elements and icons are functioning. The display test sequence will cycle as long as the key is held down.

5.4 Error Messages

Error messages indicate sample interferences and/or instrument malfunction.

5.4.1 Flashing Numeric Display

If the highest value in the range selected is flashing in the display, the sample is too turbid (or overrange) for the selected range. In automatic or manual range, **1000** flashes if the sample is over the instrument range. In manual range mode, select the next higher range mode if 9.99 or 99.9 flashes. See Section 3.2.5 on page 46 for measuring overrange samples. The display will stop flashing if a sample within range is inserted and read.

5.4.2 Error Messages

An error message indicates either an instrument failure or an operation cannot be performed. An error message can be cleared by pressing **DIAG** (display will return to previous measurement or calibration value). The meter continues to operate as best it can. If the message occurs during a calibration, calibration can continue. If the error message occurs when a calibration is being calculated, the instrument will discard the new calibration and retain the old calibration. Error messages and corrective actions are listed below.

5.4.3 CAL?

A flashing **CAL?** appears when the instrument is using the default calibration programmed at the factory. It will appear if the analyst has erased the user-entered calibration using the procedure to restore the default calibration or after and E4 error is cleared by pressing **DIAG.** Recalibrate as soon as possible when CAL? appears. CAL? (not flashing) appears when a calibration has questionable validity.

Message1	Probable Cause	Corrective Action
E1	Dilution water is ≥ 0.5 FNU.	Start calibration over with better quality dilution water or use a membrane filter to filter the water before use.
E2	Two standards have the same value or their difference is less than 60 FNU. Not all standards were read during the calibration. Standard 1 is too low (<10 FNU).	Recheck preparation of standards and repeat calibration.
E3	Low light error.	Re-read measurement. Check lamp2 Check for obstructed light path. Dilution may be necessary.
E4	EEPROM malfunction.	Check sum failed. Press **I/O**. If E4 reappears, call Hach service. If CAL? appears, recalibrate.
E5	A/D overrange.	Check for obstructed light path. Call Hach Service.
E6	A/D underrange.	Check for open lid during reading and re-read. Check for obstructed light path. If persists, call Hach Service.
E7	Light Leak.	Close lid before pressing **READ**.
E8	Bad lamp circuit.	Reinsert lamp leads at terminal block-make sure the lead ends are not touching each other.If this fails, call Hach Service.

1 Error messages 4, 5, and 6 may indicate a failure in the internal electronics.
2 Check lamp by inserting a pencil or piece of paper into the cell compartment and pressing **READ**. Light should be visible on the inserted object.

Replacement Parts & Accessories

REPLACEMENT PARTS

Description	Cat. No.
StablCal®* Calibration Set for 2100P ISO,sealed vials, < 0.1 FNU, 20 FNU, 100 FNU, and 800 FNU	26594-05
AA Batteries, 4/pkg	19380-04
Battery Door	46005-00
Carrying Case	46506-00
Gelex® Standards, set (includes standards and 3 sample cells)	24641-05
Instrument Manual	44740-88
Lamp Assembly, LED	44602-00
Oiling Cloth	47076-00
Sample Cells, 1 inch, with cap, 6/pkg	24347-06
Silicone Oil, 15 mL	1269-36

OPTIONAL ACCESSORIES AND REAGENTS

Deionized Water, 3.78 L	272-17
Bath, Ultrasonic, 2.8 L (0.75-gal), w/heater	24895-00
Battery Eliminator, 120 V	46079-00
Battery Eliminator, 230 V	46080-00
Filter, 0.2 micron, 10/pkg	23238-10
Formazin, 4000 FNU, 500 mL	2461-49
Formazin, 4000 FNU,100 mL	2461-42
Hexamethylenetetramine, 500 g	1878-34
Hydrazine Sulfate, 100 g	742-26
NiCad Rechargeable Battery (4 required)	16077-00

* StablCal® is a registered trademark of Hach Company.

OPTIONAL ACCESSORIES AND REAGENTS, continued

Description	Cat. No.
Pipet, TenSette®*, 1-10 mL	19700-10
Pipet Tips, for 1-10 mL TenSette Pipet, 50/pkg	21997-96
Pipet Tips, for 1-10 mL TenSette Pipet, 1000/pkg	21997-28
Pipet, Volumetric, Class A, 1.00 mL	14515-35
Pipet, Volumetric, Class A, 5.00 mL	14515-37
Pump, Vacuum, Hand-Operated	14283-00
Sample Degassing Kit	43975-00
Sample Filtration and Degassing Kit	43975-10
StablCal® Calibration Set for 2100P ISO Turbidimeter	
<0.1, 20, 100, 800 FNU, 500 mL each	26594-00
<0.1, 20, 100, 800 FNU, 100 mL each	26594-10
<0.1, 20, 100, 800 FNU, sealed vials	26594-05
<0.1 FNU** StablCal®Stabilized Formazin Standard, 100 mL	26597-42
20 FNU StablCal® Stabilized Formazin Standard, 100 mL	26601-42
100 FNU StablCal® Stabilized Formazin Standard, 100 mL	26602-42
800 FNU StablCal® Stabilized Formazin Standard, 100 mL	26605-42
Triton-X Solution, 118 mL (4 oz)	14096-32
Volumetric Flask, 100 mL	14574-42
Volumetric Flask, 200 mL	14574-45

* TenSette™ is a Hach Company trademark.

** <0.1 FNU StablCal® Standard is used in place of dilution water standard when performing a calibration.

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

HOW TO ORDER

By Telephone:

6:30 a.m. to 5:00 p.m. MST
Monday through Friday
(800) 227-HACH
(800-227-4224)
By FAX: (970) 669-2932

By Mail:

Hach Company
P.O. Box 389
Loveland, CO 80539-0389
U.S.A.

Ordering information by E-mail: orders@hach.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

Technical and Customer Service (U.S.A. only)

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use. Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224** or E-mail **techhelp@hach.com**.

International Customers

Hach maintains a worldwide network of dealers and distributors. To locate the representative nearest you, send E-mail to **intl@hach. com** or contact:

In Canada, Latin America, Africa, Asia, Pacific Rim:
Telephone: (970) 669-3050; FAX: (970) 669-2932

In Europe, the Middle East, or Mediterranean Africa:
HACH Company, c/o
Dr. Bruno Lange GmbH
Willstätterstr. 11
D-40549 Düsseldorf
Germany
Telephone: +49/[0]211.52.88.0
Fax: +49/[0]211.52.88.231

REPAIR SERVICE

Authorization must be obtained from Hach Company before sending any items for repair. Please contact the HACH Service Center serving your location.

In the United States:
Hach Company
100 Dayton Avenue
Ames, Iowa 50010
(800) 227-4224 (U.S.A. only)
Telephone: (515) 232-2533
FAX: (515) 232-1276

In Canada:
Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba
R3H 0X4
(800) 665-7635 (Canada only)
Telephone: (204) 632-5598
FAX: (204) 694-5134
E-mail: canada@hach.com

In Latin America, the Caribbean, the Far East, the Indian Subcontinent, Africa, Europe, or the Middle East:
Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado, 80539-0389
U.S.A.
Telephone: (970) 669-3050
FAX: (970) 669-2932
E-mail: intl@hach.com

WARRANTY

Hach Company warrants this product to the original purchaser against any defects that are due to faulty material or workmanship for a period of **one year from date of shipment**.

In the event that a defect is discovered during the warranty period, Hach Company agrees that, at its option, it will repair or replace the defective product or refund the purchase price, excluding original shipping and handling charges. Any product repaired or replaced under this warranty will be warranted only for the remainder of the original product warranty period.

This warranty does not apply to consumable products such as chemical reagents; or consumable components of a product, such as, but not limited to, lamps and tubing.

Contact Hach Company or your distributor to initiate warranty support. Products may not be returned without authorization from Hach Company.

Limitations

This warranty does not cover:

- damage caused by acts of God, natural disaster, labor unrest, acts of war (declared or undeclared), terrorism, civil strife or acts of any governmental jurisdiction
- damage caused by misuse, neglect, accident or improper application or installation
- damage caused by any repair or attempted repair not authorized by Hach Company
- any product not used in accordance with the instructions furnished by Hach Company
- freight charges to return merchandise to Hach Company
- freight charges on expedited or express shipment of warranted parts or product
- travel fees associated with on-site warranty repair

This warranty contains the sole express warranty made by Hach Company in connection with its products. All implied warranties, including without limitation, the warranties of merchantability and fitness for a particular purpose, are expressly disclaimed.

Some states within the United States do not allow the disclaimer of implied warranties and if this is true in your state the above limitation may not apply to you. This warranty gives you specific rights, and you may also have other rights that vary from state to state.

This warranty constitutes the final, complete, and exclusive statement of warranty terms and no person is authorized to make any other warranties or representations on behalf of Hach Company.

Limitation of Remedies

The remedies of repair, replacement or refund of purchase price as stated above are the exclusive remedies for the breach of this warranty. On the basis of strict liability or under any other legal theory, in no event shall Hach Company be liable for any incidental or consequential damages of any kind for breach of warranty or negligence.

Thermo Scientific Orion Star A121

Basic pH portable meter

Thermo Scientific™ Orion™ Star A121 pH Portable Meters make it easy and affordable to take simple, accurate pH measurements.

Thermo Scientific Orion Star A121 pH Portable Meters combine simplicity with accuracy. A large LCD displays pH or mV readings along with temperature. Icons provide quick updates on battery life, electrode status and calibration information. Simple button layout and onscreen messages help with calibration and setup menu choices.

Features and Benefits

- Large, informative screen clearly shows the main information, including pH or mV reading, temperature in °C or °F, meter mode, calibration information and battery life
- Don't miss a reading—AUTO-READ™ locks in the stable reading on your screen and ready indicator alerts when continuous readings are stable
- Up to 3 point pH calibration with easy recall of calibration point and slope data
- Automatic recognition of USA/NIST and DIN buffers or manual option for custom buffer values
- Non-volatile memory holds up to 50 data points
- Four AA batteries (included) provide 2000 hours of operation or purchase the universal power adapter (sold separately)
- Perfectly portable, waterproof and protected to take anywhere with a IP67-rated housing
- 3 year meter replacement warranty

Thermo SCIENTIFIC

Thermo Scientific Orion Star A121 pH Portable Meter

Specifications	
pH	**Range:** -2.00 to 16.00 **Resolution:** 0.1, 0.01 **Relative Accuracy:** ±0.01 **pH Calibration Points:** Up to 3
mV	**Range—mV:** ±1600.0 mV **Range—RmV:** ±1999.9 mV **Resolution:** 0.1 mV **Relative Accuracy:** ±0.2 mV or ±0.05 % of reading whichever is greater **Relative mV Calibration:** Yes
Temperature	**Range:** -5 to 105 ºC, 23 to 221 ºF **Resolution:** 0.1 ºC, 0.1 ºF **Relative Accuracy:** ±0.1 ºC, ±0.1 ºF **Temperature Compensation:** Automatic or manual **Temperature Offset Calibration:** Yes
Datalogging	**Number of Points:** 50 **Log Function:** Manual, Automatic with AUTO-READ
Inputs	**pH Electrode:** BNC **ATC Probe:** 8-pin mini-DIN
Power	**AC Adapter:** Optional—universal, 100-240 VAC **Battery Power:** Included—4 AAs **Battery Life:** 2000 hrs

Meter Ordering Information	
Part No.	**Description**
STARA1210	**Orion Star A121 pH Portable Meter** - Four AA batteries (installed) - Printed quick start guide and literature CD
STARA1215	**Orion Star A121 pH Portable Meter Kit** - 9107BNMD Orion Triode gel-filled, epoxy-body pH/ATC electrode - 916099 Orion 60 mL pH buffer and solutions kit - 911110 Orion rinse solution pouches - Protective armor with electrode holder and storage sleeve - Hard-sided field case - Four AA batteries (installed) - Printed quick start guide and literature CD

Accessories Ordering Information	
Part No.	**Description**
STARA-CS	Orion Star A series portable meter hard-sided field case
1210005	Orion Star A series portable meter soft-sided field case
STARA-AR	Orion Star A series portable meter armor with pH electrode holder, conductivity and DO probe holder, stand and adjustable hand-strap
STARA-ESPH	Orion pH electrode holder for portable Star A series meter armor
810017	Orion pH electrode storage sleeve for pH electrode holder
9157BNMD	Orion Triode refillable, epoxy-body pH/ATC electrode
9107WMMD	Orion Triode gel-filled, epoxy-body pH/ATC electrode with 3 m cable
1010003	Universal power adapter, 100-240 V, 50/60 Hz
910410-WA	Orion pH 4.01 buffer pouches, 10 pack
910710	Orion pH 7.00 buffer pouches, 10 pack
911010	Orion pH 10.01 buffer pouches, 10 pack
911110	Orion rinse solution pouches, 10 pack
9179BNMD	Orion Triode gel-filled epoxy-body ORP/ATC electrode
967961	Orion ORP standard solution, 5 x 60 mL

For more information, contact your local Thermo Scientific products dealer or call our customer and technical service experts at 1-800-225-1480 (for the US and Canada) or visit www.thermoscientific.com/water.

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Water Analysis Instruments

North America
Toll Free: 1-800-225-1480
Tel: 1-978-232-6000
info.water@thermo.com

Netherlands
Tel: (31) 020-4936270
info.water.uk@thermo.com

China
Tel: (86) 21-68654588
wai.asia@thermofisher.com

India
Tel: (91) 22-4157-8800
wai.asia@thermofisher.com

Singapore
Tel: (65) 6778-6876
wai.asia@thermofisher.com

Japan
Tel: (81) 045-453-9175
wai.asia@thermofisher.com

Australia
Tel: (613) 9757-4300
in Australia (1300) 735-295
InfoWaterAU@thermofisher.com

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S-STARA121-E 1213 Rev B

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Orion™ Star™ A121 pH Portable Meter, Thermo Scientific

Supplier: THERMO ORION

Take simple pH and temperature measurements with the Orion™ Star™ A121 pH Portable Meter, which offers easy operation for basic field testing.

- Measure pH, mV, or relative mV with temperature
- Perform up to a three point pH calibration with automatic buffer recognition or manual buffer entry
- User-friendly operation with basic calibration text prompts
- Easily recall calibration data for procedure checks
- Non-volatile memory preserves 50 point internal data log and meter settings, even with loss of power
- Three-year meter warranty

Take simple, routine pH, mV, and temperature measurements with the Orion™ Star™ A121 pH Portable Meter, an easy-to-use and budget-friendly instrument for basic pH analysis in the field. Perform up to a three point pH calibration with easy recall of calibration points and slope. Quickly navigate setup menus and ensure consistent calibrations using the simple keypad layout and on-screen text prompts. The simplified, easy-to-read display shows main measurement with temperature, stability indicator, and electrode condition icon. This waterproof meter has an IP67-rated housing for rugged protection in outdoor environments.

These meters are a simple, economical solution for routine outdoor sample analysis. Featuring an emphasized measure key, selectable read modes, and measurement stability indicator, they are designed to deliver reliable measurements at an affordable price. Flexible power options with AA batteries or optional universal power adapter.

CE, TUV 3-1, FCC Class A.

Ordering Information: For compatible pH electrode, see Orion™ item 9107BNMD. For compatible buffer solutions, see Orion™ item 911110.

Expand All / Collapse All

Add to Basket

Meters and Kits

Description	Includes	Supplier No.	VWR Catalog Number	Unit	Availability	Price	Your Price	Quantity
Orion™ Star™ A121 pH Portable Meter	Star™ A121 pH meter, 4 AA batteries, literature CD, printed quick start guide, and meter test certificate	STARA1210	89206-282	Each	In Stock	$562.84	$481.23	0
Orion™ Star™ A121 pH Portable Meter Kit with Triode pH/ATC Electrode	Star™ A121 pH meter, Orion Triode gel-filled epoxy-body pH/ATC electrode, pH buffer and solution kit, protective meter armor, hard-sided field case, 4 AA batteries, literature CD, printed quick start guide, and meter test certificate	STARA1215	89206-284	Each	In Stock	$953.90	$815.58	0

Meter Kit Components and Accessories

Description	Supplier No.	VWR Catalog Number	Unit	Availability	Price	Your Price	Quantity
Hard-Sided Field Case	STARA-CS	89206-424	Each	Ordered Upon Request	$196.27	$167.81	0
Protective Meter Armor	STARA-AR	89206-432	Each	In Stock	$120.13	$102.71	0

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Generate Barcodes Barcode Label Format: Avery 5161 - 1" x 4"

Generate PDF Catalog Page

User Guide

Low Maintenance
Gel-Filled pH
Electrodes

Thermo
SCIENTIFIC

Introduction

This user guide contains information on the preparation, operation and maintenance of the Thermo Scientific Orion low maintenance gel-filled pH electrodes.

Cat. No.	Description
9107BNMD	Gel-filled pH/ATC Triode with epoxy body, waterproof BNC and 8 pin MiniDIN connectors, and 1.5 meter cable
9107WMMD	Gel-filled pH/ATC Triode with epoxy body, waterproof BNC and 8 pin MiniDIN connectors, and 3 meter cable
9107WLMD	Gel-filled pH/ATC Triode with epoxy body, waterproof BNC and 8 pin MiniDIN connectors, and 6 meter cable
9107BN	Gel-filled pH/ATC Triode with epoxy body, BNC and 8 pin DIN connectors, and 1 meter cable
9107WP	Gel-filled pH/ATC Triode with epoxy body, E DIN and banana plug connectors, and 1 meter cable
9109WP	Gel-filled pH/ATC Triode with epoxy body, E DIN waterproof and banana plug connectors, and 1 meter cable
9109WL	Gel-filled pH/ATC Triode with epoxy body, E DIN waterproof and banana plug connectors, and 6 meter cable
9206BN	PerpHecT gel-filled pH electrode with epoxy body, BNC connector and 1 meter cable
9207BN	PerpHecT gel-filled pH/ATC Triode with epoxy body, BNC and phono tip connectors, and 1 meter cable
9106BNWP / 910500	Economy gel-filled pH electrode with epoxy body, waterproof BNC connector / U.S. standard connector, and 1 meter cable
911600	Economy gel-filled pH electrode with epoxy body, semi-micro tip, BNC connector and 1 meter cable
912600	Economy gel-filled pH electrode with epoxy body, flask length, BNC connector and 1 meter cable
913600	Economy gel-filled pH electrode with epoxy body, flat surface tip, BNC connector and 1 meter cable

Required Equipment

1. Thermo Scientific Orion pH meter, such as the 3-Star pH meter, 4-Star pH/ISE meter, 4-Star pH/DO meter, 4-Star pH/conductivity meter or 5-Star pH/ISE/DO/conductivity meter.

   Gel-filled pH electrodes can be used on any pH meter with a BNC or U.S. standard connection. The electrodes can also be used on meters with a variety of inputs when an adapter cable is used. Visit www.thermo.com/water for details.

   Gel-filled Triodes have temperature connectors that are compatible with specific meters, refer to the list below.

2. Thermo Scientific Orion low maintenance gel-filled pH electrode.

   - The 9107BNMD, 9107WMMD and 9107WLMD pH/ATC Triodes have a temperature connector that is compatible with the Star Series meters.
   - The 9107BN pH/ATC Triode has a temperature connector that is compatible with the A+ Series meters.
   - The 9107WP pH/ATC Triode has a temperature connector that is compatible with the 260, 265 and 1230 meters.
   - The 9109WP and 9109WL pH/ATC Triodes have a temperature connector that is compatible with the 260A, 261S, 265A and 266S meters.
   - The 9207BN pH/ATC Triode has a temperature connector that is compatible with the PerpHecT meters.

3. pH electrode storage solution, Cat. No. 910001.

4. pH buffers, at least two pH buffers are recommended for precise measurements. One buffer should be near pH 7 and buffers should be one to three pH units apart.

5. Beakers, plastic or glass.

6. Magnetic stirrer or Orion stirrer probe, Cat. No. 096019. The stirrer probe can be used with 3-Star, 4-Star and 5-Star benchtop meters.

7. Distilled or deionized water.

Electrode Preparation

1. Remove the protective shipping cap from the sensing element and save the cap for storage.

2. Clean any salt deposits from the exterior of the electrode by rinsing with distilled water.

3. Soak the electrode in pH electrode storage solution, Cat. No. 910001, for at least one hour.

   If pH electrode storage solution is not available, a temporary storage solution can be prepared by adding 1 gram of potassium chloride (KCl) to 200 mL of pH 7 buffer.

4. Connect the electrode to the meter.

Note: When connecting a MiniDIN Triode, first attach the MiniDIN connector to the meter and then connect the waterproof BNC connector. To remove the electrode from the meter, detach the waterproof BNC connector first and then remove the MiniDIN connector. ▲

Sample Requirements

The low maintenance gel-filled electrodes have an epoxy body and should not be used in samples that contain non-aqueous solutions or organic solvents.

The gel-filled electrodes contain a silver/silver chloride (Ag/AgCl) reference that is incompatible with solutions that contain silver complexing or binding agents such as TRIS, proteins and sulfides. To measure pH in these solutions, use a ROSS* pH electrode. Proteins cause the additional problem of coating the sensing bulb, so extra care should be taken to keep the electrode clean while measuring samples.

** ROSS and the COIL trade dress are trademarks of Thermo Fisher Scientific Inc.*

Measuring Hints

- Always use fresh buffers for calibration. Choose buffers that are one to three pH units apart.
- Check electrode slope daily by performing a two buffer calibration. The slope should be 92 to 102%.
- Between measurements, rinse electrodes with distilled water and then with the next solution to be measured.
- Stir all buffers and samples at a uniform rate.
- Keep buffers and samples at equal temperatures. If samples are at different temperatures, use temperature compensation, as described in the meter user guide.
- Place a piece of insulating material, such as Styrofoam or cardboard, between the magnetic stirrer and beaker to prevent measurement errors from the transfer of heat to the sample.
- To reduce the chance of error due to polarization, avoid rubbing or wiping the electrode bulb. Use a lint-free tissue and gently blot the bulb.

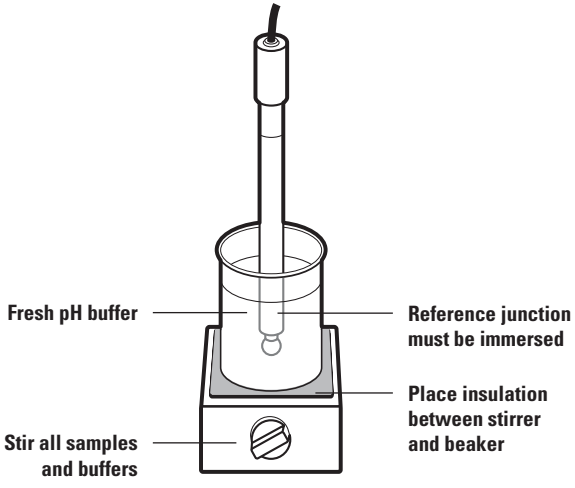


Figure 1
Measuring Hints

Electrode Calibration

General Calibration Procedure

For detailed instructions on pH calibration, manual pH calibration and temperature compensation, consult your meter user guide. When using PerpHecT electrodes with a PerpHecT pH meter, refer to the PerpHecT meter user guide for instructions on temperature calibration and LogR temperature compensated pH measurements.

One Buffer Calibration

1. Choose a buffer near expected sample pH.

2. The buffer should be at same temperature as the sample. If the buffer and samples are at varying temperatures, temperature compensation is recommended.

3. Prepare the meter according to the meter user guide.

4. Rinse the electrode first with distilled water and then with the buffer being used for calibration.

5. Place the electrode into the buffer. When the reading is stable, set the meter to the pH value of the buffer at the measured temperature. Refer to the meter user guide for a detailed procedure. **Table 1** provides pH values at various temperatures.

6. Proceed to the **pH Measurement** section.

Table 1
pH Values of Buffers at Various Temperatures

Nominal Buffer Value at 25°C	Temperature				
	0 °	**5 °**	**10 °**	**20 °**	**30 °**
1.68	1.67	1.67	1.67	1.67	1.68
3.78	3.86	3.84	3.82	3.79	3.77
4.01	4.00	4.00	4.00	4.00	4.02
6.86	6.98	6.95	6.92	6.87	6.85
7.00	7.11	7.08	7.06	7.01	6.98
7.41	7.53	7.50	7.47	7.43	7.40
9.18	9.46	9.40	9.33	9.23	9.14
10.01	10.32	10.25	10.18	10.06	9.97

Two Buffer Calibration

This procedure is recommended for precise measurements.

1. Select two buffers that bracket the expected sample pH. The first buffer should be near the electrode isopotential point (pH 7) and the second should be near the expected sample pH (pH 4 or pH 10).

2. The buffers should be at same temperature as the sample. If the buffers and samples are at varying temperatures, temperature compensation is recommended.

3. Rinse the electrode first with distilled water and then with the first buffer.

4. Place the electrode into the first buffer. When the reading is stable, set the meter to the pH value of the buffer at the measured temperature. Refer to the meter user guide for a detailed procedure. **Table 1** provides pH values at various temperatures.

5. Rinse the electrode first with distilled water and then with the second buffer.

6. Place the electrode into the second buffer. When the reading is stable, set the meter to the pH value of the buffer at the measured temperature. Refer to the meter user guide for a detailed procedure. **Table 1** provides pH values at various temperatures.

7. Proceed to the **pH Measurement** section.

Temperature					
40 °	**50 °**	**60 °**	**70 °**	**80 °**	**90 °**
1.69	1.71	1.72	1.74	1.77	1.79
3.75	3.75				
4.04	4.06	4.09	4.13	4.16	4.21
6.84	6.83	6.84	6.85	6.86	6.88
6.97	6.97	6.97	6.99	7.03	7.08
7.38	7.37				
9.07	9.01	8.96	8.92	8.89	8.85
9.89	9.83				

pH Measurement

1. Calibrate the electrode as described in the **Electrode Calibration** section.

2. Rinse the electrode with distilled water and then with the sample.

3. Place the electrode into the sample.

4. When the reading is stable, record the pH and temperature of the sample.

Electrode Storage

Short-term Storage: (up to one week) – Soak the electrode in pH electrode storage solution, Cat. No. 910001. If pH electrode storage solution is not available, a temporary storage solution can be prepared by adding 1 gram of potassium chloride (KCl) to 200 mL of pH 7 buffer. Do not store the electrode in distilled or deionized water, as it will shorten the electrode life.

Long-term Storage: (more than one week) – Rinse off any salt buildup with distilled water and remove any membrane/junction deposits. Cover the sensing surface with the protective cap containing a few drops of storage solution.

Electrode Maintenance

1. Inspect the electrode for scratches, cracks, salt crystal buildup or membrane/junction deposits.

2. Rinse off any salt buildup with distilled water. Remove any membrane/junction deposits as directed in the **General Cleaning** section.

General Cleaning

1. Soak the electrode in 0.1 M HCl or HNO_3 for 15 to 30 minutes.

2. Soak the electrode in pH electrode storage solution for at least one hour.

Cleaning Solutions

Cat. No. 900021– pH cleaning solution A for removing protein contaminants.

Cat. No. 900022– pH cleaning solution B for removing bacterial contaminants.

Cat. No. 900023– pH cleaning solution C for general cleaning.

Cat. No. 900024– pH cleaning solution D for removing oil and grease contaminants.

Cat. No. 900020– pH cleaning solution kit, includes cleaning solutions A, B, C and D.

Electrode Characteristics

Temperature Effects

The most common cause of error in pH measurements is temperature. There are at least five ways that temperature variations can affect pH: electrode slope, buffers, samples, reference element drift and temperature sensor errors

Electrode Slope Changes

The electrode slope will change with variations in temperature. Slope changes may be compensated manually, automatically with an automatic temperature compensation (ATC) probe or with LogR technology when using a PerpHecT meter and electrode. Thermo Scientific Orion pH meters calculate the slope based on the measured temperature and automatically adjust the pH value based on the temperature.

Buffer and Sample pH Changes

Buffer and sample pH values change with temperature because of their temperature dependent chemical equilibria. The pH electrode should be calibrated with buffers that have known pH values at different temperatures. Buffer values at different temperatures are given in **Table 1**. Thermo Scientific Orion pH meters automatically calibrate with the correct pH buffer values based on the measured temperature. All pH meters are unable to correct pH values back to a reference temperature because every sample has a unique pH value vs. temperature relationship. Therefore, calibration and measurements should be performed at the same temperature and pH values should be reported with temperature.

Reference Element Drift

Drift can occur when the internal reference elements inside the pH and reference portions of the electrode are reaching thermal equilibrium after a temperature change. Long-term drift or slow response can last until the sample and electrode are at the same temperature.

Temperature Sensor Errors

When a pH and temperature probe are placed into a sample that varies significantly in temperature, the readings can drift for two reasons. First, the temperature response of the electrode and temperature probe may not be similar, which prolongs equilibration and drift. Second, a sample may not have a uniform temperature. Therefore, the pH electrode and temperature probe are responding to different environments.

Using LogR technology, PerpHecT meters sense the temperature directly from the PerpHecT pH electrodes. The pH and temperature response is identical and both measurements occur at the sensing bulb. Drift is minimized and errors due to environmental differences are eliminated.

PerpHecT® Electrode Operation with PerpHecT® pH Meters

When PerpHecT electrodes are used with a PerpHecT pH meter, enhanced temperature compensation is achieved without the need of a separate ATC probe. Using LogR technology, the temperature of the solution is measured through the resistance of the pH electrode. PerpHecT pH electrodes are manufactured to meet the PerpHecT meter specifications, so optimum performance and accuracy are achieved in LogR mode.

Each PerpHecT pH electrode must be calibrated for temperature before a pH measurement is performed using LogR technology. Refer to the PerpHecT meter user guide for details. For maximum precision, a three point temperature calibration is recommended. Do not perform a one point temperature calibration if measured solutions will be below 20 °C. The following tables illustrate the expected pH compensation error for one, two and three point temperature calibrations. The accuracy values are valid only when the temperature calibration is performed within the stated temperature range. The higher temperature range data will apply to measurements made above that temperature range, provided that calibration points are within 20 °C of each other. When highly accurate pH results are desired, a separate ATC probe is recommended.

One Point Temperature Calibration

Electrode	Average temp. error 20-30 °C	Average temp. error 30-50 °C	Average pH error 20-30 °C	Average pH error 30-50 °C
9206BN	0.14	0.13	0.002	0.002
9207BN	0.38	0.27	0.005	0.003

Two Point Temperature Calibration

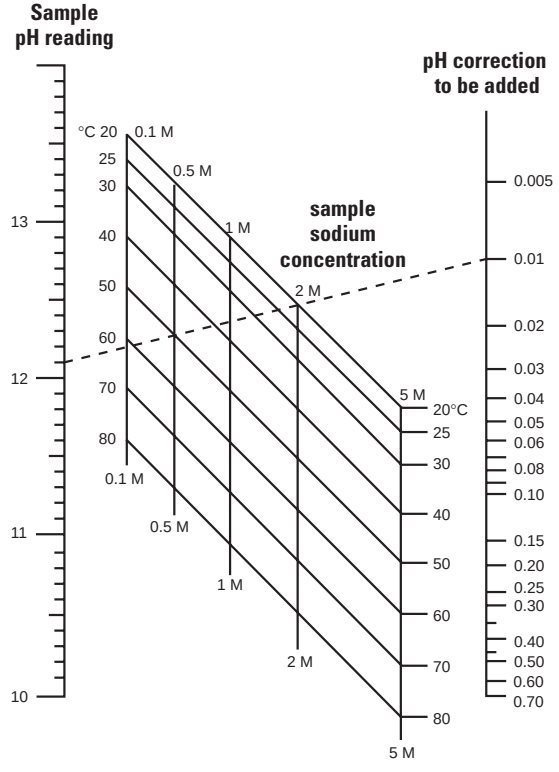
Electrode	Average temp. error 20-30 °C	Average temp. error 30-50 °C	Average pH error 20-30 °C	Average pH error 30-50 °C
9206BN	0.29	0.06	0.003	0.001
9207BN	0.16	0.07	0.002	0.001

Three Point Temperature Calibration

Electrode	Average temp. error 20-30 °C	Average temp. error 30-50 °C	Average pH error 20-30 °C	Average pH error 30-50 °C
9206BN	0.06	0.05	0.001	0.001
9207BN	0.02	0.02	0.000	0.000

Interferences

Sodium ions are the principal interference of the pH electrode and cause increasing error at higher pH (lower hydrogen ion activities) and at higher temperatures. Because the pH membrane is composed of special low sodium error glass, error due to sodium is negligible when measuring at pH values less than 12. When measuring at pH values greater than 12, add the correction value from the nomograph below to the observed pH reading.



Typical Sodium Error

Example:

pH reading	12.10
Sodium concentration	0.5 M
Temperature	50 °C
Correction	0.01
Corrected pH reading	12.11

Troubleshooting

Follow a systematic procedure to isolate the problem. The pH measuring system can be divided into four components for ease in troubleshooting: pH meter, electrode, sample/application and technique.

pH Meter

The meter is the easiest component to eliminate as a possible cause of error. Thermo Scientific Orion pH meters include an instrument checkout procedure and shorting cap for convenience in troubleshooting. Consult the pH meter user guide for directions.

Electrode

To test electrode operation:

1. Connect the electrode to a working meter that has a mV measuring mode.

2. Set the meter to the mV measuring mode.

3. Rinse the electrode with distilled water and then insert the electrode into fresh pH 7 buffer.

4. When the reading is stable, record the mV value of the pH 7 buffer. The mV value should be -30 to +30 mV.

5. Rinse the electrode with distilled water and then insert the electrode into fresh pH 4 buffer.

6. When the reading is stable, record the mV value of the pH 4 buffer. The mV value should be +150 to +210 mV.

7. Calculate the absolute mV difference between the two buffers. The mV difference should be 160 to 180 mV. The actual mV values will change as the electrode ages, but the mV difference between the two buffers should always be 160 to 180 mV.

If the electrode fails this procedure, clean the electrode thoroughly as directed in the **Electrode Maintenance** section. Replace the electrode if cleaning and maintenance fail to rejuvenate it.

Sample/Application

The electrode and meter may operate with buffers, but not with the sample. In this case, check the sample composition for interferences, incompatibilities or temperature effects.

Technique

If trouble persists, review operating procedures. Review calibration and measurement sections to be sure proper technique has been followed.

Assistance

After troubleshooting all components of your measurement system, contact Technical Support. Within the United States call 1.800.225.1480 and outside the United States call 978.232.6000 or fax 978.232.6031. In Europe, the Middle East and Africa, contact your local authorized dealer. For the most current contact information, visit www.thermo.com/water.

Warranty

For the most current warranty information, visit www.thermo.com/water.

Ordering Information

Electrodes

Refer to the **Introduction** section for a complete list of low maintenance gel-filled pH electrodes.

Accessories

Cat. No.	Description
910001	pH electrode storage solution, 475 mL bottle
910003	12 mm diameter electrode storage bottles, 3 pack
910004	8 mm diameter electrode storage bottles, 3 pack
910006	6 mm diameter electrode storage bottles, 3 pack
900020	pH cleaning solution kit, includes 1 x 15 mL bottle each of cleaning solutions A, B, C and D; pipette and beaker
900021	pH cleaning solution A, includes 4 x 15 mL bottles, pipette and beaker
900022	pH cleaning solution B, includes 4 x 15 mL bottles, pipette and beaker
900023	pH cleaning solution C, includes 4 x 15 mL bottles, pipette and beaker
900024	pH cleaning solution D, includes 4 x 15 mL bottles, pipette and beaker
910199	All-in-One pH buffer kit, includes 475 mL bottle each of pH 4.01, 7..00 and 10.01 buffers and pH electrode storage solution, and 12 mm electrode storage bottle
910168	pH 1.68 buffer, 475 mL bottle
910104	pH 4.01 buffer, 475 mL bottle
910105	pH 5.00 buffer, 475 mL bottle
910686	pH 6.86 buffer, 475 mL bottle
910107	pH 7.00 buffer, 475 mL bottle
910918	pH 9.18 buffer, 475 mL bottle
910110	pH 10.01 buffer, 475 mL bottle
910112	pH 12.46 buffer, 475 mL bottle

Visit www.thermo.com/water for additional buffers and buffer sizes.

Thermo Fisher Scientific

Environmental Instruments
Water Analysis Instruments

166 Cummings Center
Beverly, MA 01915 USA
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Dom. Fax: 978-232-6015
Int'l. Fax: 978-232-6031

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APPENDIX 4
ALS ENVIRONMENTAL QUALITY ASSURANCE MANUAL

ALS Environmental

QUALITY ASSURANCE MANUAL

ALS Environmental – Valparaiso Facility
2400 Cumberland Drive
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QUALITY ASSURANCE MANUAL

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ANNUAL REVIEW

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Signature	Title	Date
Signature	Title	Date
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3	4.2	2/4.2
4 / VAL-QS-014	4.3	2/4.3
5 / VAL-GEN-006	4.4	2/4.4
6 / VAL-GEN-007	4.5	2/4.5
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1) Introduction and Scope

The Quality Assurance Manual (QAM) outlines the quality system used at the Valparaiso laboratory of ALS Environmental (ALS Group USA Corp. dba ALS Environmental). This manual defines the policies, procedures, and documentation that: (1) assure analytical services meet a defined standard of quality, (2) provide data of known documented quality, and (3), where applicable, demonstrates regulatory compliance.

The QAM sets the standard under which all laboratory operations are performed including the laboratory's organization, objectives, and operating philosophy. The QAM has been prepared to assure compliance with the 2016 TNI Environmental Laboratory Sector Standard, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, which is consistent with the ISO/IEC 17025:2005 requirements that are relevant to environmental testing services. In addition, the policies and procedures outlined are established to be compliant with the various accreditation and certification programs listed in Appendix J.

1.1 Scope of Testing

ALS Environmental is a professional analytical service laboratory providing analytical services for a variety of matrices including, but not limited to, aqueous, solid, hazardous waste, and air. Analytical services are based upon EPA approved methods and/or other promulgated protocols. Refer to Appendix J for a list of analytical capabilities and corresponding accreditation status.

1.2 Management of the Quality Assurance Manual

The Quality Assurance Manager is responsible for maintaining the QAM. The QAM is reviewed annually by the QA Manager and laboratory personnel to ensure it still reflects current practices and meets the requirements of any applicable regulations or client specifications.

The QAM is considered confidential within the ALS Environmental and may not be altered in anyway except by approval of the Laboratory Director and QA Manager. If it is distributed to external users, it is for the purpose of reviewing the management system and may not be used for any other purpose without written permission.

1.3 A list of commonly used definitions and acronyms used in this manual is included in Appendix A.

2) Organization

The ALS Environmental Valparaiso facility is a commercial laboratory located at 2400 Cumberland Drive, Valparaiso, IN and performs a variety of testing services and activities to meet the needs of laboratory clients. The laboratory systems are designed to also meet the requirements of applicable certification and accreditation bodies.

2.1 Laboratory Organizational Structure

The laboratory is a legally identifiable organization, and a division of a publicly owned corporation, ALS Group USA Corp. dba ALS Environmental. The US tax ID number is available upon request. ALS Group USA Corp. is a wholly owned subsidiary of ALS Limited (ASX: ALQ).

2.2 Avoiding Conflict of Interest through Organizational Structure

2.2.1 Through application of the policies and procedures outlined in this QAM and use of a defined organizational structure, the laboratory assures that it is impartial and that personnel are free from undue commercial, financial, or other undue pressures that might influence their technical judgment.

2.2.2 Policies are in place to prevent outside pressures or involvement in activities that may affect competence, impartiality, judgment, operational integrity, or the quality of the work performed at the laboratory.

2.2.3 Management and technical personnel have the authority and resources to carry out their duties and have procedures to identify and correct departures from the laboratory's management system.

2.2.4 Personnel understand the relevance and importance of their duties as related to the maintenance of the laboratory's management system. Ethics and data integrity procedures ensure that personnel do not engage in activities that diminish confidence in the laboratory's capabilities. Procedures and policies are also established to ensure confidentiality is maintained.

2.3 An organization chart is provided in Appendix B that shows the operational structure and reporting relationships in the laboratory. Additional information regarding responsibilities, authority and interrelationship of personnel who manage, perform or verify testing is included in Section 3 of this QAM.

3) Management

3.1 Quality Policy Statement

The laboratory's Quality System is documented in this manual and the associated Standard Operating Procedures (SOPs). Together they describe the policies, objectives, principles, organizational authority, responsibilities, accountability, and means of implementation for ensuring quality in the work processes, products, and services. The objective of the quality system is to generate data of known and documented quality that meets specified requirements and provide for its continuous improvement. The laboratory's policy in support of this objective is to: (1) utilize good laboratory practices, (2) maintain documented quality standards, (3) uphold the highest level of service, and (4) comply with applicable Standards of accreditation (e.g. TNI). The laboratory ensures that personnel are free from any commercial, financial, or other undue pressures that might adversely affect the quality of work. This policy is implemented and enforced through the management's commitment to the principles and practices outlined in this manual. The primary responsibility for quality rests with each individual within the laboratory organization. Accordingly, every laboratory employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize him or herself with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on the ethical principles and procedures surrounding the generation of data. The laboratory maintains a strict policy of client confidentiality and holds all employees to this policy.

3.2 Management Requirements

Laboratory management ensures that the laboratory's policies and objectives for quality are documented by reference or by inclusion in the QAM, and that the QAM is: (a) communicated to, (b) made available to, (c) understood by, and (d) implemented by, all personnel concerned. Where the QAM documents specific laboratory requirements,

a separate SOP or policy is not required.

3.3 Quality Assurance Manual

The Quality Assurance Manual is maintained current and up-to-date by the quality assurance department. All employees must complete a read-receipt form stating they 1) have read the Quality Assurance Manual, 2) understand the contents, and 3) will adhere to the stated policies. The completed read-receipt form is kept on file by the quality assurance department.

3.4 Management Responsibility and Authority

3.4.1 The laboratory management team includes the Laboratory Director, Laboratory Supervisor(s), and the Quality Assurance Manager. This group has overall responsibility for technical operations and the authority needed to generate/maintain the defined level of quality stated in the Quality Policy (above) and is upheld through the application of documented policies and procedures.

3.4.2 Management is responsible for the assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting the quality of environmental tests. Management is responsible for defining the minimal level of education, qualifications, experience, and/or skills necessary for completion of the assigned responsibilities.

3.4.3 Management bears specific responsibility for maintenance of the Quality System. This includes defining roles and responsibilities of personnel, approving documents, and providing training.

3.4.4 Management ensures technical competence of personnel operating equipment, performing tests, evaluating results, and signing reports by limiting authority to those who are appropriately trained and/or supervised. Training is kept up to date as described in Section 20 of this document.

3.4.5 Laboratory management must make available all necessary equipment required for the correct performance of the scope of environmental testing presented in this QAM. Only trained and authorized personnel can operate equipment.

3.4.6 Management also bears responsibility for ensuring that audit findings and/or corrective actions are addressed and completed within required time frames. Designated alternates are appointed by management during the absence of the Laboratory Director, Technical Director, or the Quality Assurance Manager, and always if the absence is more than 15 days.

3.5 Job Descriptions and Qualifications (qualifications of key personnel are given in Appendix B)

3.5.1 Laboratory Director

The Laboratory Director is responsible for all laboratory activities as the highest-level manager. He/she provides administrative, operational, and Technical leadership through planning, allocation, management of personnel, and management of resources. He/she approves the QAM, provides resources for implementation of the QA program. The Laboratory Director position requires a BS or BA degree in Science, Engineering, or Management with five years supervisory experience in environmental laboratory operations.

3.5.2 Lab Supervisor

The Lab Supervisor reports directly to the Laboratory Director and is responsible for day-to-day supervision of laboratory operations. He/she assures production of reliable data through the monitoring of analytical procedures, corroborating analysis performed, and approving staff capability. He/she certifies that personnel with appropriate educational and/or Technical background perform all tests for which the lab is accredited according to SOP specifications. He/she reviews, schedules, and oversees the implementation of new methodologies. In the absence of the Lab Supervisor, the Laboratory Director or Quality Assurance Manager must maintain these duties. The Lab Supervisor position requires a BS or BA degree in Science, Engineering, or Management with five years supervisory experience in environmental laboratory operations.

3.5.3 Quality Assurance/Technical Manager

The Quality Assurance (QA) Manager is responsible for ensuring that the quality system is documented, implemented, and adhered to in the course of laboratory operations. He/she has direct access to the Laboratory Director and is independent of daily laboratory operations. He/she is tasked with:

- Day-to-day implementation of the quality program,
- oversight of quality control processes, including establishing control limits,
- maintaining the Quality Assurance Manual,
- maintaining quality assurance records,
- acquiring and maintaining laboratory accreditations.
- evaluating data,
- performing assessments without managerial influence,
- conducting internal audits,
- arranging for external audits,
- monitoring corrective actions, and
- notifying management of any deficiencies and/or opportunities for improvement in laboratory operations.

The QA Manager performs or coordinates a QA Management System review annually according to SOP VAL-QS-017, *QA Management Review*. He/she has the authority to place a stop work order on any non-compliant work area. In the absence of the QA Manager, the Laboratory Director must maintain these duties. The QA Manager position requires a BS or BA degree in Science, Engineering, or Management with a minimum of five years experience in environmental laboratory operations and two years experience in quality system management.

3.5.4 Project Manager(s)

Project Managers (PM) are responsible for ensuring that analyses performed by the laboratory meet all project, contract, and/or client-specified requirements. The PM is tasked with 1) relaying project requirements to the staff, 2) review of sample log-in information, 3) monitoring/communicating project progress, and 4) reviewing/issuing final reports to the client. In the absence of the Project Manager, the Laboratory Director (or designate) must maintain these duties. The PM position requires a BS or BA in Science, Engineering, or Management with five years applicable experience.

3.5.5 Safety Manager

The Safety Manager reports to the Laboratory Director and is responsible for administration of the laboratory's safety program. He/she is tasked with 1) implementing safety policies, 2) reviewing accidents and/or incidents, 3) monitoring hazardous waste disposal, and 4) conducting routine safety inspections. In the absence of the Safety Officer, the Laboratory Director (or designate) must maintain these duties. The Safety Officer position requires a high school diploma and two years applicable laboratory experience.

(Note: In lieu of formal education requirements, three years experience can be considered equivalent to one year formal education.)

3.6 Data Integrity System

Management functions include implementation of a data integrity system. The data integrity system employed at ALS Environmental is an integrated approach designed to ensure the production of defensible and quality data. The overall system consists of a three-tier approach as documented in laboratory SOP VAL-QS-015, *Data Integrity System*.

The ALS Environmental policies and requirements for Ethics and Data Quality are described in the corporate SOP CE-GEN-001, Laboratory Ethics and Data Integrity. The employee Ethics and Data Quality Agreement from the SOP is provided in Appendix C.

System policy is based upon criteria specified by ISO 17025, US EPA, TNI, and (if required) project specific criteria. System programs to support this policy include approved Standard Operating Procedures, employee training, internal/external assessments, and annual management review. Within each system program, critical components of data integrity are employed. These components include defined data quality objectives, data generation procedures, data verification, and data validation. Specifics of these components are detailed in individual documents. Prior to final release, validated data is compared to data quality objectives in order to assure its worthiness.

The laboratory has the resources and authority to operate a management system that is capable of identifying departures from that system and from procedures during testing, and initiates actions to minimize or prevent departures.

3.7 Client Confidentiality

As a function of managing client projects and conducting testing, it is recognized that the laboratory will obtain, or be provided, information which may be confidential in nature. Management ensures that each area of client interaction is treated as confidential according to following criteria.

3.7.1 The laboratory confidentiality policy is to not divulge or release any information to a third party without proper authorization.

3.7.2 All electronic data are kept confidential, based on technology and laboratory limits, as required by client or regulatory specifications.

3.7.3 Procedure(s) for maintaining confidentiality requirements are documented in SOP VAL-GEN-004, *Client Confidentiality*.

4) Document Control

4.1 The purpose of document management is to preclude the use of invalid and/or

obsolete documents. The following guidelines are used for document management.

4.2 Document Type

The laboratory manages three types of documents: 1) controlled, 2) approved, and 3) obsolete. All documents that affect the quality of laboratory data are managed appropriate to the scope and depth required.

4.2.1 A controlled document is one that is internally generated, uniquely identified, issued, and maintained as part of the quality system. Controlled documents are uniquely identified with: 1) effective date, 2) revision number, 3) page number, 4) total number of pages, and 5) the signatures of the issuing authority (i.e. management).

4.2.2 An approved document is one that has been released or acknowledged externally via secure electronic means by the issuing authority. Examples of approved documents include EPA, ASTM, and AWWA methodologies.

4.2.3 An obsolete document is one that has been superseded by a more recent version or that reflects a discontinued practice. Obsolete controlled documents are maintained according to SOP VAL-QS-011, *Record Archival*.

4.3 Document Approval, Review, and Distribution

4.3.1 Approval

All documents that affect the generation and reporting of laboratory data must be approved, at a minimum, by the Laboratory Director (or Operations Manager), QA Manager, and appropriate Department Supervisor. The Laboratory Director and QA Manager, at a minimum, must approve all documents that affect quality assurance, administrative, general, and health/safety programs. Other approvals may be required as documented in SOP VAL-QS-014, *Document Control & Laboratory Records*.

4.3.2 Review

Documents are reviewed periodically to ensure their contents are in compliance with current quality system requirements and accurately reflect current operations. In particular, SOPs are reviewed biennially to ensure they continue to reflect actual practice.

4.3.3 Distribution

Approved copies of all controlled documents are stored on the shared server in a secure (Adobe) format and are available to all personnel.

The QA department maintains a hardcopy set of controlled documents in case of server disruption. The QA department also maintains the original copy of the controlled document in a protected environment.

Specific procedures for the distribution of documents are documented in SOP VAL-QS-014, *Document Control & Laboratory Records*.

4.4 Document Management

4.4.1 The QA Manager (or designee) must maintain a master list of controlled documents referencing the document's identification. The master list is updated whenever documents are revised, retired, or become obsolete.

4.4.2 Specific procedures for document management are specified in SOP VAL-QS-

014, *Document Control & Laboratory Records*.

4.5 Changes to Documents

4.5.1 Hardcopy Documents

- All document changes are reviewed prior to promulgation and approved as documented in Section 4.2.1.
- All modifications, additions, and/or changes must be incorporated into a new hardcopy revision.
- The QA Manager (or designee) is responsible for maintaining hardcopy format as documented in Section 4.2.3.

4.5.2 Electronic Documents

- All changes to document hardcopy must be stored electronically in a secure format and be made available to all employees.
- Obsolete electronic formats are removed from service and placed in an archived folder.
- The QA Manager (or designee) is responsible for maintaining electronic formats.

4.5.3 Procedures

Procedural processes for modifications and changes to controlled documents are specified in SOPs VAL-QS-014, *Document Control & Records*, and VAL-GEN-001, *SOP Preparation & Management*.

4.6 Obsolete Documents

All obsolete documents are removed from electronic distribution, or otherwise prevented from unintended use, and archived. Procedural processes for archival of obsolete documents are specified in SOP VAL-QS-011, *Record Archival*.

4.7 Signature Policy

4.7.1 It is a policy of ALS Environmental to allow the use of electronic signatures. For data reporting an electronic signature may be applied to the report by an approved report signatory and is binding to the same extent as a handwritten wet signature.

4.7.2 To authenticate the electronic signature the identity of the signatory is verified before their electronic signature can be created. Each electronic signature shall be unique to a single individual and shall not be used by any other individual. These signatures are established using only defined procedures within the software and are verified using the two distinct components of username and password. Each use of the electronic signature requires entry of the username and the password. The report may not be changed once the signature has been applied.

4.7.3 Additionally, as a form of 'signature' used for LIMS, email, and certain internal documentation processes (e.g. acknowledgements, attestations, audit trails, etc.), and other electronic tools the user's system login credentials are used to verify and authenticate the identity of the user. Following login, these credentials are used to identify and document the user.

4.7.4 Approved Signatories:

4.7.4.1 Tenders and Contracts: Laboratory Director, Technical Sales Representative, Project Manager

4.7.4.2 Chain of Custody and Sample Receiving Documentation: Client Services Personnel, Technical Sales Representative, Project Manager, Laboratory Supervisor, Laboratory Director

4.7.4.3 Purchase Orders: Laboratory Supervisor and Laboratory Director

4.7.4.4 Final Report: Project Manager, Laboratory Director

4.7.4.5 Accreditation Documentation: QA Manager, Laboratory Director

4.7.4.6 Corrective Action Reports: QA Manager, Laboratory Director

4.7.4.7 Controlled Documents (including SOPs): Laboratory Director, Quality Officer, Operations Manager, Laboratory Supervisor

5) Review of Requests, Tenders and Contracts

5.1 All work must be reviewed prior to acceptance in order to assure that: 1) requirements are clearly defined, 2) the laboratory has adequate resources, and 3) the test method is applicable to project specifications. This process ensures that all work is given adequate attention without shortcuts that might compromise data quality. Contracts for new work can be presented as formal bids, proposals, signed documents, or by verbal/electronic inquiry.

5.2 Procedure for the Review of Work Requests

5.2.1 Review of work requests is conducted according to the guidelines specified in SOP VAL-GEN-006, *Resource Review*.

5.2.2 The Project Manager (or Sales Representative in case of bid) and Laboratory Director determines if the laboratory has the necessary accreditations and resources to meet the work request.

5.2.3 The Project Manager (or Sales Representative) will:

- Provide the perspective client with the requested bid information if laboratory capability/capacity meets project requirements, or
- Inform the perspective client of any potential conflict or inability to complete the work per specification, and
- Resolve any differences between the initial request and final contract prior to sample receipt or commencement of work.

5.2.4 Changes to the Scope of Work initiated after commencement of work must be subjected to the same review process.

5.3 Allowed Deviation from Standard Operating Procedures

5.3.1 When a client requests a modification to an SOP the Project Manager handling that project must discuss the proposed deviation with the laboratory supervisor and obtain their approval to accept the project. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP.

5.3.2 When a client request necessitates a deviation or departure from company policies or procedures involving any non-technical function, the allowed

deviation must be approved by the laboratory or the laboratory director. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

5.4 Documentation of Review

5.4.1 Executed contracts are copied in secure format and stored on the shared server. Originals are maintained in a secure area designated by the Laboratory Director.

5.4.2 Additional records are maintained by the Project Manager including pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

6) Subcontracting of Tests

6.1 A subcontract laboratory is defined as a laboratory external to the Valparaiso, IN facility which performs analyses for the laboratory. When subcontracting analytical services, the laboratory assures work requiring accreditation is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for test performance.

6.2 Procedure for Subcontracting

6.2.1 Subcontracting is conducted according to the procedures documented in SOP VAL-GEN-007, *Sample Sub-Contracting*.

6.2.2 The client must be notified of the laboratory's intent to subcontract prior to sample receipt. Acknowledgement of client acceptance must be maintained with the bid or work order information.

6.2.3 The laboratory, to which samples are subcontracted, must maintain all appropriate accreditations relative to client requirements.

6.2.4 Project managers must maintain a list of approved subcontracted laboratories within LIMS and, whenever possible, obtain copies of their respective quality assurance protocols.

6.2.5 Final reports must identify all test results from subcontracted laboratories.

6.2.6 Identification of sub-contracted laboratories within ALS Environmental must be documented in final reports.

7) Purchasing Services and Supplies

7.1 The laboratory ensures that purchased supplies and services affecting the quality of environmental tests are of the required or specified quality by using approved suppliers and products. Upon receipt, traceability of reagents, chemicals, and standards is maintained throughout the entire analytical process.

7.2 Purchasing Supplies and Services

7.2.1 Purchasing of supplies and services is conducted as described in SOP VAL-GEN-010, *Procurement*.

7.2.2 Procedures for the receipt, storage, and tracking of reagents, chemicals, and standards are documented in SOP VAL-QS-001, *Reagent & Standard Tracking*.

7.2.3 Purchased chemicals must be reagent grade or higher as specified, and the specifications for specific reagents, chemicals, and standards must be documented in the applicable method SOP.

7.2.4 A list of approved vendors must be maintained by the corporate purchasing department.

7.2.5 The Department Supervisor(s) must ensure that supplies are of the appropriate quality and/or purity prior to ordering.

8) Service to the Client

The laboratory collaborates with clients and/or their representatives to clarify their requests and ensure laboratory performance related to the work. Requests are reviewed to establish the nature of the request and the laboratory's ability to comply with the request within the confines of statutes or regulations, without risk to the confidentiality of other clients.

8.1 Client Support

8.1.1 Communication with the client, or their representative, is maintained to provide proper instruction and modification for testing. Technical staff is available to discuss any technical questions or concerns the client may have. The client, or their representative, may be provided reasonable access to laboratory areas for observation or evaluation of testing.

8.1.2 Delays or major deviations to the testing are communicated to the client immediately by the assigned Project Manager.

8.1.3 The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information not requested or agreed upon prior to sample analysis.

8.2 The laboratory seeks negative and positive feedback from clients, using mechanisms such as follow-up inquiries after project completion, surveys, and email solicitation. Feedback provides acknowledgement of performance, input on service, possible corrective or preventive actions, and opportunities for continuous improvement.

9) Complaints

9.1 Complaint resolution is an integral component of the laboratory quality system with the purpose of improving the laboratory's quality, integrity, and service. All complaints, whether external (customer) or internal in origin, are documented and investigated. Complaint resolution is conducted according to the following criteria.

9.2 Procedures for Handling Customer Complaints

9.2.1 The Project Manager or Laboratory Director must ensure that all customer complaints are documented and forwarded to the QA department. This also applies to requests for report and/or data verification.

9.2.2 The QA Manager in conjunction with the Laboratory Supervisor must investigate and resolve the complaint.

9.2.3 The QA Manager must initiate a corrective action report for any investigation that indicates laboratory error.

9.2.4 Procedure(s) for handling customer complaints are documented in SOP VAL-ADM-004, *Complaint Resolution*.

10) Facilities and Equipment

10.1 Laboratory facilities are designed and organized to facilitate testing of environmental samples. Environmental conditions are monitored to ensure that they meet method specifications, do not invalidate results, and do not adversely affect the required quality of any measurement. Access to, and use of areas affecting the quality of the environmental tests is controlled by restriction to authorized personnel only.

10.2 Separate work areas are designated by application within the facility. Each workspace is complimented by dedicated air handling systems, appropriate instrumentation with computer hardware, and a secure data management system. The floor design provides separate secure storage of samples, solvents, materials inventory, and hazardous waste.

10.3 The laboratory security features provide for sample integrity and storage. Access to the facility is limited to the front door and the receiving door. During working hours, all are monitored. Guests are escorted/monitored while in the facility.

10.4 Procedures for temperature monitoring are documented in SOP VAL-EQ-002, *Thermometer Calibration and Temperature Monitoring*.

10.5 The facility floor plan is provided in Appendix D of this manual. A listing of equipment is provided in Appendix E.

10.6 General Equipment Procedures

10.6.1 Routine preventative maintenance procedures are document in SOP VAL-EQ-004, *Preventative Maintenance*. Preventative maintenance for common laboratory instrumentation can be found in Section 16. Most major equipment is covered either under warranty, service contract, or serviced by defined outside contractors.

10.6.2 Laboratory personnel maintain equipment and instruction manuals for use.

10.6.3 Procedures for validating laboratory equipment to ensure that it meets laboratory and method specifications prior to placing into service are described in SOP VAL-QS-005, *Validation of New Instrumentation and New Methods*.

10.6.4 Procedures for ensuring test equipment (hardware and software) are protected from adjustments that can invalidate test results are documented in SOP VAL-IT-003, *IT System Security*.

10.6.5 Equipment that has been shown or is suspected to be defective is:

- Removed from service
- Isolated or clearly labeled as “Out of Service”
- Repaired or replaced
- Validated prior to returning to service
- If shown that previous tests have been affected, procedures for non-conforming work must be followed.

10.6.6 Maintenance logbooks are assigned to each piece of equipment per SOP VAL-EQ-004, *Preventative Maintenance*. A LIMS module documents the following instrument information:

- Instrument identity
- Date acquired and placed in service
- Condition, if known (new, used, refurbished)
- Applicable service contract (if any)

10.7 Support Equipment

10.7.1 Support equipment includes, but is not limited to: balances, ovens, water baths, freezers, refrigerators, incubators, temperature measuring devices, volumetric dispensing devices, and thermal/pressure sample preparation devices.

10.7.2 All support equipment must be maintained in proper working order, and all raw data records must be retained to document equipment performance.

10.7.3 All support equipment must be calibrated or verified annually using NIST traceable references where available.

10.7.4 Balances, ovens, refrigerators, freezers, and water baths must be checked daily prior to use to ensure operation within defined criteria.

10.7.5 Mechanical volumetric dispensing equipment must be checked for accuracy quarterly.

10.7.6 Various other types of support equipment have requirements based upon application. Refer to the individual method SOPs for specifics.

11) Sample Management

From sample receipt to analytical completion, sample management is critical to maintaining quality measurements. Appendix F contains a list of sample containers, preservatives, and holding times for common analytical procedures. The following procedures are utilized to maintain sample integrity during the analytical process.

11.1 Sample Receipt and Acceptance

11.1.1 Procedures for sample receipt and acceptance are documented in SOP VAL-SM-001, *Sample Receipt & Log-In*. Chain of custody procedures are established to document sample custody transfer at the time of sample receipt, using chain-of-custody (COC) forms accompanying the samples. Custody seals are sent by the lab if the sampling containers are ordered from the laboratory, and during sample receipt it is also noted if custody seals were present. Shipping records are maintained with the chain of custody records.

11.1.2 The following preservation checks are performed and documented upon receipt.

11.1.2.1 Thermal preservation

- For samples that require preservation at 4˚C, the acceptable range is “from just above freezing to 6˚C”.
- Samples that are delivered to the lab by local courier as they are collected are likely not to have reached a fully chilled temperature. This is acceptable if there is evidence that chilling has begun. On the laboratory receipt form record if ice is present and the current temperature.

11.1.2.2 Residual chlorine (from chlorinated source)

- Verify sufficient sodium thiosulfate is present (sufficient to neutralize 5mg/L chlorine for drinking water and 15 mg/L chlorine for wastewater).
- Chlorine residual is checked in the field and documented.

11.1.2.3 pH checks

- The pH of samples requiring acid and/or base preservation is checked upon sample receipt.

11.1.3 If the applicable checks performed upon sample receipt indicate the criteria are not met the sample is placed "on hold" until either the decision to proceed is agreed upon with the client and documented, or the decision to reject is confirmed with the client and documented. The condition is noted on the Chain of Custody form and laboratory receipt form. Affected data are qualified or narrated in the report.

11.1.4 The sample acceptance policy is provided to all field crews and is documented as an attachment in the above referenced SOP. Sample submission sheets from the field are maintained by the applicable Project Manager and scanned in a secure format onto the shared server.

11.2 Sample Identification

Samples are uniquely identified in a permanent electronic record to maintain sample integrity and to document receipt of all sample containers. Samples are assigned sequential numbers that cross reference specific information. This information is maintained in the LIMS database and includes:

- Client or project name
- Date and time of sampling
- Date and time of receipt at lab
- Unique laboratory identification number
- Unique field identification
- Initials of recorder
- Analyses requested
- Comments regarding rejection (if any).

11.3 Sample Storage and Transport

11.3.1 Samples are held in a secure environment with restricted access and the storage conditions are continually monitored and the conditions recorded.

11.3.2 Samples are stored apart from standards, reagents, or other potentially contaminating sources such that cross-contamination is minimized. All portions of samples, including extracts, digestates, and leachates are maintained separately pending analysis.

11.3.3 Samples that are transported under the responsibility of the laboratory are done so safely and according to method specific storage conditions.

11.4 Field Sampling

11.4.1 If field sampling is completed by laboratory personnel, sampling is based upon appropriate statistical methods, whenever practical. Sampling methods may also be those requested by the client.

11.4.2 Sampling is performed according to the applicable sampling SOP. Records are maintained of the sampling procedure, the environmental conditions, sampling location, and identity of field personnel.

11.5 Procedures for sub-sampling within the laboratory are documented in SOP VAL-QS-008, *Sub-Sampling and Sample Homogenization*.

11.6 Sample Disposal

Samples are disposed of according to Federal, State, and local regulations. Procedures for sample disposal are documented in SOP VAL-SAF-001, *Waste Disposal Procedures*.

12) Analytical Procedures

12.1 Analytical Methods

12.1.1 ALS Environmental employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates; EPA 40CFR parts 136 and 141, and Supplements; and *Standard Methods for the Examination of Water and Wastewater*. References for these methods are given in Section 23. Other published methods, such as state methods, program-specific methods, or in-house methods may be used.

12.1.2 Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection and/or reporting limits, the expected concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives.

12.1.3 The implementation of methods by the laboratory is described in Standard Operating Procedures (SOPs) specific to each method.

12.2 Standard Operating Procedures

Standard Operating Procedures are written procedures that describe in detail how to conduct laboratory processes, and are of two types: 1) analytical SOPs, which have specifically required details, and 2) general use SOPs which document administrative, quality, or broad spectrum laboratory procedures. SOPs are used to ensure consistent application and performance of laboratory procedures. SOPs, regardless of type, are maintained such that:

- SOPs are accessible to all personnel, and
- Each SOP has a unique identifier, revision number, effective date, and approval signatures.

The laboratory maintains SOPs for all accredited test methods, and for procedures that support these test methods. Appendix G documents SOPs that are currently in use. Support procedures include, but are not limited to, quality assurance, information technology, sample management, health/safety, and general laboratory practices. SOPs are prepared and managed in accordance with the specifications documented in VAL-GEN-001, *SOP Preparation & Management*.

12.3 All methods must be validated before they are put into use. Initial test method validation includes establishing a valid calibration protocol, Demonstration of Capability (DOC), determination of the Method Detection Limit and/or Limit of Detection (LOD), and the Limit of Quantitation (LOQ).

12.3.1 Before performing a DOC study an appropriate calibration range must be established and calibration conducted which meets acceptance criteria. The calibration range is within the linear range of the analysis (or instrument) and is established by analyzing a series of standards, one of which is at or below the expected LOQ. The calibration range or method-specific calibration procedures may be described in test method. Refer to Section 13 for details.

12.3.2 The Demonstration of Capability (DOC) is a procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision. In general, this procedure requires the preparation and analysis of a known concentration of each analyte spiked in four separate aliquots of laboratory pure matrix. These samples are carried through the entire preparation and analytical procedure. The resulting recovery and the standard deviation (or RSD) are determined and compared to specified limits to verify that precision and bias are acceptable.

12.3.2.1 For analytes that do not lend themselves to spiking, e.g., TSS, the DOC may be performed using quality control samples. For analytes for which spiking is not an option and for which quality control samples are not readily available, the procedure published in 40 CFR Part 136, Appendix A, test methods, is one way to perform this demonstration.

12.3.2.2 The DOC data is also evaluated to ensure that the selectivity of the method is adequate. The laboratory evaluates selectivity through procedures defined in the test method SOPs such as use of dual columns, interference checks, and analysis of required QC samples.

12.3.3 The MDL, LOD, and LOQ are established following the corporate SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* and in the SOP VAL-QS-006, *Determination of Method Detection Limits, Quantitation, and Reporting Limits*.

12.3.3.1 The MDL is used to establish the lower limit of detection, as defined by the USEPA, 40 CFR Part 136, Appendix B. The LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect, and is typically equivalent to the MDL. The LOD defines a range below the LOQ where detections must be reported with the data qualifier "J", indicating the value reported is an estimated value. The LOD is analyte and matrix-specific. No results are reported below the LOD.

12.3.3.2 The LOQ defines the lower limit for an analyte where data may be reported without qualification. For most methods it is established to be no lower than the lowest non-zero calibration standard. On a final analytical report, the LOQ may be labeled as the method quantitation limit (MQL) or practical quantitation limit (PQL).

12.3.4 Laboratory-Developed or Non-Standard Method Validation – All laboratory developed, modified standard methods, or non-standard methods require planned method development and validation following established protocols.

12.4 Ongoing Proficiency

12.4.1 Ongoing DOC's are performed when either an analyst repeats the DOC annually or generates acceptable results when analyzing performance evaluation

samples. The data for the DOC procedure is evaluated by either the section supervisor or the QA Department.

12.4.2 Ongoing demonstrations of capability are documented in the training file of each analyst or maintained by the QA department as a separate document.

12.4.3 LOD verification samples are performed to verify that procedure sensitivity is maintained such that LODs are achievable. Periodic LOQ verification ensures that quantified results are within accepted limits of precision and bias.

13) Measurement Traceability and Calibration

13.1 Measurement quality comes in part from the traceability of standards to certified materials, reagents of known purity, and support equipment accuracy. ALS Environmental addresses these components through the following policies.

13.2 Standards

13.2.1 Commercially available standards are purchased from approved vendors and must be traceable to certified materials. Certificates of analysis (when available) must be retained for all standards.

13.2.2 All standards are logged upon receipt and assigned unique identifiers. Procedures for traceability of standards are documented in SOP VAL-QS-001, *Reagent and Standard Tracking*.

13.2.3 Storage requirements are specified by the manufacturer or in the method SOP. Expiration dates are established as described in the corporate SOP CE-QA012, *Quality of Reagents and Standards*.

13.2.4 Standards/reagents received without a Certificate of Analysis must be tested and approved for use by the Supervisor prior to implementing into operations.

13.3 Chemicals and Gases

13.3.1 All chemicals must be reagent grade or higher. The expiration periods and storage requirements are provided in the method SOPs. Solvents must be HPLC or pesticide residue grade, acids for metals digestions must be trace metal grade, and gases must be high purity or higher.

13.3.2 If other than reagent grade or high purity, the purity of chemicals or gases is specified in the method SOP.

13.3.3 All chemicals must be tested and approved for use by the Department Supervisor prior to implementing into normal department operations.

13.4 Calibration

13.4.1 Balances

13.4.1.1 Balances are calibrated and verified annually by a certified outside vendor.

13.4.1.2 Balance calibration and performance is verified daily utilizing NIST traceable weights within the working range of weights to be determined on each balance.

13.4.1.3 Procedures for traceability of analytical balances are documented in SOP VAL-EQ-001, *Use and Maintenance of Balances*.

13.4.2 Thermometers

13.4.2.1 Thermometers are calibrated annually against a NIST certified reference thermometer or replaced annually.

13.4.2.2 Infrared based measurement systems are calibrated annually against a NIST certified reference thermometer or replaced annually.

13.4.2.3 Procedures for traceability of temperature measurements are documented in SOP VAL-EQ-002, *Thermometer Calibration and Temperature Monitoring*.

13.4.3 Instrument Initial Calibration

13.4.3.1 Initial instrument calibration (and continuing calibration verification) is a critical part of ensuring data of known and documented quality. In general, all initial calibrations are according to method specified criteria and documented in the applicable SOP. The SOP specifies calibration requirements, frequency, acceptance criteria, and requires the use of a second source calibration standard for verification. The following general guidelines must be followed for all multi-point initial calibrations:

- Unless specified otherwise by the method SOP, a minimum of five calibration levels must be used.
- The lowest calibration level must be equal to or less than the PQL. The lowest or highest calibration level can be dropped if non-linear. However, the PQL or UQL must be adjusted accordingly.
- Quantitation of results must be determined from the initial calibration curve (most recent) unless the test method requires the use of the continuing calibration (i.e. quantitation from the CCV).
- Reported results falling below the PQL must be qualified and documented in the case narrative.
- Results greater than the UQL must be diluted or must be considered qualified as "estimated" if reported. If the latter, this must also be documented in the case narrative.

13.4.3.2 Raw data records must be retained to allow reconstruction of instrument specific initial calibrations.

13.4.4 Continuing Instrument Calibration Verification

13.4.4.1 The validity of the initial calibration must be verified prior to sample analysis through analysis of continuing or daily calibration verification (CCV) standards. Method SOPs specify the calibration criteria, frequency, and acceptance limits. The following general guidelines apply to continuing calibration verifications.

- Continuing calibration verification must be performed at the beginning, periodically through the analytical run, and end of each analytical batch except for instances where an internal standard is used.
- For methods employing internal standards, continuing verification must be performed at the beginning of the

analytical batch.
- Continuing calibration verifications must be performed at SOP specified time intervals.
- Continuing calibration verifications must be performed for all analytical systems that have calibration verification requirements.
- Calibration must be verified for each compound, element, or other discrete chemical species.

13.4.4.2 Sufficient raw data records must be retained to reconstruct the continuing calibration verification to the initial calibration.

14) Assuring the Quality of Results

The quality of results is assured through the use of various control and internal data assessment practices. These include use of defined SOPs, sample batch definition, Quality Control (QC) analyses performed in conjunction with all sample analyses, use of established control limits for accuracy and precision, and having established procedures for internal data review and validation. These practices are described in the following sections.

14.1 As discussed in Section 12, SOPs are established to provide details of performing the test method in the laboratory. From the perspective of assuring quality, the use of SOPs ensures a test is performed consistently and promotes data comparability. SOPs include key elements of equipment operation and materials quality, analysis selectivity, calibration, and other key components of data quality, consistency, comparability, and representativeness. SOPs include written procedures for conducting QC analyses, including acceptance criteria and corrective action, as further described in this section.

14.2 Essential Quality Control Procedures

14.2.1 Batching

14.2.1.1 A sample batch is comprised of up to 20 samples of the same matrix and processed on the same working shift. All samples and QC samples within a batch are processed using the same procedures and same reagents or materials. Exceptions required by method or program are included in analytical SOPs.

14.2.1.2 The standard QC samples included in each sample batch include a method blank (MB), laboratory control sample (LCS), and matrix spike/matrix spike duplicate (MS/MSD). For certain analyses, sample duplicates (DUP) may be used in place of an MSD for a measure of precision. Results for QC samples falling outside defined limits indicate that analytical data for samples within the same batch may be suspect. In general, samples must be re-processed (re-extraction/digestion and analysis) or that the data must be qualified and narrated. Any exceptions to this or circumstances requiring special treatment of data are located in the individual method SOPs.

14.2.2 Method Blank (MB)

14.2.2.1 The MB serves as the negative control for the sample batch. The MB is carried through all analytical steps which are performed on samples. Results for the MB should be less than the MDL and must be less than the PQL.

14.2.2.2 When blank contamination is determined, the cause must be investigated and corrective action taken to eliminate the problem.

14.2.2.3 All samples associated with blank contamination must be reprocessed (preparation and analysis). Exceptions to this can be found in the corrective action section of the individual test SOPs.

14.2.2.4 Data for samples that cannot be reprocessed must be appropriately qualified or flagged when reported. Samples that are blank corrected must be narrated.

14.2.3 Laboratory Control Samples (LCS)

14.2.3.1 The Laboratory Control Sample (or Blank Spike) serves as the positive control for the sample batch. The LCS is carried through all analytical steps which are performed on samples. Results are used for monitoring accuracy of results for the sample batch. The LCS is prepared from an analyte free matrix with a known amount of analyte added. An LCS Duplicate (LCSD) may be performed if insufficient sample is available for MSD or DUP analysis.

14.2.3.2 An LCS must be analyzed at the frequency specified by the SOP and acceptance criteria must be defined or referenced within the analytical SOP. Example calculations are included in each of the method SOPs.

14.2.3.3 The LCS result must meet the defined criteria. If accuracy is not achieved, the cause must be investigated and corrective action taken to eliminate the problem.

14.2.3.4 All samples associated with a failed LCS are reprocessed (preparation and analysis). Any exceptions to this can be found in the corrective action section of the individual method SOPs.

14.2.3.5 Samples associated with a failed LCS that cannot be reprocessed must be appropriately qualified or flagged when reported.

14.2.4 Matrix Spike (MS)

14.2.4.1 Matrix spikes are environmental samples with a known amount of analyte added. These QC samples are used to assess the effect of the matrix on method performance. Matrix Spike Duplicates (MSD) are performed to assess the precision of the analysis. The MS/MSD is/are carried through all analytical steps which are performed on the parent sample used for spiking.

14.2.4.2 Matrix spike (and MSD) samples must be analyzed at the frequency specified by the SOP and acceptance criteria must be defined or referenced within the analytical SOP. Example calculations are included in each of the method SOPs.

14.2.4.3 Accuracy of matrix spike samples should meet the defined criteria. All samples associated with failed matrix spike results must be reprocessed (preparation and analysis) if the LCS also did not meet acceptance criteria. Exceptions to this can be found in the corrective action section of the individual method SOPs.

14.2.4.4 Samples associated with failed matrix spike results that cannot be

reprocessed must be appropriately qualified or flagged when reported.

14.2.5 Surrogate Spikes

14.2.5.1 Surrogates are substances with chemical properties and behaviors similar to the analytes of interest that are used to assess method performance in individual samples. Where applicable, the use of surrogates is specified in the reference method and SOP, and is generally used for organics analyses.

14.2.5.2 Surrogates are added to all samples and quality control samples prior to sample preparation. Surrogates are also included in all initial and continuing calibration standards.

14.2.5.3 Recovery criteria is defined or referenced within the analytical SOP. Example calculations are included in each of the method SOPs.

14.2.5.4 For MB and LCS analyses the surrogate recovery must fall within defined criteria. The surrogate recovery in field samples must fall within defined criteria unless bias resulting from matrix interference or dilution is demonstrated.

14.2.5.5 All samples associated with failed surrogate recovery in the MB and/or LCS must be reprocessed (preparation and/or analysis). Refer to the method SOPs for specific corrective action procedures including any special circumstances for reporting of data.

14.3 Control Limits

14.3.1 Accuracy and precision acceptance criteria (i.e. control limits) are based upon method specifications, program or project specifications, control charting, or a combination thereof. Procedures for control limits are given in the corporate SOP CE-QA009, *Control Limits*.

14.3.2 Accuracy and precision criteria are reviewed and updated at least annually, or as necessary based on significant changes made to the analysis, or as a result of internal audit findings. Since control limits may therefore change intermittently, a current list of control limits may be obtained from the laboratory upon request.

14.4 Data Review

14.4.1 The laboratory reviews all data generated in the laboratory for compliance with method, laboratory, and client requirements. All data review is documented through the use of data checklists. Procedures for data review are documented in SOP VAL-QS-009, *Data Review and Validation*.

14.4.2 The primary analyst reviews 100% of all raw data for acceptability of quality control measures and accuracy of the final result(s).

14.4.3 A peer analyst reviews 100% of all raw data including manual data entry, data calculations, and electronic transfers of data.

14.4.4 Final reports are reviewed by the project manager for comparison to historical data and client specification prior to release.

14.4.5 Departures from specified policy and procedure are documented on data quality checklists and narrated.

14.4.6 The quality assurance department must perform an independent review of raw data generation, quantitative calculations, and method performance through a combination of internal audits, client inquiries, non-conformance reports, and/or data package compilation.

14.4.7 Additional data quality indicators (DQI) that may be used to evaluate the analytical data produced are listed below. Acceptance criteria for these items must be agreed upon by both the laboratory and client or end user of the data on a project-by-project basis.

14.4.7.1 Comparability: a qualitative expression of the measure of confidence that two or more data sets may contribute to a common analysis. Comparability is achieved by utilizing the same preparation and/or analysis methods for the set of data being evaluated.

14.4.7.2 Completeness: a measure of the amount of valid data obtained from a measurement system, expressed as a percentage of the number of valid measurements that have been collected. Typical completeness goals are 90% for soil samples and 95% for water samples.

14.4.7.3 Representativeness: the measure of the degree to which data suitably represent a characteristic of a population, a process condition, or an environmental condition. Representativeness is measured by comparing data from the same samples, sample location, or sampling grid. It takes into account other DQIs, such as bias and precision, to determine if the data is a good estimate of the population being sampled.

14.5 Estimation of Uncertainty – An estimation of uncertainty consists of the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, and weighing variability. To the degree where the laboratory has a control over these processes, the laboratory estimates uncertainty using the standard deviation calculated from routine Laboratory Control Samples (LCS). Procedures for estimating uncertainty are documented in SOP VAL-QS-022, *Measurement Uncertainty*.

14.6 Data Control

14.6.1 Procedures for the validation of software applications associated with data acquisition, calculation, and reporting are document in SOP VAL-IT-001, *LIMS Data Integrity and Verification*.

14.6.2 Procedures for ensuring that reported data are free from transcription and calculation errors are documented in SOP VAL-QS-009, *Data Reduction, Review, and Validation*.

14.6.3 Procedures for ensuring proper batch QC data evaluation are documented in SOP VAL-QS-020, *Batch QC Data Evaluation*.

14.6.4 Procedures for manual integration are documented in SOP VAL-QS-016, *Manual Integration Policy*.

14.6.5 Procedures for ensuring computer and software validation as well as data

integrity, confidentiality, and security are documented in SOP VAL-IT-002, *Computer Software Installation and Maintenance*.

15) Control of Non-Conforming Environmental Testing Work

15.1 Non-conforming work is defined as work that does not meet specified acceptance criteria or project requirements. Non-conformances can include unacceptable quality control results, departures from SOPs, or method modifications. The policy for control of non-conforming work is to identify the non-conformance, determine its impact relative to data integrity/quality policy, and take appropriate action.

15.2 All employees have the authority to stop work on samples when any aspect of the process does not conform to laboratory requirements. Requests for departures from standard laboratory procedures are reviewed, approved, and documented by the Laboratory Director, Operations Manager, Department Supervisor(s), or QA Manager. The following criteria apply to the control of non-conforming work.

15.3 Evaluation & Management of Non-Conforming Work

15.3.1 Guidelines for evaluating batch QC parameters are documented in SOP VAL-QS-020, *Batch QC Data Evaluation*.

15.3.2 Specific information is documented in each applicable analytical SOP as to QC criteria, corrective action, and acceptable deviations.

15.3.3 Procedures for the management of non-conforming work are detailed in SOP VAL-GEN-005, *Departures from Documented Procedures*.

15.3.4 The laboratory must evaluate the significance of all non-conformances. If data integrity issues are indicated or suspected, corrective action must be taken prior to reporting or continuation of analytical work.

15.3.5 If non-conformances are discovered after work completion and reporting, the client must be notified of the impacted data.

15.4 The laboratory allows the release of non-conforming data only with approval by the appropriate Department Supervisor and Project Manager (or their designee) on a case-by-case basis. Such releases are contingent upon contractual or regulatory specifications.

15.4.1 Work associated with contractual specifications or analytical SOPs that document “no deviations allowed” must achieve criteria. In such cases, allowing departures from analytical data control are not permitted.

15.4.2 When sample data or QC results which include non-conforming data are to be reported, information on the circumstances should be provided in a report narrative. Data must be appropriately qualified or flagged when reported. A list of standard data qualifiers is listed in Appendix H.

16) Corrective Action, Preventive Action, and Improvement

16.1 Corrective action is the action taken to eliminate the cause(s) of an existing nonconformity, defect, or other undesirable situation in order to prevent its recurrence. Deficiencies cited in external assessments, internal quality audits, data reviews, complaint resolution, and/or managerial reviews are documented and require corrective action. Corrective action procedures for failed acceptance criteria relative to initial and continuing calibrations, quality control samples, and instrument

performance are documented in the analytical SOPs.

16.2 Procedure for Corrective Action

16.2.1 Procedures and guidelines for the corrective action process are specified in SOP VAL-QS-003, *Non-Conformance & Corrective Action Reporting*.

16.2.2 The Laboratory Supervisor, QA Manager, and/or Laboratory Director are responsible for initiating applicable corrective actions.

16.2.3 All deficiencies must be investigated. A corrective action plan must be developed, documented and implemented if the deficiency is determined to be procedural or systemic.

16.3 Selection/Implementation of Corrective Actions

16.3.1 Once a non-conformance is noted, the event must be reviewed to determine if it is indicative of a procedural or systemic deficiency resulting from a primary cause. Root cause analysis principles are encouraged and should be used where suitable.

16.3.2 If a procedural or systemic deficiency is indicated, a Nonconformance and Corrective Action Report (NCAR) must be initiated and the root cause identified.

16.3.3 In the event of uncertainty regarding the best approach for analysis/correction of the cause, the Department Supervisor, QA Manager, or Laboratory Director will collaborate on the best course of action.

16.4 Verification and Monitoring of Corrective Action

16.4.1 The Department Supervisor(s) ensures that corrective actions are discharged within the agreed upon time frame. The QA Manager also verifies that implementation and documentation of the corrective action is complete.

16.4.2 To assure that the corrective actions are effective and remain implemented, the ongoing verification of corrective actions may be included in internal audits.

16.5 Preventive Action

16.5.1 Preventive action aims at minimizing or eliminating occurrences of data quality anomalies or other laboratory issues before they occur through procedural improvement, scheduled maintenance, and data review. The following preventive action measures are taken at the laboratory.

- Review of QC data to identify quality trends
- Regularly scheduled staff quality meetings
- Annual budget reviews
- Annual managerial reviews
- Running computer system modifications in tandem with the old system to assure at least one working system

16.5.2 All employees have the authority to recommend preventative action procedures. Management is responsible for reviewing all preventative action recommendations and implementing same where deemed appropriate.

16.5.3 Preventive maintenance of equipment and instrumentation is an important component of preventive action. Routine instrument preventive maintenance is included in SOPs and outlined in the following table.

16.6 Improvement

The laboratory strives to continually improve operations, processes, and quality systems. Identifying areas for improvement, or larger improvement initiatives, is a result of monitoring the certain aspects of the laboratory's management and quality system. This includes annual managerial reviews, evaluating on-going performance indicators and balanced scorecards, internal and external audit results, review of data, and the effectiveness of corrective and preventive actions. Improvement is also a consideration in periodic budgeting and planning processes.

Equipment and Instrumentation Preventive Maintenance

Instrument	Activity	Frequency	Service
Refrigerators and Coolers	Record temperature	Daily	
	Clean coils	Annually	
Vacuum Pumps	Clean and change pump oil	Every 6 months or as needed	
Fume Hoods	Face velocity measured	Quarterly	Service Contract
	Sash operation	As needed	
	Certified	Annually	
Ovens	Clean	As needed or if temperature outside limits	
Incubators	Clean	As needed or if temperature outside limits	
Analytical Balances	Check alignment	Before daily use	Field Service
	Check calibration	Before daily use	
	Clean pans and compartment	After every use	
	Certified	Annually	
Lachat	Check sample tubing	Daily	
	Clean flow cell	As needed	
Spectrophotometer	Replace lamp	Annually	Field Service
	Replace Paper	As needed	
ICP-MS	Check Argon supply	Daily	Service Contract
	Check sample tubing	Daily	
	Clean cones	As needed	
Ion Chromatograph	Replace Columns	As needed	Service Contract
Printers	Change toner	As needed	
	Clean printer internal parts	As needed	
	Change pick-up roller wheels	As needed	
Copier	Change toner	As needed	
	Routine maintenance	As needed	

17) Control of Records

17.1 Records are a subset of documents such as daily temperatures logs, instrumental run logs, analytical spreadsheets, and computer printouts that allow for the historical reconstruction of laboratory activities relating to sample handling, processing, and analysis. Records can be on various forms of media including electronic and hard copy. Record management and archival are conducted according to the following criteria.

17.2 Records Management and Storage

17.2.1 Guidelines for the management of records are documented in SOP VAL-QS-014, *Laboratory Record Procedures*.

17.2.2 Guidelines for archiving records are described in SOP VAL-QS-011, *Record Archival*. Archived records are indexed to include the storage identification, archived material identification, and date range of archived material.

17.3 Procedures for evidentiary sample custody (if applicable) are documented in SOP VAL-SM-001, *Sample Receipt & Log-In Procedures*.

18) Audits

18.1 Audits assess the laboratory performance and verify compliance with accreditation, certification, project, and method requirements. Audits also provide management with an on-going assessment of the quality system. They are also instrumental in identifying areas where improvement in the quality system can increase the reliability of data.

18.2 Audits are of four main types: internal, external, performance, and system. In the event that analytical anomalies are identified upon completion of any audit, clients must be notified within two working days (48 hours) of any deviations that cast doubt on the accuracy of previously issued reports.

18.3 Internal Audits

18.3.1 Internal audits are conducted by the QA department throughout the year and must encompass all areas of the laboratory operation. Internal audit procedures are documented in SOP VAL-QS-012, *Internal Audits*. These audits primarily focus on technical areas, method compliance, and quality systems compliance within day-to-day operations.

18.3.2 Internal audits are also periodically conducted by the Corporate QA Manager or designee. These audits focus primarily on the quality program implementation and quality systems and evaluate these against established accreditation, program, and internal quality system standards.

18.3.3 Internal audits are regularly reported to management. Any deficiencies identified are corrected and corrective action verified by the QA Manager.

18.4 External Audits

18.4.1 Most commonly, external audits and assessments of the laboratory are performed by certification/accreditation bodies to evaluate compliance with the applicable program or Standard. Audits may also be performed by clients, or their representatives, in support of project needs. It is the laboratory's policy to encourage, cooperate and assist with all external audits, whether performed by clients or an accrediting authority.

18.4.2 Management must ensure that all applicable areas of the laboratory are accessible to auditors and that the appropriate personnel are available.

18.4.3 The QA department must ensure that any noted deficiencies or findings requiring corrective actions are adequately addressed. This normally involves collaboration with the Supervisor(s), Laboratory Director, and applicable laboratory staff. The QA department assigns appropriate corrective action(s) to each item and tracks actions to closure. This includes handling all pertinent correspondence with the auditing entity and providing documentation.

18.5 Proficiency Test Samples

The laboratory participates in proficiency test (PT) programs approximately every six (6) months. Results are evaluated independently by an approved provider.

18.5.1 Samples submitted for Proficiency Testing must be treated as typical samples in the normal production process.

18.5.2 The laboratory cannot communicate with other laboratories and does not attempt to obtain the assigned values of any PT sample from the provider.

18.5.3 The laboratory institutes corrective action procedures for failed PT samples and the root cause is identified. Root cause is the condition or event that, if corrected or eliminated, would prevent the recurrence of the deficiency.

18.6 System Audits

18.6.1 A quality system evaluation must be performed annually by the QA Staff.

18.6.2 Findings from this evaluation must be submitted to the Laboratory Director and must include findings from internal audits, external audits, performance evaluation results, and client assessments.

19) Management Review

19.1 Top management reviews the management system on an annual basis and maintains records of review findings and actions. The following Management Review topics are reviewed to ensure their suitability and effectiveness:

- the suitability of policies and procedures;
- reports from managerial and supervisory personnel;
- the outcome of recent internal audits;
- corrective and preventive actions;
- assessments by external bodies;
- the results of interlaboratory comparisons or proficiency tests;
- changes in the volume and type of the work;
- customer feedback;
- complaints;
- recommendations for improvement;
- other relevant factors, such as quality control activities, resources, and staff training.

19.2 Specific procedures regarding the Management Review are described in the SOP VAL-QS-017, *QA Management Review* and in the corporate SOP CE-QA005, *Laboratory Management Review*. Findings and follow-up actions from management reviews are recorded. Management will determine appropriate completion dates for action items and ensure they are completed within the agreed upon time frame.

20) Personnel

20.1 All personnel are responsible for complying with all quality and data integrity policies and procedures that are relevant to their area of responsibility. All personnel who are involved in activities related to sample analysis, evaluation of results or who sign test reports, must demonstrate competence in their area of responsibility. Appropriate supervision is given to any personnel in training and the trainer is accountable for the quality of the trainees work. Personnel are qualified to perform the tasks they are responsible for based on education, training, experience and demonstrated skills as required for their area of responsibility.

20.2 The laboratory provides goals for education, training and skills of laboratory staff. These goals are outlined in job descriptions. Training needs are identified at the time of employment and when personnel are moved to a new position or new responsibilities are added to their job responsibilities. Ongoing training, as needed, is also provided to personnel in their current jobs. The effectiveness of the training must be evaluated before the training is considered complete.

20.3 Job descriptions are available for all positions that manage, perform, or verify work affecting data quality. An overview of top management's responsibilities is included in Section 3 – "Management".

20.4 Job descriptions include the specific tasks, minimum education and qualifications, skills, and experience required for each position Provide overview using components from the TNI Standard. Include how job descriptions are used.

20.5 General Training

20.5.1 Employees are trained and competent in their assigned tasks before they contribute to functions that can affect data quality. It is management's responsibility to assure personnel are trained.

20.5.2 All personnel must be appropriately trained and demonstrate competency in their assigned tasks before they can contribute independently to functions that can affect data quality. Training records are used to document management's approval of personnel competency.

20.5.3 Procedures for employee training, including initial training, demonstration of competency, and ongoing training are described in SOP VAL-QS-013, *Employee Training*.

20.6 Data Integrity and Ethics

20.6.1 Data integrity is the result of multiple processes, as described in SOP VAL-QS-015, *Data Integrity System*. These processes assist in the production of valid data of known and documented quality. Data integrity and ethics procedures in the laboratory include documented data integrity procedures, initial training, on-going training, and signed/dated document of understanding for all laboratory employees. Department supervisors uphold data integrity by supporting integrated QA procedures, providing staff training, approving training results, and continuously monitoring their department's performance.

20.6.2 Employees are required to understand, through initial and ongoing ethics training, that any infractions of the laboratory data integrity procedures will result in an investigation and could lead to immediate termination, or civil/criminal prosecution. Guidelines for laboratory ethics, accountability, and responsibility are documented in SOP VAL-GEN-002, *Laboratory Ethics*.

20.6.3 The mechanism for confidential reporting of ethics and data integrity issues is comprised of: 1) unrestricted access to senior management, 2) a documented policy that personnel must not be treated unfairly for reporting any instances of ethics or data integrity breaches, and 3) anonymous reporting. Any potential data integrity issue is handled confidentially until a follow-up evaluation, full investigation, or other appropriate action has been completed and the issues clarified. Inappropriate activities are documented, including disciplinary actions, corrective actions, and notifications of clients, if applicable.

21) Reporting of Results

21.1 The result of each test must be reported accurately, clearly, unambiguously, and objectively. Data are reported without qualification if they are: 1) greater than the practical quantitation limit, 2) lower than the upper quantitation limit, and 3) without compromised sample or method integrity.

21.2 Report formats are designed to meet the client's data reporting requirements and accurately report each test performed and to minimize potential for misunderstanding or misuse. Test reports may include various levels of supplemental information such as QC data reporting or inclusion of raw data to produce validation-level reports, as requested by the client.

21.3 Procedures for the producing and formatting of test results are documented in SOP VAL-ADM-005, *Report Formatting*.

21.4 Reporting Results from Subcontractors

21.4.1 Test results obtained from tests performed by subcontractors outside the ALS network must be clearly identified on the test report.

21.4.2 Test results from subcontractors outside the ALS network must be reported in writing or electronically with a copy of the subcontractor's report attached.

21.5 All test results transmitted by telephone, fax, telex, e-mail, or other electronic means must comply with the requirements of SOP VAL-GEN-004, *Client Confidentiality*.

21.6 Amendments to a test report after it has been issued must be in accordance with the specifications listed in SOP VAL-ADM-005, *Report Formatting*.

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
03.0	11/17/2017	Laura Cooper	Updated approved signatories (Sec. 4.7.4)
03.0	11/17/2017	Laura Cooper	Updated Fume Hood velocity check frequency (Sec. 16.6)
03.0	11/17/2017	Laura Cooper	Updated App G and I
03.0	11/17/2017	Laura Cooper	Updated Standard Methods reference
04.0	7/17/2018	Chad Stoike	Added IC instrumentation.
05.0	3/8/2019	Laura Cooper	Added Microbiology information (App. F, G)
05.0	3/8/2019	Laura Cooper	Updated Appendix B, I
06.0	3/31/2020	Laura Cooper	Updated section 16.1, Appendix B, G, and J
07.0	3/31/2021	Laura Cooper	Updated Appendix B, G, and J

23) References for Quality System Standards, External Documents, Manuals, and Test Procedures

The following list represents key references for the laboratory quality program and systems. Also listed are the references for test methods used by the laboratory. SOPs referenced throughout this QAM are listed in Appendix G. Other normative documents related to the QA program implementation are included in Appendix I.

- TNI Standard – Environmental Laboratory Sector, Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis,* EL-V1-2016.
- International Standard – *General Requirements for the Competence of Testing and Calibration Laboratories,* ISO/IEC 17025:2005(E)
- Selected USEPA Approved Methods, 40 CFR, Part 136, Table 1B; including changes incorporated in the *Methods Update Rule (MUR) published* August 27, 2017.
- USEPA Methods published in Appendix A, B and C of 40 CFR, Part 136.
- *Standard Methods for the Examination of Water and Wastewater*, *Online Edition 2011*.
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, through Updates III (December 1996) and Update IV (February 2007), and new published methods online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.
- Selected APHA, AWWA, and ASTM methods.

APPENDIX A – Glossary

DEFINITIONS

Accreditation Body: The territorial, state or federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.
Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT): Analytes for which a laboratory is required to successfully analyze a PT sample in order to obtain or maintain accreditation, collectively defined as: matrix, technology/method, analyte.
Primary Accreditation Body (Primary AB): The accreditation or certification body responsible for assessing a laboratory's total quality system.
Proficiency Testing (PT): A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation/Certification: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of up to 20 environmental samples of the same matrix prepared or analyzed as a group in the same 24 hour period. An analytical batch can include prepared samples originating from various preparation batches and can exceed 20 samples.
Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.
Chain of Custody: A record that documents the possession of a sample from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses.
Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method.
Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.
Demonstration of Capability: A procedure to establish the ability of the analyst to generate

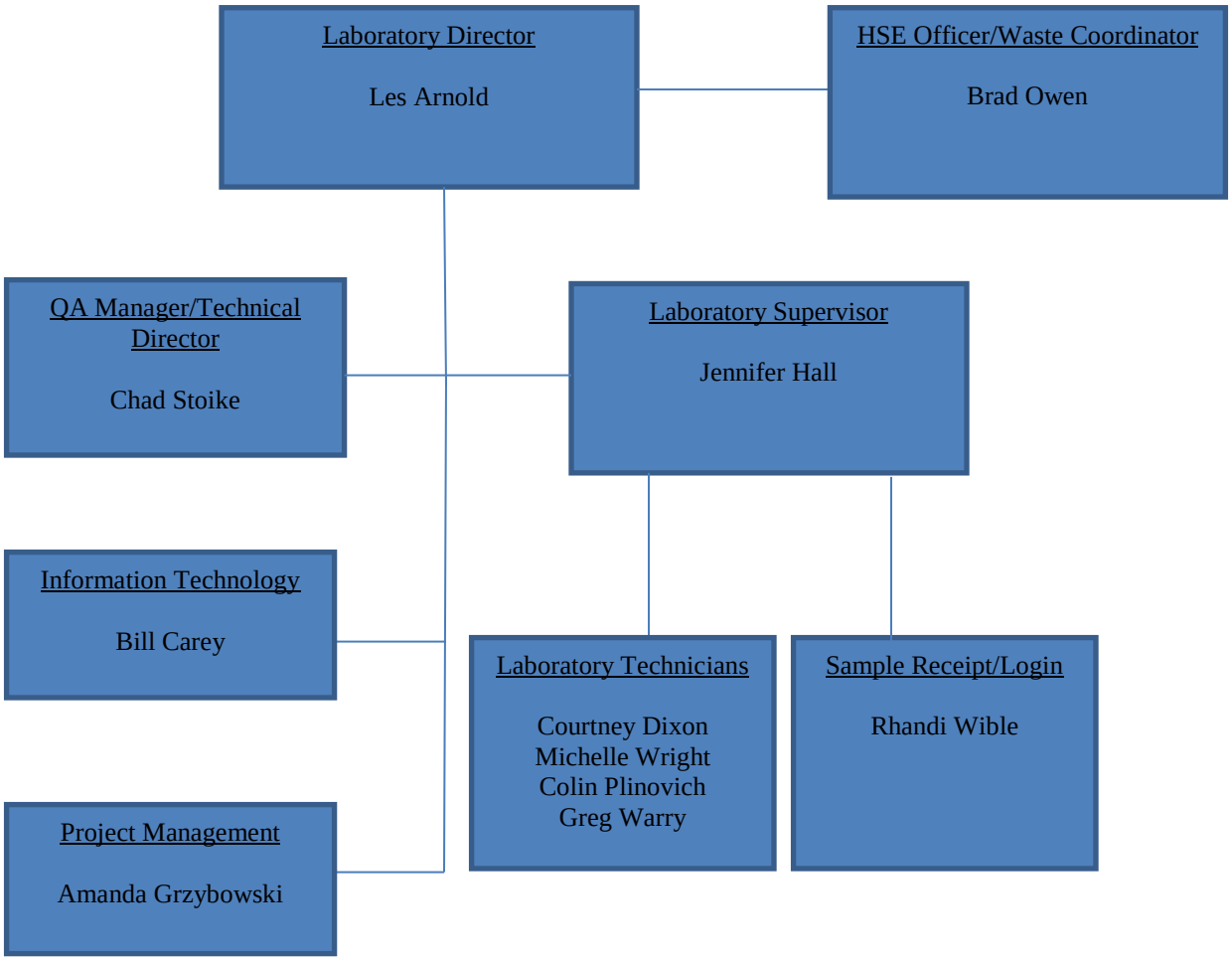
analytical results of acceptable accuracy and precision.
Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Holding Time: The maximum time that can elapse between two specified activities, typically between sampling and the beginning of preparation or analysis.
Laboratory Control Sample (or Blank Spike): A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure, and used to establish analytical control of the batch.
Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility.
Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
Matrix: The substrate of a test sample.
Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.
Quality Assurance: A system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality.
Raw Data: The analytical output, records, and documentation generated during sampling and analysis. This includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC results, chromatograms, instrument outputs, and handwritten records.
Reference Material: Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location.
Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target

analyte or parameter within the measurement system.
Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard Operating Procedures (SOPs): A written document that details the process for conducting an operation, analysis, or action, with thoroughly prescribed techniques and steps.
Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In calibration, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

ACRONYMS

AB	Accrediting Body
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
BLK	Blank
°C	degrees Celsius
ICAL	Initial Calibration
CAS	Chemical Abstract Service
CCV	Continuing calibration verification
COC	Chain of custody
DOC	Demonstration of Capability
EPA	Environmental Protection Agency
GC/MS	Gas chromatography/mass spectrometry
ICP-MS	Inductively coupled plasma-mass spectrometry
ICV	Initial calibration verification
IDC	Initial Demonstration of Capability
ISO/IEC	International Organization for Standardization/International Electrochemical Commission
LCS/LFB	Laboratory control sample/Laboratory fortified blank
MDL	Method Detection Limit
mg/Kg	milligrams per kilogram
mg/L	milligrams per liter
MRL	Minimum Reporting Level
MS	Matrix spike (MSD - Matrix Spike Duplicate)
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
ODC	Ongoing Demonstration of Capability
PQL	Practical Quantitation Limit
PT	Proficiency Test(ing)
QA	Quality Assurance
QC	Quality Control
QAM	Quality Assurance Manual
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
TNI	The NELAC institute
ug/L	micrograms per liter
VOC	Volatile organic compound

APPENDIX B – Organization Charts and Key Personnel Qualifications



Les Arnold

3352 128th Avenue | Holland, MI 49524 | +1 616 399 6070

ALS Environmental

Director of Operations, Eastern USA

Oversees all of the ALS Environmental laboratories in the Eastern United States from an operations standpoint, and is responsible for strategic planning, financial performance and business development for all locations. He is experienced in all facets of laboratory operations including laboratory analysis, field services, project management, customer service, and sales & marketing.

Education

Indiana Vocational Technical College, Cary, Indiana
Environmental Technology

Previous Experience

ALS Environmental — **Laboratory Director, '01 – '11**
Holland, MI
Responsible for managing all facets of laboratory operations including laboratory analysis, field services, project management, customer service, and sales & marketing. Also responsible for strategic planning, financial performance, and business development.

Severn Trent Laboratories — **Laboratory Manager, '94- '01**
Valparaiso, IN
Responsible for managing all facets of laboratory operations including laboratory analysis, field services, project management, customer service, and sales & marketing.

Severn Trent Laboratories — **Sr. Project Manager, '88- '94**
Valparaiso, IN
Primary responsibilities included technical project management and client service. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory.

Severn Trent Laboratories — **Field Services Manager, '83- '88**
Valparaiso, IN

Chad Stoike

3352 128th Avenue | Holland, MI 49424 | +1 616 399 6070

QA Manager / Technical Director

2014 – Present

Responsible for the implementation and management of the laboratory's quality assurance program including current NELAP and state accreditations, the addition of new or expanded accreditations, review/maintenance of performance data, employee training, and customer inquiries. Review, recommend, and validate emerging analyses and/or technologies for laboratory utilization.

Previous Experience

ALS Environmental
Holland, MI
Quality Assurance Assistant, 2012-2014

Assisted in the implementation and management of the laboratory's quality assurance program including NELAP and state accreditations, the addition of new or expanded accreditations, review/maintenance of performance data, employee training records, and customer inquiries.

ALS Environmental
Holland, MI
Metals Department Supervisor, 2007-2012

Responsible for employee and workload management, acquisition, processing and validation of legally defensible data, ICPMS, CVAA and CVAF analysis and maintenance, while promoting strict adherence to prescribed methodologies within a safety conscious environment.

e-Lab Analytical
Holland, MI
Quality Assurance Assistant, 2006-2007

Maintained Standard Operating Procedure documentation. Assisted in the internal quality system review process. Performed an intensive data review in support of the laboratory compliance program.

e-Lab Analytical
Holland, MI
Wet Chemistry Technician, 2005-2006

Performed general chemistry techniques in accordance with laboratory Standard Operating Procedures. Technologies include Ion Chromatography, Flow-Injection Analysis, Solid Phase Extraction, titrations, distillations, and gravimetric determinations.

Education

Grand Valley State University – Allendale, MI
B.S. - Chemistry, 2005, Honors Graduate

Jennifer Hall

2400 Cumberland Drive | Valparaiso, IN 46383 | +1 219 299 8127

Laboratory Supervisor

2015 - Present

Responsible for all operations for the Valparaiso, IN environmental laboratory including laboratory analysis, technical direction, human resources, employee training, method development, and laboratory safety.

Previous Experience

TestAmerica Laboratories
Valparaiso, IN
Wet Chemistry Analyst, 1995-2000

Main responsibilities included: being primary analyst for Cyanides, Ammonias, TKN, Phenols, Phosphates, Nitrate/Nitrite. Also performed basic titrations and other qualitative tests such as TSS, TVS, Bulk Density, and BOD/CBOD. Have extensive experience with running and troubleshooting Lachats, OI-analyticals, and Systea discrete analyzers. Also have 10 years' experience running a Dionex IC for chloride, fluoride, orthophosphate, nitrate/nitrite, sulfate, and bromide.

TestAmerica Laboratories
Valparaiso, IN
Wet Chemistry Supervisor, 2000-2005

Responsible for the operation and management of the Wet Chemistry laboratory. Main functions included supervision and training of personnel, formulation of standard operating procedures, quality control and final approval of all inorganic laboratory data. Also responsible for scheduling analysis to ensure all hold times and due dates were meet. Position was relinquished due to an opportunity to learn GC/MS in organics department.

TestAmerica Laboratories
Valparaiso, IN
Microbiology Department 1997-2013

Analyzed drinking waters, ground waters and swimming pools for total coliform and E-coli.

TestAmerica Laboratories
Valparaiso, IN
Organics Chemist 2005-2013

Functions included operation, calibration, and maintenance of GC and GC/MS instrumentation for analysis of volatile and semi-volatile organics by EPA methods.

Education

Purdue University,
West Lafayette, IN
B.S. Environmental Science
1993

APPENDIX C – Ethics and Data Integrity Employee Agreement

ETHICS AND DATA INTEGRITY AGREEMENT

I state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at ALS.

I agree that in the performance of my duties at ALS:

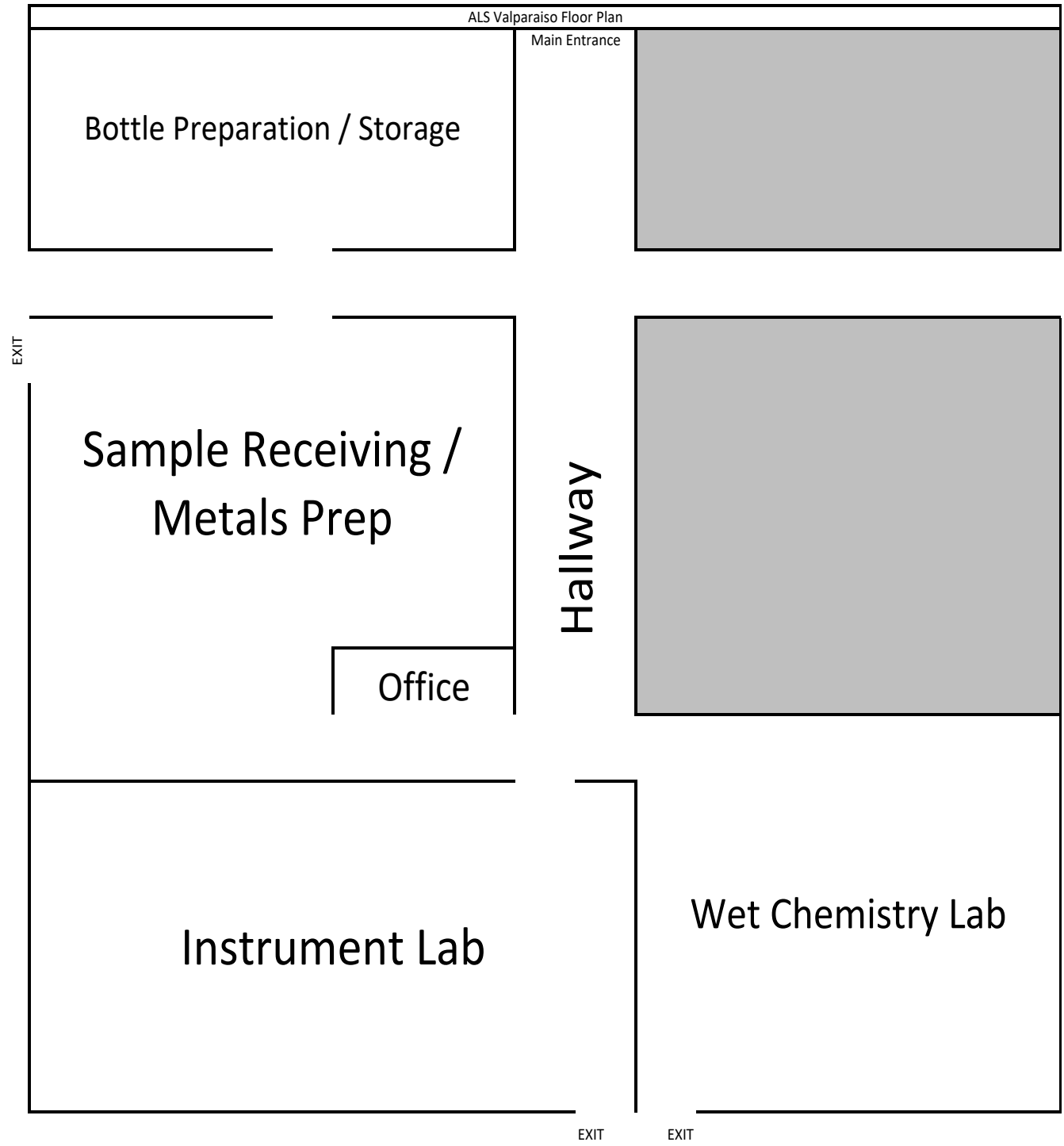
1. I shall not intentionally report data values that are not the actual values obtained;
2. I shall not intentionally report the dates, times and method citations of data analyses that are not the actual dates, times and method citations of analyses;
3. I shall not intentionally represent another individual's work as my own;
4. I shall not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operating Procedures, or as defined by company policy.
5. I agree to inform ALS of any accidental or intentional reporting of non-authentic data by other employees.
6. I have read this ethics and data integrity agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination.
7. I agree to adhere to the following protocols and principals of ethical conduct in my work at ALS. All work assigned to me will be performed using ALS approved methods and procedures and in compliance with the quality assurance protocols defined in the ALS Quality System.
8. I will not intentionally falsify nor improperly manipulate any sample or QC data in any manner. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to ALS. All such modifications and their justification will be clearly and thoroughly documented in the raw data and appropriate laboratory record, and will include my initials or signature and the date.
9. I will not make false statements to, or seek to otherwise deceive ALS staff, managers or clients. I will not knowingly, through acts of commission, omission, erasure or destruction, improperly report any test results or conclusions, be they for client samples, QC samples, or standards.
10. I will not condone any accidental or intentional reporting of unauthentic data by other ALS staff and will immediately report such occurrences to my Supervisor, Lab Director, Quality Assurance Manager, or Human Resources. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.
11. If a supervisor, manager, director or other member of the ALS leadership group requests me to engage in or perform an activity that I feel is compromising data validity or defensibility, I have the right to not comply with the request. I also have the right to appeal this action through an ALS local Quality Staff, Corporate Quality Assurance or Human Resources.
12. I understand that if my job includes supervisory responsibilities, I will not instruct, request or direct any subordinate to perform any unethical or non-defensible laboratory practice. Nor will I discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.
13. I understand that employees who report violations of this policy will be kept free from intimidation and recrimination arising from such reporting.

I have read, and understand the above policy and realize that failure to adhere to it may result in disciplinary action, up to and including termination. Compliance with this policy will be strictly enforced with all personnel employed by the company.

Employee Name _____ Signature _____

ALS Location_____ Date _____

APPENDIX D – Laboratory Floor Plan



APPENDIX E – Analytical Equipment

ITEM	MODEL #	SERIAL #
METALS EQUIPMENT:		
Agilent ICPMS	7900	JP14240281
CETAC ASX-520 Autosampler	ASX-520	US1214134A520
CEM MARS6 Microwave	910900	MJ5211
WET CHEMISTRY:		
Lachat Flow Injection Analyzer	QC8500	150200001822
CETAC ASX-260 Autosampler	ASX-260	011525A260
Thermo Spectrophotometer	Genysys 20	3SGT021002
Binder Laboratory Oven	ED 115UL	14-10478
Ohaus Balance	PA224	B444194975
Ohaus Balance	PA124	B444194972
VWR BOD Incubator	10753-894	300026519
HACH LDO Meter	HQ 40D	150500000772
VWR SYMPHONY pH Meter	B30PCI	15043S0023
Ion Chromatograph	Dionex Aquion	18010559

APPENDIX F – Containers, Preservation and Holding Times

Parameter	Containers [1]	Preservative	Holding Time [2]
Ammonia as N	P, G – 500 mL	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis)
Biological Oxygen Demand (BOD)	P, G – 1000 mL	>0°C to ≤6°C	48 hours (collection to analysis)
(Carbonaceous) Biological Oxygen Demand (CBOD)	P, G – 1000 mL	>0°C to ≤6°C	48 hours (collection to analysis)
Chemical Oxygen Demand (COD)	P, G – 500 mL	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis)
Cyanide (Total and Amenable to Chlorination)	P, G - 1000 mL	>0°C to ≤6°C; NaOH to pH>12; 0.6g ascorbic acid	14 days (collection to analysis)
Cyanide (Total or Reactive) / Soil	P, G – 100 g in 250-ml wm bottle.	>0°C to ≤6°C	14 days (collection to analysis)
Nitrate as N	P, G – 250 mL	>0°C to ≤6°C	48 hours (collection to analysis)
Nitrate-Nitrite as N	P, G – 250 mL	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis)
Nitrite as N	P, G – 250 mL	>0°C to ≤6°C	48 hours (collection to analysis)
Oil and Grease	G – 1000 mL wm	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis
pH (hydrogen ion)	P, G – 250 mL	None required	analyze immediately
(Total) Phenols (wet method)	G / amber – 1000 mL	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis)
(*ortho*-) Phosphate	P, G – 250 mL	Filter immediately; >0°C to ≤6°C	48 hours (collection to analysis)
(Total) Phosphate	P, G – 250 mL	>0°C to ≤6°C; H_2SO_4 to pH<2	28 days (collection to analysis
Residue (Suspended Solids) (TSS)	P, G – 500 mL	>0°C to ≤6°C	7 days (collection to analysis)
Total Coliform, E. Coli	120mL IDEXX vessel	Sodium thiosulfate >0°C to ≤6°C after 1 hr from collection	8 hours (non-potable water) 30 hours (potable water
Chromium VI SM 3500 Cr D/SW846-7196A/7199	P, G – 250 mL	>0°C to ≤6°C	24 hours (collection to analysis)
Chromium VI EPA 218.6	P, G – 250 mL	>0°C to ≤6°C; Ammonium Acetate Buffer	28 days (collection to analysis)
Metals by 6020A/200.8 (except Chromium IV and Hg)	P, G – 1000 mL	SCO_3 to pH<2	6 months (collection to analysis)

[1] (P) Polyethylene/Plastic; (G) Glass

[2] Recommended Holding Times from 40CFR136 and/or USEPA SW-846.

APPENDIX G – Standard Operating Procedures*

SOP Number	Rev #	SOP Title	Effect Date
		General Operations	
VAL-GEN-001	0	Preparation of Standard Operating Procedures	09/01/2015
VAL-GEN-002	0	Laboratory Ethics, Accountability, & Responsibility	09/01/2015
VAL-GEN-003	0	Glassware Cleaning	09/01/2015
VAL-GEN-004	0	Client Confidentiality & Electronic Data Transfer	09/01/2015
VAL-GEN-005	0	Departures from Documented Procedures	09/01/2015
VAL-GEN-006	0	Resource Review	09/01/2015
VAL-GEN-007	0	Subcontract Sample Submittal	09/01/2015
VAL-GEN-008	0	QC Criteria Development	09/01/2015
VAL-GEN-009	0	Management/Control of Standard Operating Procedures	09/01/2015
VAL-GEN-010	0	Procurement of Services & Materials	09/01/2015
		Quality Assurance	
VAL-QS-001	0	Chemical Purchase, Receipt, Storage, & Tracking	09/01/2015
VAL-QS-002	0	Report Revisions	09/01/2015
VAL-QS-003	0	Non-Conformance & Corrective Action Reporting	09/01/2015
VAL-QS-004	0	Control Charting	09/01/2015
VAL-QS-005	0	Validation of New Instruments & Methods	09/01/2015
VAL-QS-006	0	Method Detection and Method Quantitation Limits	09/01/2015
VAL-QS-007	0	Reagent Water	09/01/2015
VAL-QS-008	0	Sub-Sampling and Sample Homogenization	09/01/2015
VAL-QS-009	0	Data Reduction, Review, & Validation	09/01/2015
VAL-QS-010	0	Laboratory Calculations & Significant Figures	09/01/2015
VAL-QS-011	0	Record Archival	09/01/2015
VAL-QS-012	1	Internal Audits	01/01/2016
VAL-QS-013	0	Employee Training	09/01/2015
VAL-QS-014	0	Laboratory Record Control Procedures	09/01/2015
VAL-QS-015	0	Data Integrity System	09/01/2015
VAL-QS-016	0	Manual Integration Policy	09/01/2015
VAL-QS-017	1	Quality Assurance Management Review	01/01/2016

VAL-QS-020	0	Batch QC Evaluation	09/01/2015
VAL-QS-021	0	Spectrophotometric Calibrations	09/01/2015
VAL-QS-022	0	Estimation of Uncertainty	09/01/2015
		Information Technology	
VAL-IT-001	0	Data Integrity & Verification	09/01/2015
VAL-IT-002	0	Software Installation & Maintenance	09/01/2015
VAL-IT-003	0	IT Security	09/01/2015
VAL-IT-004	0	Test Code Management	09/01/2015
VAL-IT-005	0	Electronic Time Changes	09/01/2015
		Safety/Waste	
VAL-SAF-001	0	Waste Disposal Procedures	09/01/2015
VAL-SAF-003	0	Lockout/Tagout	09/01/2015
VAL-HSE-001	0	Valparaiso Emergency Response Plan	03/01/2019
VAL-HSE-002	0	Valparaiso Disaster Management Plan	03/01/2019
VAL-HSE-003	0	Valparaiso Laboratory Waste Management Plan	12/01/2020
		Wet Chemistry	
VAL-WC-001	2	TSS Determination	09/25/2019
VAL-WC-002	2	Cyanide	01/20/2020
VAL-WC-003	2	BOD – CBOD	09/25/2019
VAL-WC-004	2	Ammonia	01/21/2020
VAL-WC-005	0	Oil and Grease (HEM)	09/15/2015
VAL-WC-006	0	pH Measurement	09/15/2015
VAL-WC-007	2	Hexavalent Chromium	01/20/2020
VAL-WC-008	1	Phenolics	01/20/2020
VAL-WC-009	0	Specific Conductance	09/15/2015
VAL-WC-010	1	Phosphorus by FIA	01/20/2020
VAL-WC-011	0	COD	09/15/2017
VAL-WC-012	2	Nitrate-Nitrite by FIA	01/20/2020
VAL-WC-013	2	IC-Hexavalent Chromium	01/20/2020
		Metals	
VAL-MET-001	0	Metals by ICPMS	09/15/2015
VAL-MET-002	1	ICPMS Microwave Digestion	05/03/2019
VAL-MET-003	0	Hardness by Calculation	09/15/2015

VAL-MET-004	0	Metals Aqueous Digestion	11/21/2019
		Microbiology	
VAL-MB-001	0	Total Coliform and E. Coli by ColiLert	03/08/2019
		Equipment	
VAL-EQ-001	0	Balance Use and Maintenance	09/01/2015
VAL-EQ-002	1	Thermometers and Temperature Monitoring	05/03/2019
VAL-EQ-003	0	Calibration of Volumetric Apparatus	09/01/2015
VAL-EQ-004	0	PM and Maintenance Records	09/01/2015
		Sample Management	
VAL-SM-001	1	Sample Receipt	11/17/2017
VAL-SM-003	0	LIMS Log-In Procedures	09/01/2015
		Administration	
VAL-ADM-003	1	Work Order Reporting	01/27/2021
VAL-ADM-004	0	Client Complaint Resolution	09/01/2015
VAL-ADM-005	0	Work Order Formatting	09/01/2015

* SOP listings can change between QAM revisions. Contact the laboratory QA Manager for a current listing.

APPENDIX H – Data Qualifiers

ND	-	Not Detected at or above the Reporting Limit
U	-	Not detected at or above the Method Detection Limit
J	-	Value between the Method Detection Limit and Method Reporting Limit
B	-	Detected in the associated method blank
S	-	Accuracy outside defined recovery limits
R	-	Precision outside defined deviation limits
P	-	Dual column results > 40% difference
E	-	Estimated – value above quantitation range
H	-	Processed (analyzed or extracted) outside recommended hold time
O	-	Sample Concentration > 400% analyte spike level
*	-	Value exceeded Maximum Contaminant Level
Z	-	Laboratory defined as explained in narrative

APPENDIX I – Master List of Controlled Documents

The Master List of Controlled Documents can be found within the ALS Valparaiso Training module located within the GlobalDocs->Employee Training folder. The listings can change between QAM revisions. Contact the laboratory QA Manager for a current listing.

Other normative documents:

- TNI (The NELAC Institute) Quality Manual template, Revision 1, February 23, 2011.

APPENDIX J – Laboratory Accreditations

Certification Authority*	Lab ID
Florida Department of Health (NELAC)	E871119
Indiana Department of Health (Drinking Water)	C-64-03, M-64-07
Illinois Environmental Protection Agency (NELAP - secondary)	200087
Commonwealth of Pennsylvania Department of Environmental Protection (NELAP – secondary)	68-05922
West Virginia Department of Environmental Protection	414

*Listing of Certified Parameters available upon request

APPENDIX 5
LABORATORY STANDARD OPERATING PROCEDURES

METALS BY ICP-MS

EPA 200.8 / SW846 6020A

SOPID:	VAL-MET-001	Rev. Number:	R03	Effective Date:	10/15/2021

Approved By: Jennifer Hall — Date: 10/15/21
Department Supervisor

Approved By: — Date: 10/15/21
Quality Assurance

Archival Date:		Doc Control ID#:		Editor:	

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1) Scope and Applicability

1.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of a large number of elements as either dissolved (aqueous only) or total recoverable metals.

1.2 This method is applicable to a variety of matrices including: drinking water, non-potable water, solid/chemical materials, and biological tissue.

1.3 ICP-MS has been applied to the determination of over 60 elements in various matrices. The method is applicable to analytical ranges of approximately 0.002 mg/L to 900 mg/L for aqueous matrices and 0.5 mg/kg to 900 mg/kg for solid matrices.

1.4 Method detection limits, quantitation limits, and linear ranges will vary with matrices, instrumentation, and operating conditions.

1.5 SW-846 Method 6020A is used to determine the analytes listed in Tables 20.1-A. This table lists more elements than the current version of Method 6020A. The additional elements are included based upon results of demonstrations of precision and accuracy and completion of method detection limit studies for aqueous and solid matrix.

1.6 Method 200.8 is used to determine the analytes listed in Table 20.1-B. This table lists more elements than the current version of Method 200.8. The additional elements are included based upon results of demonstrations of precision and accuracy and completion of method detection limit studies for aqueous matrix.

1.7 Internal standards are used for each analyte determined by ICP-MS. The internal standard mix used consists of ^{6}Li, ^{45}Sc, ^{89}Y, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. ^{89}Y is used for analysis in helium gas mode.

2) Summary of Procedure

2.1 Prior to analysis, samples that require total ("acid-leachable") values must be digested using appropriate sample preparation methods as specified in SOP VAL-MET-002, *Microwave Assisted Acid Digestion of Aqueous Samples and Extracts* or SOP VAL-MET-014, *Metals Aqueous Digestion (block)*.

2.2 Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. Ions are produced by radio frequency inductively coupled plasma, entrained in the plasma gas, and introduced into a mass spectrometer. The ions are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3) Definitions

3.1 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.3 Matrix Spike (MS): An aliquot of background sample spiked with a known concentration

of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.4 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentration of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.5 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.6 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.7 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.8 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes. The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.9 Standard Curve: A plot of concentrations of known analyte standards versus the instrument response to the analyte.

3.10 Internal Standard: A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the analytical test method.

3.11 Linear Dynamic Range (LDR): The concentration range through which the instrument response is linear.

3.12 Low-Level Quality Control sample (LLQC): A clean matrix sample spiked at the MQL and carried through the entire preparation and analysis process.

3.13 Low-Level Initial Calibration Verification (LLICV): A sample spiked at the MQL, used to validate the lower end of the initial calibration.

3.14 Low-Level Continuing Calibration Verification (LLCCV): A sample spiked at the MQL and analyzed periodically throughout an analytical sequence, monitoring continued performance of the lower end of a calibration.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data

handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP VAL-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Routine preventative maintenance must be performed as scheduled and documented to assure optimum instrument performance. Typical routine maintenance includes inspection and replacement of sample delivery tubing. Maintenance performed shall be recorded in a dedicated instrument maintenance logbook. Refer to VAL-EQ-004 for additional information.

6) Interferences

6.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z) as those being monitored. A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Isotope ratios should be established prior to the application of any corrections.

6.2 Isobaric molecular and double-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature [3,4]. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature [5], the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal): corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = (m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal), (where the final term adjusts for any selenium contribution at 77 m/z).

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes [6]).

6.3 The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant

fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol de-solvation and/or mixed plasma has been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

6.4 Physical interferences can be associated with sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.04% (400 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When completing analysis by Method 6020A, if the intensity level of an internal standard falls below 70 percent of the intensity of the calibration standard used for reference, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed. When completing analysis by Method 200.8 and the intensity of the internal standard is less than 60 percent or greater than 125 percent of the intensity of the calibration standard used for reference, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

6.5 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler or skimmer cone, spray chamber design, and the type of nebulizer affects the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.

7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (HN-QS-009).

7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.

7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.

7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances

7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,

7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Aqueous samples shall be collected in 500 ml plastic containers and preserved to a pH of <2 with HNO_3.

8.1.1 Drinking water samples may be collected in 250 mL unpreserved plastic containers

8.2 Dissolved metal analyses shall be field filtered through a 0.45µ filter and preserved to a pH of <2 with HNO_3. Filtering should be completed in the field at time of sampling.

8.3 Sample pH should be verified at time of sample receipt and adjusted if necessary.

8.3.1 If adjusted at time of receipt, non-drinking water samples shall be stored for a period of 24 hours after which the pH adjustment will be verified.

8.3.2 If adjusted at time of receipt, drinking water samples shall be stored for a period of 16 hours after which the pH adjustment will be verified.

8.4 Samples may be stored at room temperature. The holding time is six months for aqueous matrices that are preserved to a pH of <2.

9) Equipment and Supplies

9.1 Inductively coupled plasma-mass spectrometer (Agilent 7900): Capable of providing resolution, better than or equal to 1.0 amu at 5% peak height. The system must have a mass range from at least 5 to 250 amu and a data system that allows for corrections of isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon/helium and a peristaltic pump for the sample solution is required.

9.2 Various Class A volumetric flasks: 10.0, 25, 50, 100, 250, etc.

9.3 Variable volume pipettes: 1.0 and 5.0 ml.

10) Standards and Reagents

10.1 Argon gas supply: High-purity grade (99.99%).

10.2 Helium gas supply: High-purity grade (99.99%).

10.3 Nitric acid, concentrated (trace metal grade)

10.4 Hydrochloric acid, concentrated (trace metal grade)

Note: Acids used in the preparation of standards and samples for ICP-MS must be of high purity. Re-distilled acids are recommended due to the high sensitivity of the instrumentation.

10.5 Diluent Solution

10.5.1 Prepare as a solution containing 6% HNO3 – 4% HCl.
10.5.2 Prepare fresh daily.

10.6 Stock Spike Standards:

10.6.1 Metals Mix standard w/ Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Sr, Sn, Tl, V, and Zn @ 10 mg/L **and** Fe, K, Ca, Na, and Mg @ 1000 mg/L **and** B at 50 mg/L. (available from VHG ZALSLAB901-500 or equivalent)
10.6.2 Ti and Si Spike Stock @ 1000 ppm (available from Environmetal Express)

10.6.2.1.1 Single Element Working Spike Ti @ 10 mg/L and Si @ 50 mg/L.

10.6.2.1.2 Add 5 ml Ti and 25 ml Si Stock to 300 ml DI water in a 500 ml volumetric flask.
10.6.2.1.3 Acidify with 30 ml Nitric and 15ml Hydrochloric acid.
10.6.2.1.4 Bring to final volume with DI water.

10.6.3 Low-level Metals Mix Standard I w/ As, Ba, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, and V @ 0.5 mg/L **and** Be and Cd @ 0.2 mg/L **and** Al, Li, and Zn @ 1.0 mg/L **and** B @ 2.0 mg/L **and** Fe @ 8.0 mg/L **and** Mg, K, and Na @ 20 mg/L **and** Ca @ 50 mg/L. (available from VHG ZALSLAB1103-100 or equivalent)
10.6.4 Low-level Metals Mix Standard II w/ Sn @ 0.2 mg/L **and** Sb, Mo, and Ti @ 0.5 mg/L. (available from VHG ZALSLAB1104-100 or equivalent)
10.6.5 Low-Level Standard III – Si @ 100 mg/L
10.6.5.1.1 Add approximately 20 mL DI water to a 50 mL volumetric flask. Acidify each using 3 mL Nitric acid and 2 mL Hydrochloric acid.
10.6.5.1.2 Add 1.0 mL of 10,000 Si Standard (section 10.7.8)
10.6.5.1.3 Dilute to volume with DI.

10.7 Initial Calibration Stock Standards (available from SPEX or equivalent):

10.7.1 Stock 1: 10 mg/L – Ag, Al, As, Ba, Be, Cd, Co, Cu, Cr, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn : 1,000 mg/L – Fe, K, Ca, Na, Mg (ICAL Stk #1)
10.7.2 Stock 2: 1,000 mg/L – B
10.7.3 Stock 3: 1,000 mg/L _ Sr
10.7.4 Stock 4: 1,000 mg/L – Ti
10.7.5 Stock 5: 1,000 mg/L - Sn
10.7.6 Stock 6: 1,000 mg/L – Li
10.7.7 Stock 7: 1,000 mg/L - Si
10.7.8 Stock 8: 10,000 mg/L – Si
10.7.9 Stock 9: 1,000 mg/L – U
10.7.10 Stability of stock standards shall be consistent with the manufacturer's expiration date.

10.8 Intermediate Stock Standards for B @ 50 mg/L, Li @ 20 mg/L; Sr, Ti, Sn @ 10 mg/L; and Si @2000 mg/L:

10.8.1 Add approximately 40 mL of DI water to (3) 100 mL volumetric flasks. Acidify each using 6 mL Nitric acid and 4 mL Hydrochloric acid.
10.8.2 Quantitatively add 1.0 mL each of Stock 4, 5, and 6 (from Section 10.7) to first flask. (ICAL Stk #2)
10.8.3 Quantitatively add 5.0 mL of Stock 2 and 2.0 mL of Stock 7 (from Section 10.7) to the second flask. (ICAL Stk #3)
10.8.4 Quantitatively add 20.0 mL of Stock 8 (from Section 10.7) to the third flask. (ICAL Stk #4)
10.8.5 Bring each to a final volume of 100 ml with DI water.
10.8.6 The intermediate stock standard is stable for a period of 6 months. The expiration date may not exceed that of any parent solution.

10.9 Working Initial Calibration Standards:

10.9.1 Working Calibration Stock Standard (Int500)

10.9.1.1 Add approximately 125 ml of DI water to a 200 ml Class A volumetric flask. Acidify with 12 ml Nitric acid and 6 ml Hydrochloric acid.
10.9.1.2 Add 10 ml of ICAL Stk #1 (Section 10.7.1), 10 mL of ICAL Stk #2 (Section 10.8.2), 5 ml of ICAL Stk #3 (Section 10.8.3), 5 ml of ICAL Stk #4 (Section 10.8.4).
10.9.1.3 Bring to a final volume of 200 ml with DI water.
10.9.1.4 The working standard must be replaced weekly and the expiration date may not exceed that of any parent solution.

10.9.2 Calibration Standards

10.9.2.1 Prepare, at a minimum, five (5) initial calibration standards from the Working Calibration Stock Standard (Section 10.9.1) as detailed in Table 10.9.2.

Table 10.9.2

Standard (Note 1)	Amount of Working Calibration Stock	Final Volume (Note 2)	Final Concentration
Level I	0 ml	50 ml	0 µg/L
Level II	1.0 mL of Level V	50 ml	0.2 µg/L
Level III	1.0 ml of CCV	50 ml	2 µg/L
Level IV	2.5 ml of CCV	50 ml	5 µg/L
Level V	5.0 ml of CCV	50 ml	10 µg/L
Level VI	5 ml (Int500)	50 ml	50 µg/L
Level VII	9 ml (Int500)	50 ml	90 µg/L
Level VIII	20 ml (Int500)	50 ml	200/500/20000 µg/L

Note (1): Additional standards may be added to extend the calibration range.
Note (2): All standards must be adjusted to a final acid concentration of 6% HNO_3 and 4% HCl solution.

10.10 Stock Calibration Check Solutions (ICV):

10.10.1 ICV Std#1: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sb, Se, Tl, V, Zn @ 10 mg/L. (available from SPEX)
10.10.2 ICV Stk #2: Ca, Fe, K, Mg, Na @ 200 mg/L. (available from SPEX)
10.10.3 ICV Stk Std #3: Mo, Sn, Sr, Ti @ 10 mg/L. (available from SPEX)
10.10.4 ICV Stk Std #4: B@25 mg/L, Li, U@ 10 mg/L

10.10.4.1.1 Add approximately 40 ml of DI water to a 100 ml Class A volumetric flask. Acidify with 6 ml Nitric acid and 4 ml Hydrochloric acid.
10.10.4.1.2 Add 2.5 ml of the 1,000 mg/L Boron standard (section 10.7.2), 1.0 mL of the 1,000 mg/L Lithium standard (section 10.7.6), and 1.0 mL of the 1,000 mg/L Uranium standard (section 10.7.9).
10.10.4.1.3 Bring to a final volume of 100 mL with DI water.
10.10.4.1.4 Solution is stable for a period of 6 months.

10.10.5 ICV Stk Std #5: Si @ 1,000 mg/L. (available from Environmental Express or equivalent)

Note: The stock standard(s) for the ICV solution must be obtained from a second source supplier or, if purchased from the same supplier, be a different solution warrantied to be prepared from a different lot of parent constituents.

10.10.6 Working ICV Std @ 80/200/8000

10.10.6.1.1 Add approximately 40 ml of DI water to a 50 ml Class A volumetric flask. Acidify with 3 ml Nitric acid and 2 ml Hydrochloric acid. Add volumes of stocks (section 10.10) from table below:

Standard Name	Volume (mL)
ICV Stk Std #1	0.4
ICV Stk Std #2	2.0
ICV Stk Std #3	0.4
ICV Stk Std #4	0.4
ICV Stk Std #5	0.4

10.10.6.1.2 Bring to a final volume of 50 mL with DI water.
10.10.6.1.3 Expiration based on parent std. Typically prepared weekly.

10.11 Continuing Calibration Verification (CCV) Solution:

10.11.1 Working CCV Solution @ 100/250/10000.

10.11.1.1.1 Add approximately 20 ml of DI water to a 50 ml Class A volumetric flask. Acidify with 3 ml Nitric acid and 2 ml Hydrochloric acid. Add 10 mL of working calibration stock standard -Int500 (section:10.9.1).
10.11.1.1.2 Bring to volume with DI water.
10.11.1.1.3 Prepare weekly.

10.11.2 The stock standard(s) for the ICV solution must be obtained from a second source supplier or, if purchased from the same supplier, be a different solution warrantied to be prepared from a different lot of parent constituents.

10.12 Low-Level Initial Calibration Verification solution (LLICV/LLCCV) spike @ MQL:

10.12.1 Add approximately 20 mL DI water to a 50 mL volumetric flask and acidify with 3 mL Nitric acid and 2 mL Hydrochloric acid.

10.12.2 Pipet 0.5 mL Low-Level Metals mix standard I (section 10.6.3), 0.5 mL Low-Level Metals mix standard II (section 10.6.4) 0.5 mL Low-Level Metals mix standard III (section 10.6.5)
10.12.3 Bring to volume with DI water.
10.12.4 Prepare fresh weekly

10.13 Initial Calibration Blank (ICB/CCB):

10.13.1 Prepare reagent water with a 6% HNO_3 & 4% HCl content.

10.14 Interference Check Sample A (ICSA) Stock Standard – Available from SPEX: Cl @ 10,000 mg/L; C @ 2,000 mg/L; Al, Ca, Fe, K, Mg, NA, S @ 1,000 mg/L; Mo, Ti @ 20 mg/L.

10.15 Interference Check Sample A (ICSA) Working Standard

10.15.1 Add 2.5 ml of ICSA (Section 10.14) to a 50 ml Class A volumetric flask.
10.15.2 Dilute to 50 ml with diluent solution (Section 10.5).
10.15.3 Prepare weekly.

10.16 Interference Check Sample AB (ICSAB) Working Standard

10.16.1 Add 10 mL of working calibration stock standard -Int500 (section:10.9.1).
10.16.2 Add 2.5 ml ICSA (Section 10.14)
10.16.3 Dilute to 50 ml with diluent solution (Section 10.5).
10.16.4 Prepare weekly.

10.17 Linear Dynamic Range (LDR) Check Solution

10.17.1 Add 10 ml Stock Spike (Section 10.6.1 and 10.6.2) to a 50 ml Class A volumetric flask.
10.17.2 Bring to volume with diluent (Section 10.5).
10.17.3 This solution should be replaced weekly or if degradation is noted. The expiration date may not exceed that of any parent solution.

10.18 Internal Standard Stock Standard:

10.18.1 Multi-Element Mix containing Li, Sc, Y, In, Tb, Ho, and Bi @ 10 mg/L. Available from VHG Labs.

10.19 Internal Standard – Working Solution:

10.19.1 Add 12 mL conc. HNO3 and 6 mL conc. HCl to 100 mL of DI in a 200 ml Class A volumetric flask.
10.19.2 Add 10 ml of Multi-Element Mix (Section 10.18.1). Bring to volume with DI.
10.19.3 This solution should be replaced if degradation is noted. The expiration date may not exceed that of any parent solution.

10.20 ICP-MS Tune Stock Solution:

10.20.1 Tuning solution containing 10 mg/L of Be, Mg, Co, In, Ba, Ce, Li, Rh, Tl, U, Y, and Pb.

10.21 ICP-MS Working Tune Solution @ 100 ppb:

10.21.1 Dilute 1 ml of the ICP-MS tune stock solution (Section 10.20.1) to 100 mL.
10.21.2 Working tune solution must be replaced every 6 months or if degradation is noted. The expiration date of this solution may not exceed that of its parent.

10.22 Stock Spiking Solution:

Multi-element standards documented in Sections 10.6.1 and 10.6.2 shall be used for spiking.

10.22.1 Water Spike:

10.22.1.1 A 500µl volume of spike solutions 10.6.1 and 10.6.2 is added to the 50.0 ml volume of aqueous sample after transfer to the digestion vessel. Following digestion (VAL-MET-002), the digestate is brought to a final volume of 50 mL for block digestion and 55.0 ml for microwave digestion. Theoretical spike value is 0.1 mg/L for the trace metals, 10 mg/L for Ca/Fe/Mg/Na/K, and 0.5 mg/L for B and Si.

11) Method Calibration

11.1 Start-up Procedure

11.1.1 Visual check of instrument:

11.1.1.1 Inspect auto-sampler tubing; peristaltic pump tubing should be replaced daily.
11.1.1.2 Inspect sampling cone and skimmer cone for deposit build up; if build up is noticed, either clean or replace cone.
11.1.1.3 Verify argon gas flow; ensure there is 100 PSI coming into the instrument.
11.1.1.4 Check vacuum pressure and oil levels.
11.1.1.5 Check that the heat exchanger unit is turned on.
11.1.1.6 Record maintenance in routine maintenance logbook.

11.1.2 Turn plasma on and let the instrument stabilize for approximately 30 minutes.
11.1.3 During stabilization, verify basic instrument operating parameters. These parameters should be set at approximately:

11.1.3.1 RF power = 1500V
11.1.3.2 RF matching = 1.8V
11.1.3.3 Peristaltic Pump = 0.1 rps
11.1.3.4 S/C Temp = 2^0 C.

11.1.3.5 Small adjustments to the EM voltage and/or maintenance may be required to meet subsequent tuning specification. This may be done using the Autotune function in the software.

11.1.4 After instrument stabilization, perform an instrument tune using the ICP-MS Tune solution (Section 10.24). This is a preliminary tune to evaluate performance across the operating mass range of the instrument.

11.1.4.1 Analyze the ICP-MS tune solution in 5 replicates prior to the initial calibration.
11.1.4.2 Adjust mass calibration such that the unit mass falls within $\pm$ 0.1 amu of the expected value.
11.1.4.3 Acceptance Criteria:

11.1.4.3.1 Resolution should be ~ 0.75 amu at 5% peak height, and **must** be <0.90 amu.

11.1.4.3.2 Mass calibration must be +/- 0.1 amu from the true value.
11.1.4.3.3 Relative standard deviations (RSD) of absolute signals from the five replicates must be < 5% for all analytes.
11.1.4.3.4 Internal standard criteria are not applicable to the ICP-MS tune solution.

11.1.5 A P/A factor update shall be performed utilizing the 10ug/L standard incorporated in the initial calibration curve. This should be updated on a regular basis when a calibration curve begins to fail, a new calibration curve is used, and after instrument maintenance.

11.1.6 A five-point calibration (minimally) must be conducted daily utilizing a calibration blank and four calibration standards (Section 10.9.2).

11.1.6.1 All measurements must be based upon at least three integrations.
11.1.6.2 Reported values must use the average of the multiple integrations.
11.1.6.3 Results of the calibration blank must be < 3 times the current IDL for each element.
11.1.6.4 Internal standard criteria must be achieved for all analyses.

11.2 Initial Calibration Curve:

11.2.1 A linear regression (first order fit) of the instrument response versus the concentration of the standards is employed for subsequent quantitation. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:
y = instrument response (peak area)
a = slope of the line (coefficient of x)
x = concentration of the calibration standard
b = blank intercept

11.2.2 The analyst should not force the line through the origin, but have the intercept calculated from the five data points.

11.2.3 The regression calculation correlation coefficient (r) must be $\geq$ 0.998.

11.3 % Relative Error

11.3.1 Compare calibration % relative error in external spreadsheet.
11.3.2 Criteria <50% for Low point and <30% for Mid point

11.4 Initial Calibration Verification (ICV):

11.4.1 The initial calibration must be verified utilizing a second source calibration verification standard at a concentration below the mid-point of the calibration curve (Section 10.11).
11.4.2 The ICV must be run after each new initial calibration curve.
11.4.3 Must meet accuracy performance criteria of 90-110% as outlined in the applicable LIMS test code.
11.4.4 Internal standard criteria must be achieved for the ICV analysis.

11.5 Low-Level Initial Calibration Verification (LLICV):

11.5.1 The LLICV is analyzed at the laboratory MQL to verify the lower end of the initial calibration. (Section 10.12)
11.5.2 The LLICV must be run after each new initial calibration
11.5.3 Must meet accuracy performance criteria of 70-130% as outlined in the applicable LIMS test code.
11.5.4 Internal standard criteria must be achieved for the LLICV analysis.

11.6 Interference Check Solutions (ICS):

11.6.1 The ICS (Section 10.15 & 10.16) must be analyzed at the beginning of an analytical sequence and every 8 hours during the analytical run.
11.6.2 Must meet accuracy performance criteria of 80-120% as outlined in the applicable LIMS test code.
11.6.3 Internal standard criteria must be achieved for each ICS analysis.

11.7 Continuing Calibration Verification (CCV):

11.7.1 A same source standard must be analyzed at the beginning of each daily batch, after a maximum of 10 samples run (including the Method Blank, LCS, and MS/MSD), and at the end of the analytical run.
11.7.2 Must meet accuracy performance criteria of 90-110% as outlined in the applicable LIMS test code.
11.7.3 Internal standard criteria must be achieved for each CCV analysis.

11.8 Low-Level Continuing Calibration Verification (LLCCV):

11.8.1 A low-level sample (section 10.20) must be analyzed at the beginning of each daily sequence, after a maximum of 10 samples run (including QC), and at the end of the analytical sequence.
11.8.2 Must meet accuracy performance criteria of 70-130%, for samples of a similar concentration, as outlined in the applicable LIMS test code.
11.8.3 Internal standard criteria must be achieved for each LLCCV analysis.

12) Sample Preparation/Analysis

12.1 Digestion procedures are presented in the applicable sample preparation SOP (VAL-MET-002).

12.2 When internal standard response falls outside acceptance criteria (<70% for 6020A and <60% or >125% for 200.8), dilute the sample and reanalyze.

12.3 Typical Analytical Sequence:

12.3.1 Initial Calibration curve, minimum four standards and a blank
12.3.2 Initial Calibration Verification standards (once daily)
12.3.3 Initial Calibration Verification Blank (once daily)
12.3.4 Low-Level Initial Calibration Verification Standard (once daily)
12.3.5 Interference Check Sample A (ICSA)
12.3.6 Interference Check Sample AB (ICSAB)
12.3.7 Continuing Calibration Verification (CCV)
12.3.8 Low-Level Continuing Calibration Verification Standard (LLCCV)
12.3.9 Continuing Calibration Blank (CCB)
12.3.10 Method blank (one MB per preparation batch of 20 or less)
12.3.11 Laboratory Control Sample (one per preparation batch of 20 or less)
12.3.12 Client sample(s)
12.3.13 Matrix spike

12.3.13.1 For Method 200.8, prepare at a 10% frequency (one per every 10 samples)
12.3.13.2 For Method 6020A, prepare at a 5% frequency (one per preparation batch of 20 or less)

12.3.14 Matrix spike duplicate

12.3.14.1 For Method 200.8, prepare at a 10% frequency (one per every 10 samples)
12.3.14.2 For Method 6020A, prepare at a 5% frequency (one per preparation batch of 20 or less)

12.3.15 Continuing Calibration Verification Standard (CCV after every 10 samples)
12.3.16 Continuing Calibration Blank (CCB after every ten samples)
12.3.17 Low-Level Continuing Calibration Verification Standard (LLCCV after every 10 samples)
12.3.18 Client samples and batch QC samples (dilution test sample, PDS, MB, LCS and MS) – total of ten or less samples
12.3.19 Continuing Calibration Verification Standard (CCV at end of analytical sequence)
12.3.20 Continuing Calibration Blank (CCB at end of analytical sequence)
12.3.21 Low-Level Continuing Calibration Verification Standard (LLCCV at end of analytical sequence)

12.4 Dilution test:

12.4.1 If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank), an analysis of a fivefold dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected.

12.5 Post-Digestion Spike (PDS) Addition:

12.5.1 An analyte spike added to a portion of a prepared sample should fall within the laboratory derived acceptance criteria.
12.5.2 The spike addition should be based on the indigenous concentration of each element of interest in the sample.
12.5.3 If the spike is not recovered within the specified limits, the sample should be diluted and reanalyzed to compensate for the matrix effect.
12.5.4 Results must agree to within 10% of the original determination.
12.5.5 The use of a standard-addition analysis procedure may also be used if the dilution technique proves inconclusive.
12.5.6 Post Digestion Preparation:

12.5.6.1 To a 10 ml portion of digestion sample, add 100 µl of Metals mix standard I. (Section 10.6.1)
12.5.6.2 The theoretical spike is 100 ug/L for the trace metals, 10,000 ug/L for minerals, and 500 ug/L for Boron.

12.6 Method of Standard Additions (MSA):

12.6.1 When MS/MSD and PDS criteria are not met, the method of standard additions may be used to determine an accurate analyte level.
12.6.2 The MSA is an extension of the PDS where three PDS are performed on the same sample.

12.6.2.1 Ideally, the first PDS is spiked at approximately 50% of the estimated analyte concentration.

The second PDS is spiked at ~100% and the third at ~150%.

12.6.3 The MSA analyte concentration is determined using linear regression using the four data points. An MS Excel spreadsheet calculation is employed to calculate results from MSA.

13) Troubleshooting

13.1 Refer to Agilent 7900 hardware manual for specific technical troubleshooting guidance.

14) Data Acquisition

14.1 Create a prep batch (as applicable) in LIMS.

14.2 The data acquired is transferred via Mass Hunter™ to LIMS electronically. Calculations are performed by Mass Hunter™ software and LIMS.

14.3 Analyst review of data is performed on the raw data and in LIMS prior to being validated. If results are above the analytes detectable range, it will be reported as "----------". Appropriate dilutions must be performed to generate reportable data.

15) Calculation, and Data Reduction Requirements

15.1 Calculation of Linear Regression Correlation Coefficient, r

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{n}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{n}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{n}\right)}}$$

Where:
X = individual values for independent variable
Y = individual values for dependent variable
n = number of pairs of data.
df = n-2

15.2 Calculation of the CCV % drift:

15.2.1 % Drift= [(Calculated conc - Theoretical conc) x 100] / Theoretical conc

15.3 Calibration Relative Error Calculation

$$\% \text{ Relative Error} = (x'_i - x_i) / x_i * 100$$

Where:
x_i = True value for the calibration standard.
x'_i = Measured concentration of the calibration standard.

The calibration relative error must be calculated using the low-point and mid-point standards.

15.4 The calibration curve versus sample response data produces the metal concentration in solution.

15.4.1 Equation for water samples:

$$Concentration(ug/L) = Sample\,\mathrm{Re}sponse(ug/L)\,x\,Dilution\,Factor\,(If\ \ Applicable)$$

15.4.2 Equation for soil samples (external calibration):

$$Concentration(ug/kg) = \frac{Sample\ Response(ug/L)xFV}{Weight\ of\ \ Sample\,(g)}\,x\,Dil.\,Factor\,(If\ \ Applicable)$$

Where:

FV = final volume of digestion, ml

15.4.3 If additional dilutions are used, the result must be multiplied by the total dilution factor.

15.5 QC Calculations: Calculate the percent recovery for various QC samples (MS, MSD, LCS) according to the following equations:

15.5.1 % Recovery, %R (for MS/MSD and LCS)

$$\%\mathrm{R} = \frac{(\mathrm{SSR} - \mathrm{SR})}{\mathrm{SA}} \times 100$$

Where:

SSR = Spiked Sample Result (mg/L or mg/kg).
SR = Sample Result (unspiked)
SA = Spike Amount Added (mg/L or mg/kg).

15.5.2 % Recovery, %R (for standards and CCV)

$$\%R = \frac{(SSR)}{SA} \times 100$$

Where:

SSR = Spiked Sample Result (mg/L or mg/kg).
SA = Spike Amount Added (mg/L or mg/kg).

15.5.3 % RPD (for precision or replication evaluation)

$$\%RPD = \frac{|SR_1 - SR_2|}{½(SR_1 + SR_2)} \times 100$$

Where:

SR_1 = Sample result for replicate 1.
SR_2 = Sample result for replicate 2.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Instrument Detection Limit (IDL)

16.1.1 IDL determinations should be determined every three months and maintained with the instrument logbook.
16.1.2 IDL determinations are to be completed by averaging the standard deviations of seven measurements of a reagent blank, over a minimum of three non-sequential analytical runs.

16.2 Initial Calibration:

16.2.1 A calibration curve must be generated daily or whenever ICV/CCV fail to achieve acceptance criteria.
16.2.2 Acceptance Criteria:

16.2.2.1 Curve must be determined from a minimum of four standards and a calibration blank.
16.2.2.2 The regression coefficient "r" must be ≥0.998
16.2.2.3 All responses must be based upon the average of three integrations at a minimum
16.2.2.4 The Relative Error at the midpoint shall meet the criteria specified in the method at the midpoint (CCV Criteria) and at the lowest level the criteria shall be ± 50%.

16.2.3 Curve Failure Corrective Action:

16.2.3.1 Check standards and/or perform maintenance as necessary to correct problem.
16.2.3.2 Process a new initial calibration curve

16.3 Initial Calibration Verification (ICV):

16.3.1 Perform daily after generation of the initial calibration curve.
16.3.2 Acceptance criteria:

16.3.2.1 Must meet accuracy performance criteria of 90-110% as outlined in the applicable LIMS test code.

16.3.3 ICV Failure Corrective Action:

16.3.3.1 Evaluate condition and age of standards being used and/or perform any needed system maintenance.
16.3.3.2 Reanalyze the ICV and /or generate a new calibration curve as necessary to achieve acceptable calibration criteria.

16.4 Low-Level Initial Calibration Verification (LLICV):

16.4.1 Perform daily after generation of the initial calibration curve.
16.4.2 Acceptance criteria:

16.4.2.1 Must meet accuracy performance criteria of 70-130% as outlined in the applicable LIMS test code.

16.4.3 LLICV Failure Corrective Action:

16.4.3.1 Evaluate condition and age of standards being used and/or perform any needed system maintenance.
16.4.3.2 Reprocess the LLICV and /or generate a new calibration curve as necessary to achieve acceptable calibration criteria.

16.5 Continuing Calibration Verification (CCV):

16.5.1 The CCV must be run prior to sample analysis, after every 10 samples (including QC samples), and at the end of the analytical sequence.
16.5.2 Acceptance Criteria:

16.5.2.1 Must meet accuracy performance criteria of 90-110% as outlined in the applicable LIMS test code.

16.5.3 CCV failure Corrective Action:

16.5.3.1 If the calibration does not meet the criteria, re-analyze the standard.
16.5.3.2 If subsequent analysis is outside of criteria, perform a new calibration curve.
16.5.3.3 All samples processed following the last acceptable CCV must be re-analyzed.

16.6 Low-Level Continuing Calibration Verification (LLCCV):

16.6.1 The LLCCV must be run prior to sample analysis, after every 10 samples (including QC samples), and at the end of the analytical sequence.
16.6.2 Acceptance Criteria:

16.6.2.1 Must meet accuracy performance criteria of 70-130% for analytes of a similar concentration, as outlined in the applicable LIMS test code.

16.6.3 LLCCV failure Corrective Action:

16.6.3.1 If the calibration does not meet the criteria, re-analyze the standard.
16.6.3.2 If subsequent analysis remains outside of criteria, perform a new calibration curve.
16.6.3.3 All samples of similar concentration (<CCV), processed following the last acceptable LLCCV must be re-analyzed.

16.7 Continuing Calibration Blank (CCB):

16.7.1 The calibration blank must be run prior to sample analysis, after every 10 samples (including QC samples), and at the end of the analytical sequence.
16.7.2 Acceptance Criteria:

16.7.2.1 All analytes are must be less than three times the IDL.

16.7.3 CCB failure Corrective Action:

16.7.3.1 If the calibration blank does not meet the criteria, re-analyze the blank.
16.7.3.2 If subsequent analysis falls outside of criteria, perform any necessary maintenance and perform a new calibration curve.

16.7.3.3 All samples processed following the last acceptable CCB must be re-analyzed.

16.8 Linear Dynamic Range (LDR) Assessment

16.8.1 A LDR sample must be processed to assess linearity above the highest calibration standard.

16.8.2 Acceptance Criteria:

16.8.2.1 All analytes are must be within 10% of the true value of the LDR standard.

16.8.2.2 Sample concentrations greater than 90% of the LDR must be diluted and re-analyzed.

16.8.2.3 The LDR should be verified every 6 months (minimally) or whenever a modification in instrument hardware or operating conditions presents the potential for a change in the LDR.

16.8.3 LDR assessment failure Corrective Action:

16.8.3.1 If the LDR does not meet criteria for an analyte, no data for that analyte falling between the highest calibration standard and the LDR standard can be reported.

16.9 Blanks:

16.9.1 Rinse Blank(s)

16.9.1.1 Rinse blanks should be used to flush system components between blanks, standards, and samples.

16.9.1.2 Allow sufficient time to remove traces of the previous sample prior to new sample introduction.

16.9.1.3 Rinse blanks are not to be routinely run before QC samples. If carryover is an issue, rinse-out times may need to be addressed.

16.9.2 Calibration Blank(s)

16.9.2.1 See Section 16.7.

16.9.3 Method Blank(s)

16.9.3.1 A method blank must be processed with each batch of 20 or less samples of the same matrix and prepared on the same working shift.

16.9.3.2 Acceptance Criteria:

16.9.3.2.1 All analytes of interest should be less than one half the PQL and must be less than the PQL.

16.9.3.2.2 Method blank values exceeding the PQL indicate laboratory/reagent contamination and should be considered suspect.

16.9.3.2.3 Method blank values exceeding the PQL may be considered useable if:

16.9.3.2.3.1 The blank analyte concentration is < 5% of the sample analyte concentration,

16.9.3.2.3.2 less than 5% of the regulatory limit,

16.9.3.2.3.3 or less than 3 times the MDL (whichever is greater),

16.9.3.2.3.4 All associated samples are appropriately qualified, and Project Management notification/approval is completed.

16.9.3.2.4 Other approved QA program requirements must be followed when the acceptable blank contamination specified in the approved QA project plan differs from the above.

16.9.3.3 Corrective Action:

16.9.3.3.1 If the method blank results do not meet the acceptance criteria above, then the laboratory must take corrective action to locate and reduce the source of the contamination.
16.9.3.3.2 All samples associated with the contaminated method blank must be reprocessed.
16.9.3.3.3 If samples cannot be reprocessed due to insufficient sample volume or other similar circumstances, a non-conformance must be documented in the data checklist for the analytical run. This must provide sufficient detail for project narration and to ensure all appropriate data flags are entered into LIMS.
16.9.3.3.4 Data reported with an associated contaminated method blank must be flagged with a "B".

16.10 Laboratory Control Sample (LCS):

16.10.1 The LCS must be processed with each batch of 20 or less samples of the same matrix and processed on the same shift.
16.10.2 Acceptance Criteria:

16.10.2.1 Must meet accuracy performance criteria as outlined in the applicable LIMS test code.

16.10.3 LCS Corrective Action:

16.10.3.1 If the LCS recovery does not meet acceptance criteria, the sample batch must be reprocessed.
16.10.3.2 If samples cannot be reprocessed due to insufficient sample volume or other similar circumstances, a non-conformance must be documented in the data checklist for the analytical run. This must provide sufficient detail for project narration and to ensure all appropriate data flags are entered into LIMS.
16.10.3.3 Data reported with a failed LCS must be flagged and narrated as to potential bias characteristics.

16.11 Low-level Quality Control Sample (LLQC):

16.11.1 The LLQC is digested and must be processed quarterly.
16.11.2 Acceptance Criteria:

16.11.2.1 Must meet accuracy performance criteria of 70-130% as outlined in the applicable LIMS test code.

16.11.3 LLQC Corrective Action:

16.11.3.1 If the LLQC recovery does not meet acceptance criteria, investigate the cause of the failure.
16.11.3.2 Reprocess the LLQC once the cause of the failure has been identified and corrected.
16.11.3.3 If a cause cannot be identified and corrected, spike LLQC at a higher concentration, process, and adjust PQLs accordingly.

16.12 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

16.12.1 A MS/MSD pair must be processed at a 10% frequency for Method 200.8 and at a 5% frequency for Method 6020A. MS/MSD samples must be of the same matrix and processed during the same working shift.

16.12.2 Acceptance Criteria:

16.12.2.1 Must meet accuracy and precision performance criteria as outlined in the applicable LIMS test code.

16.12.2.2 Recovery values should not be evaluated if the spike concentration is less than 25% of the parent concentration.

16.12.3 MS/MSD Corrective Action:

16.12.3.1 If the MS/MSD pair generates recovery values outside acceptance criteria, the deviation may be due to matrix effects. The LCS, internal standard recoveries, and calibration results must all be evaluated in order to determine if matrix interference is present. (Note that the MS/MSD are used to evaluate the matrix effect, not to control the analytical process.) If both the MS/MSD fall outside accuracy criteria for the same analyte, a matrix effect is suspected, assuming the LCS achieves accuracy criteria, and all internal standard recoveries are consistent.

As an example, if the matrix spikes exhibit low recovery but good precision, laboratory control samples exhibit acceptable accuracy, and internal standard recovery is consistent, the presence of matrix interference is probable.

16.12.3.2 If the MS/MSD pair generates inconsistent recovery values and/or suspect LCS values are present, laboratory error (and not matrix inference) is suspected.

As an example, if precision between the MS/MSD pair is poor and the LCS presents divergent results, the presence of laboratory error is probable.

16.12.3.3 If the MS/MSD fails acceptance criteria, the data must be evaluated for error or possible matrix effect.

16.12.3.4 If laboratory error is indicated, all associated samples must be reprocessed. If samples cannot be reprocessed due to limited sample volume or other similar circumstances, all reported values must be qualified and narrated as to potential bias or usability.

16.12.3.5 If matrix interference is indicated, associated samples may be reported with appropriate qualification and narration.

16.12.3.6 A non-conformance must be documented in the data checklist for either scenario and must contain sufficient detail for project narration and to ensure all appropriate data qualifiers have been entered into LIMS.

16.13 Internal Standards (IS):

16.13.1 Internal standards must be added to all samples with the exception of the ICPMS tuning solution. We utilize an automatic internal standard introduction system via a peristaltic pump.

16.13.2 Acceptance Criteria:

16.13.2.1 For samples processed according to USEPA 6020A, the IS results must be >70% of the original response in the initial calibration.

16.13.2.2 For samples processed according to USEPA 200.8, the IS results must fall between 60%-125% of the original response in the initial calibration.

16.13.2.3 Analytical results associated with IS failures may not be reported.

16.13.3 IS failure corrective action:

16.13.3.1 If criteria are not met, the cause of the problem must be determined, corrected, and the samples re-analyzed.
16.13.3.2 The sample must undergo a five-fold (1+4) dilution to alleviate potential matrix interference. Note: Greater dilutions may be necessary for samples contributing significant matrix interference.
16.13.3.3 Samples undergoing a necessary dilution due to IS failure must be notated as such if the target analyte concentration falls below the reporting limit.
16.13.3.4 If samples cannot be re-analyzed, all associated results must be qualified as "Unusable".

16.14 Reported Analyte Concentration

16.14.1 Reported concentrations for applicable analytes must be reported from the least dilute analysis that achieves all required quality control parameters.

16.15 Interference Check Solution:

16.15.1 The interference check solutions must be processed at the beginning of each analytical sequence and every 8 hours during an analytical run.
16.15.2 Acceptance Criteria:

16.15.2.1 Must meet accuracy performance criteria as outlined in the applicable LIMS test code.
16.15.2.2 All internal standard criteria must be achieved for the interference check solution analysis.

16.15.3 Interference Check Solution Failure

16.15.3.1 All samples associated with a failure of the ICS must be reprocessed.
16.15.3.2 If samples cannot be re-analyzed, all sample results must be qualified as unusable.

16.16 Dilution Test Check

16.16.1 If the sample analyte concentration is within the linear dynamic range and sufficiently high (>100 times the reagent blank), a sample dilution test should be completed at a five-fold dilution.
16.16.2 Acceptance Criteria

16.16.2.1 Must meet precision performance criteria as outlined in the applicable LIMS test code.

16.16.3 Dilution Test Failure

16.16.3.1 In the event of a dilution test failure, the sample must be closely inspected for indications of matrix interference.
16.16.3.2 A post digestion spike or standard addition should be completed on the failed sample to verify matrix interference.

16.17 Post Digestion spike requirements

16.17.1 One post digestion spike (PDS) must be completed for each batch of $\leq$ 20 samples.
16.17.2 The PDS should be spiked at the same level as the MS/MSD.
16.17.3 Acceptance Criteria

16.17.3.1 Must meet accuracy performance criteria as outlined in the applicable LIMS test code.

16.17.4 PDS Failure

16.17.4.1 If the spike is not recovered within the recommended limits, the sample must be diluted and reanalyzed.

16.17.4.2 The results of the diluted re-analysis must agree within ± 10% of the original determination.

16.17.4.3 If the PDS fails the various acceptance criteria, the sample should be processed using standard additions as detailed in Section 12.6.

16.18 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Demonstration of Proficiency:

19.1.1 Initial Demonstration of Proficiency

19.1.1.1 The laboratory must determine linear dynamic range, method detection limits, and evaluation of quality control samples prior to sample analysis by this procedure.

19.1.2 Routine Demonstration of Proficiency

19.1.2.1 Each analyst must demonstrate initial proficiency with sample preparation and/or analytical determination by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix.

19.1.2.2 Each analyst must demonstrate ongoing proficiency annually with each sample preparation and/or analytical determination method by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix or by passing performance in approved PT evaluations.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed on instrumentation (ex: change detector, auto-sampler, etc.).

19.3 On-going laboratory performance must be documented via performance evaluation studies and must be completed approximately every 6 months.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R00	9/15/15	CES	New SOP
R01	5/3/19	LC	Added sections 8.1.1 and 8.3.2 for DW
R02	9/15/21	PAL	Changes to standards section 10; added Calibration residual; removed sections 17.6-7
R03	10/15/21	LC	Added Appendix A for low-level Ag

21) References and Related Documents

21.1 Environmental Protection Agency, “Method 6020A Inductively Coupled Plasma Mass Spectrometry”, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Revision 1, February 2007.

21.2 U.S. Environmental Protection Agency, “Method 200.8, Inductively Coupled Plasma – Mass Spectrometry,” Methods for Chemical Analysis of Water and Wastes, EPA-821-R-99-017, Revision 5.5, 1999.

21.3 ALS Environmental Quality Assurance Manual, Revision 6.0 (or most current)

21.4 Appendix A, Low-Level

21.5 Table 20.1-A - ICP-MS Analyte Listing for SW 846-6020A

21.6 Table 20.1-B - ICP-MS Analyte Listing for Method 200.8

21.7 Table 20.2 - LCS Acceptance Criteria

21.8 Table 20.3-A - Internal Standard Criteria for CLP SW 846-6020A

21.9 Table 20.3-B - Internal Standard Criteria for CLP Method 200.8

21.10 Table 20.4 - Calibration and QC Summary

Appendix A – Low-Level Ag

1. Scope and Applicability

1.1. Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of a large number of elements as either dissolved (aqueous only) or total recoverable metals. This method will be used for low level silver. Additional elements may be added based on need for lower reporting limits.

1.2. This method is applicable to a variety of matrices including: drinking water, non-potable water, solid/chemical materials, and biological tissue.

1.3. ICP-MS has been applied to the determination of over 60 elements in various matrices. The method is applicable to analytical ranges of approximately 0.015 µg/L to 2 µg/L for aqueous matrices for silver.

1.4. Method detection limits, quantitation limits, and linear ranges will vary with matrices, instrumentation, and operating conditions.

1.5. Method 200.8 is used to determine the analytes listed in Table 20.1-B. This table lists more elements than the current version of Method 200.8. The additional elements are included based upon results of demonstrations of precision and accuracy and completion of method detection limit studies for aqueous matrix.

2. Standards and Reagents

2.1. Argon gas supply: High-purity grade (99.99%).
2.2. Helium gas supply: High-purity grade (99.99%).
2.3. Nitric acid, concentrated (trace metal grade)
2.4. Hydrochloric acid, concentrated (trace metal grade)

Note: Acids used in the preparation of standards and samples for ICP-MS must be of high purity. Re-distilled acids are recommended due to the high sensitivity of the instrumentation.

2.5. Diluent Solution

2.5.1. Prepare as a solution containing 6% HNO3 – 4% HCl.
2.5.2. Prepare fresh daily.

2.6. Stock Spike Standards:

2.6.1. Metals Mix standard w/ Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Sr, Sn, Tl, V, and Zn @ 10 mg/L **and** Fe, K, Ca, Na, and Mg @ 1000 mg/L **and** B at 50 mg/L. (available from VHG ZALSLAB901-500 or equivalent)

2.6.2. Low-level Metals Mix Standard I w/ As, Ba, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, and V @ 0.5 mg/L **and** Be and Cd @ 0.2 mg/L **and** Al, Li, and Zn @ 1.0 mg/L **and** B @ 2.0 mg/L **and** Fe @ 8.0 mg/L **and** Mg, K, and Na @ 20 mg/L **and** Ca @ 50 mg/L. (available from VHG ZALSLAB1103-100 or equivalent)

2.7. Initial Calibration Stock Standards (available from SPEX or equivalent):

2.7.1. Stock 1: 10 mg/L – Ag, Al, As, Ba, Be, Cd, Co, Cu, Cr, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn : 1,000 mg/L – Fe, K, Ca, Na, Mg (ICAL Stk #1)

Stability of stock standards shall be consistent with the manufacturer's expiration date.

2.8. Working Initial Calibration Standards:

2.8.1. Working Calibration Stock Standard (Level VI) (Sec. 10.9.2.1)

2.8.1.1. Add approximately 125 ml of DI water to a 200 ml Class A volumetric flask. Acidify with 12 ml Nitric acid and 6 ml Hydrochloric acid.
2.8.1.2. Add 5 ml of Int500 (Section 10.9.1).
2.8.1.3. Bring to a final volume of 50 ml with DI water.
2.8.1.4. The working standard must be replaced monthly and the expiration date may not exceed that of any parent solution.

2.8.2. Calibration Standards

2.8.2.1. Prepare, at a minimum, five (5) initial calibration standards from the Working Calibration Stock Standard (Section 1.8.1) as detailed in Table 1.10.2.1.

Table 1.10.2.1

Standard (Note 1)	Amount of Working Calibration Stock	Final Volume (Note 2)	Final Concentration
Ag Level I	0 ml	50 ml	0 µg/L
Ag Level II	0.375 mL of (Ag Level VII)	50 ml	0.015 µg/L
Ag Level III	1.25 ml of (Ag Level VI)	50 ml	0.05 µg/L
Ag Level IV	0.1 ml (Level VI)	50 ml	0.1 µg/L
Ag Level V	0.5 ml (Level VI)	50 ml	0.5 µg/L
Ag Level VI	1.0 ml (Level VI)	50 ml	1.0 µg/L
Ag Level VII	2.0 ml (Level VI)	50 ml	2.0 µg/L

Note (1): Additional standards may be added to extend the calibration range.
Note (2): All standards must be adjusted to a final acid concentration of 6% HNO_3 and 4% HCl solution.

2.9. Stock Calibration Check Solutions (ICV): (working ICV Std sec. 10.10.6)
Note: The stock standard(s) for the ICV solution must be obtained from a second source supplier or, if purchased from the same supplier, be a different solution warrantied to be prepared from a different lot of parent constituents.

2.9.1. Working ICV Std @ 0.4

2.9.1.1.1. Add approximately 40 ml of DI water to a 50 ml Class A volumetric flask. Acidify with 3 ml Nitric acid and 2 ml Hydrochloric acid. Add volumes of stocks (section 10.10) from table below:

Standard Name	Volume (mL)
ICV Stk Std #1	0.25

2.9.1.1.2. Bring to a final volume of 50 mL with DI water.
2.9.1.1.3. Expiration based on parent standard. Typically prepared monthly.

2.10. Continuing Calibration Verification (CCV) Solution:

2.10.1. Working CCV Solution @ 0.5.

2.10.1.1.1. Add approximately 20 ml of DI water to a 50 ml Class A volumetric flask. Acidify with 3 ml Nitric acid and 2 ml Hydrochloric acid. Add 0.25 mL of working CCV (Sec. 10.11).
2.10.1.1.2. Bring to volume with DI water.
2.10.1.1.3. Prepare weekly.

2.10.2. The stock standard(s) for the ICV solution must be obtained from a second source supplier or, if purchased from the same supplier, be a different solution warrantied to be prepared from a different lot of parent constituents.

2.11. Low-Level Initial Calibration Verification solution (LLICV/LLCCV) spike @ MQL:

2.11.1. Add approximately 20 mL DI water to a 50 mL volumetric flask and acidify with 3 mL Nitric acid and 2 mL Hydrochloric acid.
2.11.2. Pipet 0.15 mL Low-Level Metals standard (section 10.12)
2.11.3. Bring to volume with DI water.
2.11.4. Prepare fresh weekly

2.12. Initial Calibration Blank (ICB/CCB):

2.12.1. Prepare reagent water with a 6% HNO_3 & 4% HCl content.

2.13. Linear Dynamic Range (LDR) Check Solution
2.13.1. LDR is limited to calibration range

2.14. Internal Standard Stock Standard:

2.14.1. Multi-Element Mix containing Li, Sc, Y, In, Tb, Ho, and Bi @ 10 mg/L. Available from VHG Labs.

2.15. Internal Standard – Working Solution:

2.15.1. See section 10.19

2.16. ICP-MS Tune Stock Solution:

2.16.1. Tuning solution containing 10 mg/L of Be, Mg, Co, In, Ba, Ce, Li, Rh, Tl, U, Y, and Pb.

2.17. ICP-MS Working Tune Solution @ 100 ppb:

2.17.1. See section 10.21

2.18. Stock Spiking Solution:

2.18.1. Multi-element standards documented in Sections 10.6.1 and 10.6.2 shall be used for spiking.

2.18.2. Water Spike:

2.18.2.1. A 500µl volume of Cal Standard VI solution 1.8.1is added to the 50.0 ml volume of aqueous sample after transfer to the digestion vessel. Following digestion (VAL-MET-002), the digestate is brought to a final volume of 50 mL for block digestion and 55.0 ml for microwave digestion. Theoretical spike value is 0.5 ug/L for Ag

3. Method Calibration

3.1. See section 11 for analysis procedure using low level silver method. All QC criteria are the same as the standard method.

Table 20.1-A

Analyte List: SW 846-6020A

Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

(Additional analytes may be added based upon appropriate performance data.)

Table 20.1-B

Analyte List: Method 200.8

Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

(Additional analytes may be added based upon appropriate performance data.)

TABLE 20.2 - LCS ACCEPTANCE CRITERIA FOR METALS ANALYSIS BY ICP/MS

Analyte	Water Spike Amt, mg/L	6020A Water Lower %R Limit	6020A Water Upper %R Limit	200.8 Water Lower % R Limit	200.8 Water Upper % R Limit	Soil Spike Amt, mg/Kg	Soil Lower % R Limit	Soil upper % R Limit
Aluminum	0.1	80	120	85.0	115	5	80	120
Antimony	0.1	80	120	85.0	115	5	80	120
Arsenic	0.1	80	120	85.0	115	5	80	120
Barium	0.1	80	120	85.0	115	5	80	120
Beryllium	0.1	80	120	85.0	115	5	80	120
Cadmium	0.1	80	120	85.0	115	5	80	120
Calcium	10.0	80	120	85.0	115	500	80	120
Chromium	0.1	80	120	85.0	115	5	80	120
Cobalt	0.1	80	120	85.0	115	5	80	120
Copper	0.1	80	120	85.0	115	5	80	120
Iron	10.0	80	120	85.0	115	500	80	120
Lead	0.1	80	120	85.0	115	5	80	120
Potassium	10.0	80	120	85.0	115	500	80	120
Magnesium	10.0	80	120	85.0	115	500	80	120
Manganese	0.1	80	120	85.0	115	5	80	120
Molybdenum	0.1	80	120	85.0	115	5	80	120
Nickel	0.1	80	120	85.0	115	5	80	120
Selenium	0.1	80	120	85.0	115	5	80	120
Silver	0.1	80	120	85.0	115	5	80	120
Sodium	10.0	80	120	85.0	115	500	80	120
Strontium	0.1	80	120	85.0	115	5	80	120
Thallium	0.1	80	120	85.0	115	5	80	120
Tin	0.1	80	120	85.0	115	5	80	120
Titanium	0.1	80	120	85.0	115	5	80	120
Vanadium	0.1	80	120	85.0	115	5	80	120
Zinc	0.1	80	120	85.0	115	5	80	120

Table 20.3-A
Metals Analysis by ICP/MS: SW 846-6020A
Internal Standard Criteria for CCV, CCB and samples; Determined by CLP Method

Samples and QC samples	Isotope	Ref IS	Lower %	Upper %
Li	7	Sc	-	-
Be	9	Sc	-	-
B	11	Sc	-	-
Na	23	Y	-	-
Mg	24	Y	-	-
Al	27	Y	-	-
K	39	Y	-	-
Ca	44	Y	-	-
Sc (IS)	45	-	70	-
Ti	47	Y	-	-
V	51	Y	-	-
Cr	53	Y	-	-
Mn	55	Y	-	-
Fe	56	Y	-	-
Co	59	Y	-	-
Ni	60	Y	-	-
Cu	63	Y	-	-
Zn	66	Y	-	-
As	75	Y	-	-
Se	82	Y	-	-
Sr	87	Y	-	-
Y (IS)	89	-	70	-
Mo	98	Y	-	-
Ag	107	In (2), Y (3)	-	-
Cd	111	In	-	-
In (IS)	115	-	70	-
Sn	118	In	-	-
Sb	121	In (2), Y (3)	-	-
Ba	135	In	-	-
Tl	203	Bi	-	-
Pb	207	Bi	-	-
Bi (IS)	209	-	70	-

Table 20.3-B Metals Analysis by ICP/MS: Method 200.8 Internal Standard Criteria for CCV, CCB and samples; Determined by CLP Method				
Samples and QC samples	Isotope	Ref IS	Lower %	Upper %
Li	7	Sc	-	-
Be	9	Sc	-	-
B	11	Sc	-	-
Na	23	Y	-	-
Mg	24	Y	-	-
Al	27	Y	-	-
K	39	Y	-	-
Ca	44	Y	-	-
Sc (IS)	45	-	60	125
Ti	47	Y	-	-
V	51	Y	-	-
Cr	53	Y	-	-
Mn	55	Y	-	-
Fe	56	Y	-	-
Co	59	Y	-	-
Ni	60	Y	-	-
Cu	63	Y	-	-
Zn	66	Y	-	-
As	75	Y	-	-
Se	82	Y	-	-
Sr	87	Y	-	-
Y (IS)	89	-	60	125
Mo	98	Y	-	-
Ag	107	In (2), Y (3)	-	-
Cd	111	In	-	-
In (IS)	115	-	60	125
Sn	118	In	-	-
Sb	121	In (2), Y (3)	-	-
Ba	135	In	-	-
Tl	203	Bi	-	-
Pb	207	Bi	-	-
Bi (IS)	209	-	60	125

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
ICPMS tuning sample.	Prior to initial calibration and calibration verification.	RSD < 5%. Amu +/- 0.1 true value.	Retune instrument then reanalyze tuning solution.
Initial calibration (minimum 4 standards and a blank).	Daily initial calibration prior to sample analysis.	r > 0.998.	N/A.
Calibration Relative Error	Daily initial calibration prior to sample analysis.	<50% - low point <30% - mid point	N/A.
Initial Calibration verification (second source).	Daily after initial calibration,	All analytes within ±10% of expected value.	Correct problem and repeat initial calibration.
Calibration blank.	Before beginning a sample run, after every 10 samples and at end of the analysis sequence.	No analytes detected > 3 x IDL.	Correct problem then analyze calibration blank and previous 10 samples.
Calibration verification (Instrument Check Standard).	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence.	All analyte(s) within ±10% of expected value.	Correct problem then repeat calibration and reanalyze all samples since last successful calibration.
Demonstrate ability to generate acceptable accuracy and precision using four replicate LCS analyses.	Once per analyst.	All analyte(s) within ± 15% of the expected value.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria.
Method blank.	One per preparation batch.	No analytes detected > 3 x MDL.	Correct problem, re-digest and analyze method blank and all samples processed with the contaminated blank.

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Interference check solutions (ICS-A and ICS-AB).	At the beginning of an analytical run and every 8 hours.	ICS-A: All non-spiked analytes < ½ MQL; Spiked analytes within ±20% of true value. ICS-AB: Within ±20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples.
LCS for the analyte.	One LCS per preparation batch.	All analytes within ± 15% of the expected value for 200.8 and +/- 20% for 6020A.	Correct problem, re-digest and reanalyze the LCS and all samples in the affected preparation batch.
Dilution test.	Each preparatory batch.	5X dilution must agree within ±10% of the original determination for analytes present at concentrations > 100x concentrations found in reagent blank.	Perform post digestion spike addition for failed analytes.
Post digestion spike addition.	When dilution test fails.	Recovery within 80%-120% of expected results.	Dilute the sample; reanalyze post digestion spike addition.
MS/MSD	5% frequency for 6020A, 10% frequency for 200.8.	QC advisory acceptance criteria, 70% - 130% for 200.8. 75% - 125% for 6020A.	Describe in Laboratory Review Checklist.
Internal Standards (ISs).	Every sample.	Sample IS intensity: *SW 846-6020a samples must meet >70% criteria. EPA 200.8 samples must meet 60-125% criteria.*	Perform corrective action and/or dilution and reprocess all effected samples.
MDL study.	Performed Annually	Detection limits established shall be < 1/3 the MQLs in Tables 21.1	None.

Table 20.4
Summary of Calibration and QC Procedures for Method 200.8 & 6020A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
IDL study.	Performed Quarterly	Average of standard deviation of reagent blank analyzed 7 times on at least 3 non-consecutive days.	None.
Low-level Initial Calibration Verification (LLICV)	Performed daily after Initial calibration	70%-130% of expected value spike at MQL.	Correct problem and repeat initial calibration.
Low-level Continuing Calibration Verification (LLCCV)	Performed before analysis of samples and after every 10 samples in the sequence.	70%-130% of expected value spike at MQL.	Correct problem then repeat calibration and reanalyze all samples of similar concentration since last successful calibration verification.
Low-level Quality Control Sample (LLQC)	One LLQC per quarter.	70%-130% of expected value spike at MQL. Carried through entire preparation process.	Correct problem, re-digest and reanalyze. If problem cannot be corrected, spike at a higher concentration and update PQLs accordingly.

MICROWAVE ASSISTED ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS ANALYSIS BY ICP-MS SPECTROSCOPY

CEM-NPDES

SOPID:	VAL-MET-002	Rev. Number:	R01	Effective Date:	09/15/2021

Approved By: Jennifer Hall
Department Supervisor

Date: 9/15/21

Approved By:
QA Manager

Date: 09/15/21

Archival Date:		Doc Control ID#:		Editor:	

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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MICROWAVE ASSISTED ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS ANALYSIS BY ICP-MS SPECTROSCOPY

1) Scope and Applicability

1.1 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by inductively coupled argon plasma mass spectroscopy (ICP-MS). The procedure is an acid digestion for determining total available metals on samples requiring digestion by a CEM NPDES Metals digestion procedure (21.2).

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current standards in effect for the National Environmental Laboratory Accreditation Program (NELAP).

1.3 The analytes cited in the spiking solutions in Section 10 are the analytes currently validated and acceptable for this procedure.

1.4 Individual project requirements may override criteria listed in this SOP.

2) Summary of Procedure

2.1 A representative aliquot of aqueous sample is digested with a mixture of 3 mL concentrated nitric and 2 mL concentrated hydrochloric acid, in a 75-mL Teflon digestion vessel for 30 minutes using microwave heating. The sample is transferred to a clean sample vial for analysis.

3) Definitions

3.1 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.2 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.3 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.4 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.5 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.6 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.7 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an

analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.8 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP VAL-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Sample Preparation	**Hazards**	**Preventative Measures**
Acidification of samples prior to digestion.	Concentrated Acid	Wear gloves, safety glasses and lab coat, and face shield. Work in fume hood. Avoid skin contact with acids.
Opening of sample containers following digestion.	Pressurized Steam	Investigate digestion vessels for defects prior to use. Wear gloves, safety glasses and lab coat, and face shield. Work in fume hood. Allow vessels to cool prior to opening. Avoid skin contact with digestates.

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

6) Interferences

6.1 In order to identify problem matrices and method error, blanks, spikes, spike duplicates and check samples are run at regular intervals, as specified in each relevant analytical method.

6.2 Samples that are oily or continuously vent in the microwave shall be digested by an alternate hot block digestion method.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.
7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (VAL-QS-009).
7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances
7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Both plastic and glass containers are suitable.

8.2 Aqueous waste waters must be acidified to a pH of < 2 with HNO_3 at the time of sampling as a means of preservation. NOTE: Aqueous samples that arrive to the lab in an unpreserved state must be acidified prior to metals analysis. The Sample Receiving Dept. checks the pH of all preserved samples that arrive at the lab. If the pH is > 2, an aliquot of nitric acid is added to the sample and shall be placed hold. The pH of these samples must then be verified after 24-hours. If the pH is < 2, the sample is considered available for processing. If the pH is still > 2, additional nitric acid must be added and the pH verified < 2 after another 24-hour period. All pH readings must be documented in the Sample Receiving Preservation Log.

8.3 Samples and sample digestates may be stored at room temperature.

8.4 Samples must be digested within 180 days of collection.

9) Equipment and Supplies

9.1 Microwave with advanced composite digestion vessels, CEM Mars Express 6, or equivalent.

9.2 50-mL graduated cylinder, Class A. (note 9.2.1)

9.2.1 Alternatively, a 68 mL Class A Environmental Express digestion cup may be utilized to measure sample volume needed - Environmental Express SC-475, or equivalent.

9.3 65-mL Teflon vessels with pressure relief caps, or equivalent.

9.4 8 fl-oz. Polyethylene bottles, disposable, VWR #16059-068 or equivalent.

9.5 Bottle-top Acid Dispensers capable of dispensing 0.5 mL to 10 mL of acid- from Thermo, or equivalent.

9.6 Thermo Finntip tips, VWR catalog #53515-050 or equivalent.

10) Standards and Reagents

10.1 Acids used in the preparation of standards and for sample processing must be of high purity. Distilled acids are recommended.

10.2 Concentrated HNO_3 (Trace metal grade)

10.3 Concentrated HCl (Trace metal grade)

10.4 LCS and MS/MSD spiking solution (NIST traceable):

10.4.1 A 27 element standard is used each at 10 ppm, with the exception of the minerals (Fe, Ca, Mg, Na, and K at 1000 ppm) and Boron at 50 ppm. (Available from VHG, Custom Standard 901)

10.4.2 Ti Standard @ 10 ppm and Si Standard @ 50 ppm:

10.4.2.1 Prepare using NIST traceable 1000 ppm Ti and Si stock standards.

10.4.2.2 In a 500 ml volumetric flask, add 5 ml Ti stock standard, 25 ml Si stock standard, 10 ml HNO_3, and 5 ml HCl to 300 ml DI water.

10.4.2.3 Bring to final volume of 500 ml with DI.

10.4.3 LCS/MS/MSD Aqueous Spiking:

10.4.3.1 Add 500 µl of each spike solution (10.4.1 and 10.4.2) to 50ml reagent water (LCS) for method CEM-NPDES, and 50ml sample matrix (MS/MSD) for method CEM-NPDES that has been transferred to the digestion vessel.
10.4.3.2 Spiking must occur prior to the addition of any reagents.
10.4.3.3 Following digestion, the digestate is a final volume of 55 mL for method CEM-NPDES.

10.5 LLQC Spiking solution (NIST traceable):

10.5.1 Low-level Metals Mix Standard I w/ As, Ba, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, and V @ 0.5 mg/L **and** Be and Cd @ 0.2 mg/L **and** Al, Li, and Zn @ 1.0 mg/L **and** B @ 2.0 mg/L **and** Fe @ 8.0 mg/L **and** Mg, K, and Na @ 20 mg/L **and** Ca @ 50 mg/L. (available from VHG ZALSLAB1103-100 or equivalent)
10.5.2 Low-level Metals Mix Standard II w/ Sn @ 0.2 mg/L **and** Sb, Mo, and Ti @ 0.5 mg/L. (available from VHG ZALSLAB1104-100 or equivalent)
10.5.3 LLQC Aqueous Spike

10.5.3.1 Add 500 µl of each solution (10.5.1 and 10.5.2) to 50ml reagent water for CEM-NPDES, which has been transferred to the digestion vessel.
10.5.3.2 Spiking must occur prior to the addition of any reagents.
10.5.3.3 Following digestion, the digestate is a final volume of 55 mL for method CEM-NPDES.

11) Method Calibration

11.1 Perform support equipment (balances, etc.) calibration checks as required for daily use.

12) Sample Preparation/Analysis

12.1 Samples for dissolved metals analysis may be digested or analyzed directly depending on client requirements. Regardless of whether the samples are digested, a filtered MBLK and LCS must accompany all samples within a filter batch. This QC is to be processed in the same manner as the samples. It is recommended that samples requiring digestion be filtered in a separate batch from those being analyzed directly.

12.2 Any apparatus coming into contact with the sample (containers, graduated cylinders, etc) must be rinsed with both 10% HNO_3 and reagent water.

12.3 The MARS 6 is capable of holding up to 40 digestates at one time. Due to the volume of sample used for this procedure, the microwave can only digest 24 samples at one time. This enables the lab tech to digest up to 18 samples and all required batch QC. The microwave carrousel has 40 available positions in two separate rings. The outer ring has 24 available slots, and the inner ring has 16. The outer ring is the ring that is fully utilized and the inner ring is not utilized.

12.4 The microwave carrousel itself contains Kevlar liners for each available digestion slot. This Kevlar liner serves as a shield in case a digestion vessel ruptures. The vessels themselves have been designed with the pressure release system, so rupturing should be rare, but it is possible with different matrices.

12.5 Log into LIMS to create a batch of samples

12.6 Using a Class A graduated digestion cup (9.2.1), transfer a 50 mL (CEM-NPDES) representative aliquot of the well-mixed sample to a digestion vessel (9.3). Fill two digestion vessels with an equivalent amount of DI to be used for the MB and the LCS. NOTE: The number of the digestion vessel must be documented with sample ID of the sample it contains in the microwave digestion log. This is necessary because there is no place to record the sample number on the digestion vessel itself. Also, record the sample volume used in the appropriate column of the microwave digestion log.

12.7 Spike LCS and MS/MSD samples as described in section 10.4.

12.8 For samples to be analyzed by method CEM-NPDES, use a bottle-top dispenser to add 3.0 mL of concentrated nitric acid and 2.0 mL of hydrochloric acid to each digestion vessel. This step shall be performed in a fume hood because of the possibility of a vigorous reaction between the sample and concentrated nitric acid. If a vigorous reaction does occur, allow the sample to pre-digest before capping the vessel.

12.9 Screw a cap on each of the vessels ensuring that the caps are not cross threaded. Tighten the cap just hand tight. The threads will expand during heating in the microwave.

12.10 Transfer the vessels to the microwave carrousel, being careful to ensure they are in their properly designated positions.

12.11 The carrousel then fits onto the notch in the bottom of the microwave. The proper positioning of the carrousel enables it to spin.

12.12 Recall the stored program “MP_NPDES_W” from the stored methods menu. Review the method to make sure that changes were not inadvertently made to the program.

12.12.1 Program for Method CEM-NPDES

Stages:	1
Power:	1800W
Ramp Time:	0:00 minutes
Hold Time:	30:00 minutes
Temperature:	165°C
Temp Guard:	Off
Sample Size:	50 mL
Acid Added:	3 mL HNO3, 2 mL HCl
Final Volume:	55 mL

12.13 Verify that the fume hood is turned on and start the program.

12.14 Once the carrousel of samples has completed its program, verify that each vessel reached the required temperature within +/- 5 degrees by inspecting the run report.

12.15 Upon completion of the program, the microwave will enter a 15 minute cool-down program. Ensure that the microwave has been adequately vented of fumes prior to sample removal.

12.16 Carefully uncap and vent each sample in a fume hood.

12.17 Transfer the sample into an Environmental Express 68mL digestion vial. Label the vial with sample and batch IDs.

12.18 Verify that the final volume of the digestate is within 5 mL of the anticipated final volume. If greater than 5 mL was lost in the digestion process, repeat the digestion on

a new aliquot of sample. Excessive venting may indicate the need for the digestion of a reduced volume of sample or for the sample to be digested via the hot-plate digestion method.

12.19 Take the volume verified, labeled digestates to the Metals Lab for analysis, and provide a copy of the sample preparation documentation to the analysis laboratory for review.

13) Troubleshooting

13.1 Refer to determinative method or instrument documentation for guidance.

14) Data Acquisition

14.1 Sample preparation data recorded must be entered into the LIMS for later use in analytical and QC calculations. LIMS assigns a prep batch number for the data entered. Provide a copy of the sample preparation documentation to the analysis laboratory for review.

14.2 Sufficient information must be included in the comments section of the preparation documentation to reconstruct the reasoning behind any deviation from the initial volumes utilized within this SOP.

15) Calculation, and Data Reduction Requirements

15.1 LIMS utilizes instrument measurements in conjunction with the digestion data to perform calculations and reporting after analysis has been completed.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Method Blank (MBLK):

16.1.1 Preparation:

16.1.1.1 The method blank is a digestion tube containing 50 ml of DI water, digested with the same reagents as an actual sample.

16.1.2 Frequency:

16.1.2.1 One per batch of sample digestions or every 20 samples (whichever is less).

16.1.3 Acceptance Criteria:

16.1.3.1 All analytes of interest should be less than one-half the method reporting limit (MRL) and must be less than the MRL.

16.1.4 Corrective Action:

16.1.4.1 If the method blank results do not meet the acceptance criteria above, the laboratory must take corrective action to locate and reduce the source of the contamination and re-digest and reanalyze all samples associated with the failed method blank.

16.1.4.2 If samples cannot be re-run because of insufficient sample, a non-conformance/corrective action report must be initiated and issued to project management and to the QA Manager. The NCR/CAR must

provide sufficient detail for project narration and meet the requirements documented in VAL-QS-003.

16.1.4.3 Data reported with an associated contaminated method blank must be flagged with a “B”.

16.2 Laboratory Control Sample (LCS):

16.2.1 Preparation

16.2.1.1 Add 500 ul of the spiking solution (Section 10.4) to the method appropriate amount of DI water and process as a sample.

16.2.2 Frequency:

16.2.2.1 One set per batch of sample digestions or every 20 samples (whichever is less).

16.2.3 Acceptance Criteria and Corrective Actions:

16.2.3.1 Refer to the relevant section in the determinative method.

16.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD):

16.3.1 Preparation

16.3.1.1 Add 500 ul of spiking solution (Section 10.4) to the method appropriate amount of client sample and process as a sample.

16.3.2 Frequency:

16.3.2.1 Matrix spikes shall be prepared with each batch of client samples or every 10 samples (whichever is less) for Method 200.8.

16.3.3 Acceptance Criteria and Corrective Action:

16.3.3.1 Refer to the relevant section in the determinative method.

16.4 Sample Duplicate

16.4.1 Frequency:

16.4.1.1 A sample duplicate should be processed with each digestion batch.

16.4.1.2 A matrix spike duplicate may be substituted for the duplicate analysis unless required otherwise by project specifications.

16.4.2 Criteria and Corrective Actions:

16.4.2.1 Refer to the relevant section in the determinative method.

16.5 Low-Level Quality Control Sample (LLQC):

16.5.1 Preparation:

16.5.1.1 The LLQC is prepared by adding 500 µL of NIST traceable standard (section 10.5) to the method appropriate amount of DI and processing as a sample.

16.5.2 Frequency:

16.5.2.1 One LLQC per quarter. The results of the LLQC are used for determining the ongoing acceptability of results at similar concentration.

16.5.3 Criteria and Corrective Actions:

16.5.3.1 Refer to the relevant section of the determinative method.

16.6 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored for no less than 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the Run logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

17.4 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

17.5 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments in laboratory data review checklist for

exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) must be determined on an annual basis (at minimum) or whenever major modifications are performed.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R00	9/15/15	CES	New SOP
R01	9/15/21	PAL	Removed cover page graphics; clarified section 9.2.1

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, “Method 3015A Microwave Assisted Acid Digestion of Aqueous Sample and Extracts”, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW846 Update III, June 13, 1997.

21.2 Waste Water – NPDES, CEM Digestion Application Note, CEM Corportation, December 3, 2014.

21.3 ALS Environmental Quality Assurance Manual, Revision (most current)

DETERMINATION OF HEXAVALENT CHROMIUM BY ION CHROMATOGRAPHY

METHOD: EPA 218.6 / EPA 7199

SOPID: VAL-WC-013 Rev. Number: ~~R04~~ 05, LC 7/30/21 Effective Date: 07/31/2021

Approved By: Jennifer Heil Date: 7/30/21
Department Supervisor

Approved By: Date: 07/29/21
Technical Director / Quality Manager

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

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Signature	Title	Date

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1) Scope & Applicability

1.1 This Standard Operating Procedure (SOP) is based upon and compliant with EPA Method 218.6 and SW846 7199.

1.2 This method is applicable to the analysis of non-potable water.

1.3 The effective range of the method is 0.25 – 5.0 ug/L (ppb) for standard level analysis and 0.05 – 2.0 ug/L (ppb) for low-level analysis.

2) Summary of Procedure

2.1 A small volume of sample, 5 mL, is introduced into an ion chromatograph. $CrO4^{2-}$ is separated from other matrix components on an anion exchange column. $CrO4^{2-}$ is derivatized with 1,5-diphenylcarbazide in a post-column reactor and is detected spectrophotometrically at a wavelength of 530 nm.

3) Definitions

3.1 IC: Ion Chromatograph

3.2 ASTM Type II: De-ionized (DI) water meeting purity characteristics of ASTM Type II laboratory distilled water (resistance $\geq$ 10 MΩ-cm, anion free water).

3.3 Preparation Batch: A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix) processed under the same conditions within an 8 hour working shift.

3.4 Analytical Batch: A group of samples from one or more preparation batches, processed under the same sequence, within a 24 hour period.

3.5 Calibration Standard: A solution prepared from the primary dilution standard solution or stock standard solutions.

3.6 Initial Calibration Standards: A series of calibration solutions used to establish instrument calibration and develop calibration curves for individual target anions.

3.7 Initial Calibration Check Standard: An individual calibration solution prepared from a second source supplier and analyzed prior to sample analysis for the verification of the previously established calibration curve.

3.8 Continuing Calibration Check Standard: An individual calibration solution analyzed after every tenth field sample analyses to verify the previously established calibration curve on a continuing basis.

3.9 Field Duplicates: Two separate samples collected at the same time and place, under identical circumstances, and treated the same throughout field and laboratory procedures.

3.10 Sample Duplicate: Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures.

3.11 Laboratory Control Sample (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory.

3.12 Matrix Spike (MS): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory.

3.13 Matrix Spike Duplicate (MSD): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory and in identical concentrations as that of the MS.

3.14 Method Blank (MBLK): An aliquot of reagent water or other blank matrix prepared and analyzed in identical fashion as other samples.

3.15 Linear Calibration Range (LCR): The concentration range in which the instrument response is linear.

3.16 Material Safety Data Sheet (MSDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.17 Performance Evaluation (PE) Sample: A certified solution of method analytes whose concentration is unknown to the analyst.

3.18 Sample: Any field aliquot, however identified, submitted for analysis.

3.19 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the reporting limit (RL).

3.20 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.21 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136.

4) Responsibilities

4.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

4.2 Analyst - It is the responsibility of the analyst(s) to:

4.2.1 Each must read and understand this SOP and follow it as written. Any deviations or non-conformances must be documented and submitted to the QA Manager for approval.
4.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (VAL-QS-009).
4.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.
4.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
4.2.5 The analysts must submit data for peer or supervisor review.

4.3 Section Supervisor - It is the responsibility of the section supervisor to:

4.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.
4.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.
4.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.
4.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

4.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

4.5 QA Manager: The QA Manager is responsible for

4.5.1 Approving deviations and non-conformances
4.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,
4.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

5) Interferences

5.1 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in a chromatogram. These interferences can lead to false positives as well as reduced detection limits as a consequence of elevated baseline noise.

5.2 Close attention shall be given to the potential for carryover from one analysis to the next. It is the responsibility of the user to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

5.3 Matrix interferences are cause by contaminants that are present in the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the water. Matrix components may directly interfere by producing a signal at or near the retention time of the Cr(VI) peak; however, the method is extremely selective due to the chromatographic separation of the analyte from matrix components, coupled with the discrimination of the post-column reagent for the chromate anion. Sample ionic strength may enhance or suppress Cr(VI) response; however, the 4-mm column system used during method development tolerate typical concentrations of common anions in drinking water in combination with method preservative. Acceptable method performance has been demonstrated for samples with hardness up to 350 mg/L as CaCO3 and total organic carbon content of 3 mg/L.

5.4 To ensure sample integrity, Cr(VI) must be protected from reduction, and Cr(III), if present, must not oxidize to Cr(VI) during sample storage. Preservation of samples using the ammonium sulfate buffer to the required pH within 24 hours will minimize the oxidation of Cr(III) and prevent the reduction of Cr(VI).

6) Safety

6.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

6.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses

6.3 Hexavalent chromium is toxic and a suspected carcinogen and should be handled with appropriate precautions.

6.4 Preparation of the post-column reagent and the ammonium hydroxide preservative require the use of concentrated acid and concentrated base. These reagents should be prepared in a hood, adding acid to water, and wearing a full-face shield with chemical

resistant gloves.

6.5 This task may include CHEMICAL, BIOLOGICAL, OPERATIONAL and/or EQUIPMENT hazards. Staff must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

HAZARD ASSESSMENT		
Job Task #1: Sample Preparation	**Hazards**	**Preventative Measures**
Sample filtration	Filter rupture	Wear gloves, safety glasses and lab coat. Avoid skin contact with samples.
Spiking QC samples	Carcinogenic standard	Wear gloves, safety glasses and lab coat. Avoid skin contact with samples.
Job Task #2: Preparing Reagents and Standards	**Hazards**	**Preventative Measures**
Standard 1	Acid	Wear appropriate PPE including face shield, prepare in hood, add acid to water.
Standard 2	Base	ear appropriate PPE including face shield, prepare in hood, add base to water.

6.6 Hazard information related to this activity, which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Samples may be collected in either plastic or glass bottles. Volume collected shall be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal. A volume of 250 mL is sufficient.

7.2 Samples shall be filtered through 0.45 um filters, and preserved with 1.0 mL $NH_4OH/(NH_4)_2SO_4$ preservative (Section 9.9) per 100 mL sample.

7.2.1 When samples are received in the lab, samples are verified for filtration using the COC and checked for pH. If the pH is not within 9 - 9.5, the samples shall be immediately adjusted for pH. Sample pH shall be adjusted to 9.3 – 9.7 by adding ammonium sulfate buffer (Section 9.9) within 24 hours of collection. For further guidance in pH adjustment, see Section 7.4.3.

7.3 Samples must be placed on ice upon collection and maintained at a temperature of $\leq$ 6˚C without freezing.

7.4 Samples have a holding time of 24 hours.

7.4.1 40 CFR 136 preservation guidelines allow for an extension of holding time to 28 days for compliance samples.

7.4.2 To achieve the 28-day holding time, use the ammonium sulfate buffer solution (Section 9.9) to adjust the sample to a pH of 9.3 – 9.7.

7.4.3 When adjusting the pH using the ammonium sulfate buffer, if the pH range cannot be achieved without diluting the sample by more than 10%, use a minimum amount of ammonium hydroxide (Section 9.2), supplemented by buffer solution (Section 9.9) as necessary.

8) Apparatus and Equipment

8.1 Ion Chromatograph—Analytical system complete with ion chromatograph and all

required accessories including syringes, analytical columns, compressed gasses, UV-Visible detector, and external pump apparatus.

8.1.1 Anion guard column: Dionex NG-1, 2 X 50 mm (P/N 088763) or equivalent.
8.1.2 Anion separator column: Dionex AS-7 column, 2 X 250 mm (P/N 063097), or equivalent.
8.1.3 UV-Visible detection device: Dionex ICS Variable Wavelength Detector, or equivalent.
8.1.4 External pump: Dionex AXP model, or equivalent.
8.1.5 Instrument Parameters - The following parameters serve as baseline for system operations. For optimized performance, fine adjustments may be made under the supervision of a skilled analyst:

8.1.5.1 Eluent pump flow rate= 0.36 mL/min
8.1.5.2 Post-column reagent flow rate = 0.20 mL/min
8.1.5.3 Pressure Limits= 1500-2500 psi

8.2 Computer software capable of processing all associated data:

8.2.1 Dionex Chromeleon, version 7.0, or equivalent.

8.3 Computer capable of processing all associated software- Dell Dimension 8800, or equivalent.

8.4 Analytical balance capable of weighing to 0.0001 gram - OHaus Adventurer PA124, or equivalent.

8.5 Weigh boats, plastic, disposable- VWR catalog #12577-005, or equivalent.

8.6 Syringe capable of delivering 10 µL- VWR catalog #0376-183, or equivalent.

8.7 Glass A volumetric flasks and pipets as required- VWR, or equivalent.

8.8 Bottles, high-density polyethylene (HDPE), opaque or glass, amber 250-mL, for sampling and storage of calibration solutions, or equivalent.

8.9 Automatic pipet capable of dispensing 0.200 mL to 1.00 mL- Eppendorf variable volume 2000, VWR catalog #53511-582, or equivalent.

8.10 Automatic pipet capable of dispensing 0.020 mL to 0.200 mL- Eppendorf variable volume 200, VWR catalog #53513-408, or equivalent.

8.11 Automatic pipet capable of dispensing 2.0 mL to 10.0 mL- Finn variable volume pipet, VWR catalog #53515-050, or equivalent.

9) Standards and Reagents

9.1 Reagent water: Distilled or de-ionized water.

9.2 Ammonium hydroxide (NH_4OH)- J.T. Baker Catalog # JT9731-3, or equivalent.

9.3 Ammonium Sulfate [$(NH_4)_2SO_4$)]- VWR Catalog # BDH9216 (500g), or equivalent.

9.4 Methanol (CH_3OH), HPLC grade- VWR Catalog # BDH85681.400, or equivalent.

9.5 1,5-Diphenylcarbazide (1,5-Diphenylcarbohydrazide)- Manufactured by J.T. Baker, purchased from VWR catalog #K620-03, or equivalent.

9.6 Sulfuric Acid (Conc.) - VWR catalog #EMD SX1244-5, or equivalent.

9.7 Buffered DI (used for blanks and dilutions) – adjust the pH of approximately 1L of filtered

DI water within the range of 9.3 – 9.7 using the buffer solution (9.9). The pH range was set as per footnote 20 of 40 CFR part 136 Table II EPA Method Update Rule and EPA FAQ-Cr6. The solution is to be used for diluting working standards and high level samples, as well as for blank QC samples. Prepare fresh before use.

9.8 Post-column reagent – Add 28 mL of sulfuric acid to approximately 500 mL of reagent water in a 1-liter volumetric flask. Mix and cool to room temperature. While this solution is cooling, weight 0.50 grams of 1,5-Diphenylcarbazide into a 100-mL beaker, add 75 mL of methanol, and allow the solid to dissolve. Transfer this solution to a 100-mL volumetric flask; bring to volume with methanol and mix. Add the entire contents of the volumetric flask to the sulfuric acid solution and dilute to 1.0 L with reagent water. Store at room temperature for up to five days after preparation.

9.9 $NH_4OH/(NH_4)_2SO_4$ preservative—dissolve 3.3 g Ammonium Sulfate in 75 mL of reagent water. Add 6.5 mL of Ammonium Hydroxide and dilute to 100 mL. Store at room temperature for up to one month.

9.10 Working Eluent Solution—dissolve 66 g Ammonium Sulfate in approximate 1 L of reagent water. Add 13 mL of Ammonium Hydroxide and dilute to 2 L. Store at room temperature for up to 1 month.

9.11 Hexavalent Chromium Stock Solution (1000 mg/L) – purchased from VWR catalog #BDH 82025-970, or equivalent. Store refrigerated above the freezing point of water up to 6ºC until manufacturer's expiration date.

9.11.1 **NOTE** – All stocks and standards derived from this solution and its derivative are to be preserved with 1.0 mL of NH4OH/(NH4)2SO4 preservative per 100 mL, and stored refrigerated above the freezing point of water up to 6ºC for up to 1 month.

9.11.2 Calibration Stock (1000 µg/L) - Dilute 0.1 mL of Hexavalent Chromium Stock solution to 100 mL.

9.11.2.1 Calibration Standards – prepared in 100 mL volumetric flasks. Dilute calibration stock (9.11.2) in amounts according to chart below, and dilute to 100 mL.

Level	Volume added (mL)	Cr(VI) concentration (µg/L)
1	0	0
2*	5**	0.25
3	10**	0.50
4	0.100	1.00
5	0.200	2.00
6	0.500	5.00

* - serves as Minimum Reportable Level (MRL) standard

** - The volumes outlined are taken from a dilution of the Calibration Stock Solution: Dilute 0.5 mL of Calibration Stock in 100 mL of reagent water in a volumetric flask.

9.11.2.2 Continuing Calibration Verification standard, (CCV 1.0 µg/L): Dilute 0.1 mL of Calibration Stock in 100 mL of reagent water in a volumetric flask.

9.11.3 Low-Level Calibration Stock (10 µg/L) – to a 100 mL volumetric flask, add 1 mL of 1 ppm stock solution (9.11.2) along with 1 mL of buffer (9.7) and dilute with DI water (9.1) to the line.

9.11.3.1 Low-Level Calibration Standards – prepared in 100 mL volumetric flasks. Dilute low-level calibration stock (9.11.3) in amounts according to chart below, and dilute to 100 mL.

Level	Volume added (mL)	Cr(VI) concentration (µg/L)
1	0	0
2*	0.2 (10ppb stock)	0.05
3	0.5 (10ppb stock)	0.10
4	1.0 (10ppb stock)	0.20
5	2.0 (10ppb stock)	0.50
6	0.10 (1ppm stock)	1.00
7	0.20 (1ppm stock)	2.00

* - serves as Minimum Reportable Level (MRL) standard

9.11.3.2 Low-Level Continuing Calibration Verification standard, (CCV 0.40 µg/L): Dilute 0.2 mL of 1 ppm Calibration Stock in 500 mL of reagent water in a volumetric flask.

9.12 Hexavalent Chromium Verification Stock Solution (1000 mg/L) – purchased from High Purity catalog #100012-7, or equivalent. Store refrigerated above the freezing point of water up to 6ºC until manufacturer's expiration date.

9.12.1 Quality Control Stock (1000 µg/L) – Dilute 0.10 mL of Hexavelent Chromium Verification Stock Solution to 100 mL. Preserve with 1.0 mL of $NH_4OH/(NH_4)_2SO_4$ preservative and store refrigerated above the freezing point of water up to 6ºC for up to 30 days.

9.12.2 Initial Calibration Verification Stock (ICV, 10 µg/L) – Dilute 1.0 mL of Quality Control Stock (9.12.1) to 100 mL. Preserve with 1.0 mL of $NH_4OH/(NH_4)_2SO_4$ preservative and store refrigerated above the freezing point of water up to 6ºC for up to 1 month.

9.12.3 (Low-Level Curve) Initial Calibration Verification Standard (LLC-ICV, 0.50 µg/L) – Dilute 5 mL of Initial Calibration Verification Stock (9.12.2) to 100 mL. Store refrigerated above the freezing point of water up to 6ºC for up to 1 month.

9.13 Laboratory Control Sample (1.0 µg/L): Pipet 0.05 mL of Calibration Stock Solution (9.11.2) into 50 mL of sample.

9.14 Low-Level Laboratory Control Sample (0.40 µg/L): Dilute 0.20 mL of 1 ppm Cal. stock in 500 mL of reagent water in a volumetric flask.

9.15 Matrix Spike (2.0 µg/L): Pipet 0.1 mL of Calibration Stock Solution (9.11.2) into 50 mL of sample.

9.16 Low-Level Matrix Spike (0.25 µg/L): Pipet 0.625 mL of Low-Level Calibration Stock Solution (9.11.3) into 25 mL of sample.

10) Calibration

10.1 Prepare the series of working calibration standards listed in Section 9.11.2.1 or 9.11.3.1. All standards must be recorded in the standards logbook.

10.2 In the Chromeleon schedule, identify the standards as Level 1 through Level 6 calibration standards and analyze them (or Level 1 through Level 7 low-level calibration standards for low-level analysis). An Initial Calibration Verification (ICV), being of a second source

from the curve standards, must be analyzed immediately after the calibration and must pass quality control acceptance criteria. If the ICV fails, the curve points must be reanalyzed for a new calibration.

10.3 Following analysis, access the method in the Chromeleon Method Editor section. Access the component table. Detail each analyte and select the curve type linear. All 5 (5) data points must be used in the curve. The linearity for each analyte shall be ≥ 0.995 (include origin).

10.4 Calculate the relative error of the low-point and mid-point calibration standards against the true values. The calculated relative error of the low-point standard must fall within +/-50%. The calculated relative error of the mid-point standard must fall within +/-10%. If the relative error of either point falls outside of the acceptance criteria, determine the cause of the problem, correct, and repeat the calibration sequence.

10.5 Print a copy of the calibration curve plots to include with the calibration standards analysis.

10.6 A new calibration shall be performed weekly.

11) Procedure

11.1 Instrument Start-Up

11.1.1 Fill the eluent reservoir with eluent, and the external pump tank with post-column reagent. Set the external pump flow to 0.36 mL/min.

11.1.2 In the software program, start the instrument and allow the baseline to stabilize. Refer to the instrument's user manual for details. The pressure shall come up to about 1500 psi or more. The flow shall read approximately +- 0.02 mL/minute from the calibrated flow rate.

11.1.3 Initiate the Analysis Coversheet for this analysis, recording the methods used in the analytical run, standard and reagent numbers, pipette numbers used for preparations and dilutions, instrument and batch numbers, analyst information, etc.

11.2 Initialize the Program, Quantification Method and Sequence Method

11.2.1 From the software program, prepare the instrument with the Program for 218.6 analysis. Refer to the instrument's user manual for details.

11.2.2 From the software program, select the proper program for the Quantification of 218.6 Cr (VI). Refer to the instrument's user manual for details.

11.2.3 From the software program, prepare the Sequence for the analysis. Refer to the instrument's user manual for details.

11.2.4 Name the sequence YYMMDD.seq.

11.2.5 In the Dilution Factor column, enter "1". The LIMS data transfer will pull the dilution factor from the sample name.

11.2.6 If the IC should shut down after the analysis run, the last line of the schedule shall have a name of "Shutdown", and the shutdown method (ShutdownCr6.pgm) under the method column.

11.2.7 An example of the sequence of samples and QC checks is as follows:

11.2.7.1 Calibration Standards 1-6
11.2.7.2 ICV
11.2.7.3 ICB
11.2.7.4 LLICV/LLCCV
11.2.7.5 CCV/LCS

11.2.7.6 CCB/MBLK
11.2.7.7 6 Samples
11.2.7.8 MS
11.2.7.9 MSD
11.2.7.10 CCV
11.2.7.11 CCB
11.2.7.12 10 Samples
11.2.7.13 CCV
11.2.7.14 CCB
11.2.7.15 8 Samples
11.2.7.16 MS
11.2.7.17 MSD
11.2.7.18 CCV
11.2.7.19 CCB
11.2.7.20 LLCCV
11.2.7.21 Shutdown

11.2.8 Save the schedule. Click File/Save As. Name the file by the date followed by run letter. For example, the file name for the first schedule run on March 15, 2018 would be 180315A.seq.

11.2.9 Print a hard copy of the schedule to include it in the data package.

11.3 Sample Preparation

11.3.1 Check the sample histories in LIMS for estimated dilutions based on previous analyses. Samples that are suspected to be high in anion concentrations shall also be diluted. Keep detection limits in mind when setting up dilutions, as the dilution will raise the detection limits by that factor.

11.3.2 All samples must be at room temperature at the time of analysis. Ensure this by having all samples sit at room temperature for at least one hour prior to beginning analysis.

11.3.3 Note the date and time for each sample and schedule the samples to run in an order that the hold times will be met.

11.3.4 Samples containing finely divided particles require pre-filtering through a 0.45-micron filter. The use of a 0.45-micron filter syringe is an acceptable procedure for filtering. When a sample is filtered, all associated quality control standards and blanks must be filtered in the same fashion.

11.3.5 Write sample # on vial, pour to vial mark. Place vial cap on top of vial, invert twice to mix, and using the tool, press the cap down into the vial ending with the cap flush with the top of the vial. Samples may need to be diluted in order to ensure that results fall within calibration range.

11.3.6 Refer to the sequence in the schedule and position the samples in the appropriate tray position.

11.3.7 Load the trays into the autosampler with the black dots lining up on the right-hand side of the delivery bin. If using a carousel model autosampler, place tubes in carousel slots corresponding to those listed in the analytical sequence.

11.3.8 Select the sequence and click “start” in order to start the analysis.

11.4 Data Review

11.4.1 Review the sequence to make certain that all samples were identified properly, all dilution factors were correct, and the correct method calibration was used to process the samples.

11.4.2 Review each sample report and its chromatogram to make certain that the retention time windows have properly identified each analyte. If peaks have not been identified properly, they can be renamed through the Optimize/Name Peaks menu item. If this option is used, both the original and modified chromatogram must be included with the raw data. If the identity of a peak is in question, the sample shall be spiked and reanalyzed for confirmation.

11.4.3 Review each sample chromatogram to make certain that baselines are drawn correctly. If modifications are required, they can be made through the Optimize/Adjust Baseline menu item. Whenever manual integrations are performed both the original and modified chromatogram must be included with the raw data. A comment shall also be included as to the reason for the manual integration.

11.4.4 Review each sample report to make certain that the quantified concentrations are within the lowest and highest calibration points. If results are beyond the highest standard, rerun the sample at a dilution.

11.4.5 Review all quality control samples for comparison to the acceptance criteria in Section 13.

11.4.6 Review prep and analytical dates and times for each sample to make certain that all analytes were analyzed within the appropriate hold times. Any sample prepared or analyzed after the holding time shall be qualified by adding an analyte specific comment in LIMS.

11.4.7 All quality control samples, method blanks, LCS, etc., must undergo all preparation and analytical steps that are performed. For example, if client samples are filtered prior to analysis the QC samples must be filtered also.

12) Maintenance and Troubleshooting

12.1 Refer to maintenance logs and instrument manuals for guidance regarding general maintenance and troubleshooting specific problems related to the instrumentation used in this method.

12.2 Routine Maintenance

Frequency	Task	Action
Daily	Check for leaks	Find source and repair
Weekly	Check lines for crimping and discoloration	Replace as needed
Weekly	Inspect guard column frit for discoloration	Replace as needed
Monthly	Injection valve	Clean
Monthly	Autosampler line	Replace
Annually	Waste lines	Replace

13) Quality Assurance/Quality Control Requirements

13.1 All policies and procedures in the most current revision of ALS-VAL-QAM shall be followed when performing this procedure.

13.2 Quality Control Requirements:

QUALITY CONTROL REQUIREMENTS

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Initial/Continuing Calibration Blank (ICB/CCB)	N/A	Beginning of batch, after every 10 samples, and conclusion of batch, following CCV.	< MDL	Positive blank indicates laboratory or reagent contamination. Contamination must be identified and eliminated before analyzing samples. (See Section 8.3)
% Relative Error of Calibration	Low-point and Mid-point calibration standards	After initial calibration.	Low-point: +/-50% Mid-point: +/-10%	Repeat calibration.
Initial Calibration Verification (ICV) (Second Source)	1.0 µg/L (standard-level) 0.5 µg/L (low-level)	After initial calibration.	90-110 %	Repeat the analysis using a fresh std. If the result is still unacceptable the cause must be determined and resolve before sample analysis.
Minimum Reportable Level (LLICV/LLCCV)	0.25 µg/L (standard-level) 0.02 µg/L (low-level)	At the beginning and end of each analytical sequence.	50-150 %	Repeat the analysis using a fresh std. If the result is still unacceptable the cause must be determined and resolved before sample analysis.
Continuing Calibration Verification (CCV)	1.0 µg/L (standard-level) 0.4 µg/L (low-level)	At the beginning of each analytical sequence, following every tenth sample, and conclusion of sequence.	95-105 %	Repeat once using a fresh standard. If still unacceptable reanalyze all associated samples.
Method Blank (MBLK)	0 µg/L	One per each analytical batch of no more than 20 samples.	< Reporting Limit (0.25 µg/L) or < 1/10th sample concentration. Note: *<MDL for West Virginia Compliance*	Repeat the analysis. If still unacceptable reanalyze all associated samples.
Laboratory Control Sample (LCS)	1.0 µg/L (standard-level) 0.4 µg/L (low-level)	One per each analytical batch of no more than 20 samples.	90-110 %	Repeat the analysis. If the result is still unacceptable the cause must be determined and resolved before sample analysis.

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Matrix Spike (MS)	Sample concentration + : 2.0 µg/L (standard-level) 0.25 µg/L (low-level) (dilution corrected)	One per every 10 samples with at least one per batch	90-110 %	If all other quality control parameters are in control the problem is judged to be matrix related. Report with a qualifying statement.
Matrix Spike Duplicate (MSD)	Sample concentration + : 2.0 µg/L (standard-level) 0.25 µg/L (low-level) (dilution corrected)	One per every 10 samples	90-110% RPD ≤ 20 %	Accuracy: If all other quality control parameters are in control the problem is judged to be matrix related. Report with a qualifying statement. Precision: Repeat analysis of parent and MS/MSD, and all associated samples.

13.3 Method Blanks

13.3.1 If the method blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of contamination must be investigated, and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

13.3.1.1 **For West Virginia compliance samples, the method blank must be evaluated to the method detection limit. If the method blank concentration is greater than MDL AND is greater than 1/10 the sample concentration, the source of contamination must be investigated, and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.**

13.4 Matrix Spike and Duplicate

13.4.1 Samples selected for spike and duplicate analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

13.5 Acceptance limits were developed based on the reference methods.

13.6 MDL

13.6.1 MDL studies must be performed according to SOP VAL-QS-006, Method Detection and Quantitation Limits, or the reference method, whichever is more frequent. For this method, the MDL study must be conducted annually. Seven (7) replicates of the MDL standard are analyzed over the course of 3 days.

13.6.2 Prepare the MDL standards at the concentration of the reporting limit. The suggested MDL level is based on historical results. The level can be adjusted to achieve optimal results.

13.6.3 Conduct the MDL analyses and evaluate the results according SOP VAL-QS-006, Method Detection and Quantitation Limits.

13.7 Demonstration of Capability

13.7.1 Analyze four replicates of the Laboratory Control Standard. All four results shall be within 10% of the true value. If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples begins.

13.7.2 Using the data generated in Section 13.7.1, calculate the percent relative standard deviation (%RSD) of the replicate analysis as indicated below. To be acceptable, the %RSD must be less than 20%.

$$\%RSD = \frac{Sample\ Standard\ Deviation\ (S)}{Mean\ Recovered\ Concentration}$$

If the standards analyzed do not meet this requirement, the DOC shall be repeated and within requirements before independent analysis of samples is begun.

13.7.3 Ongoing proficiency must be established annually.

13.8 Retention Times

13.8.1 The retention time for an analyte will be established during a new calibration. The retention time window will be based on the retention times of calibration standards used for that current calibration.

13.8.2 The width of the retention time window used to make identifications shall be based upon actual measurements of retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst shall weigh heavily in the interpretation of chromatograms.

14) Data Reduction and Reporting (or Documentation and Records)

14.1 Calibration Relative Error Calculation

$$\%\ Relative\ Error = (x'_i - x_i) / x_i * 100$$

Where:
x_i = True value for the calibration standard.
x'_i = Measured concentration of the calibration standard.

14.2 QC Calculations: LIMS calculates the percent recovery for various QC samples (MS, MSD, LCS) according to the following equations:

14.2.1 % Recovery, %R (for MS and MSD Samples)

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SR = Sample Result (unspiked).
SA = Spike Amount Added (mg/L or mg/kg).

14.2.2 % Recovery, %R (for standards and LCS)

$$\%R = \frac{(SSR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SA = Spike Amount Added (mg/L or mg/kg).

14.2.3 RPD (for precision or duplicate evaluation)

$$RPD = \frac{|SR_1 - SR_2|}{½(SR_1 + SR_2)} \times 100$$

Where:
SR_1 = Sample result for duplicate 1.
SR_2 = Sample result for duplicate 2.

14.3 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

14.4 For samples diluted due to matrix interferences, the reporting limits are also increased by the sample dilution factor.

14.5 When entering data into LIMS do not round off results: LIMS will automatically perform rounding appropriate to the method. LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

14.6 Report the actual result, even if it is less than the reporting limit.

14.7 All data is stored both electronically and hard copy for no less than 10 years.

14.8 Complete all pertinent sections in the respective logbooks. If not applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.

14.9 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.

14.10 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

15) Method Performance

15.1 Method Detection Limits (MDLs) must be determined and/or verified on an annual basis (at minimum) or whenever major modifications are performed.

15.2 MDL verification shall be completed every 3 months according to the guidelines documented in SOP VAL-QS-006, Method Detection and Quantitation Limits. The QA Manager shall maintain documentation.

15.3 Performance evaluation shall be conducted semi-annually through the analysis of performance evaluation samples. The QA Manager shall maintain documentation.

16) Pollution Prevention

16.1 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and

reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

17) Waste Management

17.1 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP VAL-SAF-001, Waste Disposal Procedures, must be followed.

18) Corrective Actions for Out-of-Control Data

18.1 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

19) Contingencies for Handling Out-of-Control or Unacceptable Data

19.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

19.2 When affected sample analysis cannot be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

19.3 All analysts must report sufficient comments in LIMS for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

19.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS.

19.5 The QA Staff must conduct periodic audits to evaluate compliance with this SOP.

20) Training

20.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

20.2 Each analyst must demonstrate ongoing proficiency annually by generating data of acceptable accuracy and precision for target analytes in a clean matrix or by performance of an approved PE sample.

21) Summary of Changes

Table 20.1 - Summary of Revision Changes			
Revision Number	Effective Date	Document Editor	Description of Changes
R01	9/25/19	LC	Added low-level analysis details
R02	1/20/20	LC	Added calibration relative error check
R03	7/10/20	LC	Updated 9.9 to 33g ammonium sulfate.
R04	1/26/21	CES	Added Section 7.3 for thermal preservation ≤ 6˚C
R04	1/26/21	CES	Table 13.2 updated to assess ICB/CCB to MDL and MBLK to MDL for West Virginia Compliance samples.
R05	7/31/21	PAL	Update std range (9.11.3.1)

22) References and Related Documents

22.1 U.S. Environmental Protection Agency, Method 218.6 rev. 3.3, May 1994.

22.2 U.S. Environmental Protection Agency, Method 7199 rev. 0, December 1996.

22.3 ALS Environmental Quality Assurance Manual, Revision (most current)

22.4 40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

22.5 U.S. EPA Region5 LSASD Analytical Services Branch, SOP# AIG032A, Rev. 8, April 28, 2020

TOTAL SUSPENDED SOLIDS

EPA 160.2 / SM 2540 D

SOPID:	VAL-WC-001	Rev. Number:	R03	Effective Date:	07/31/2021

Approved By: Jennifer Hall — Department Supervisor — Date: 7/30/24

Approved By: [signature] — QA Manager — Date: 07/30/21

Approved By: [signature] — General Manager — Date: 7/30/2024

Archival Date: _____ Doc Control ID#: _____ Editor: _____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date

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TOTAL SUSPENDED SOLIDS

1) Scope and Applicability

1.1 These procedures are used to determine the amount of suspended (non-filterable) solids present in a sample.

1.2 These methods are suitable for the determination of solids in drinking, surface, and ground waters, as well as domestic and industrial wastewaters.

1.3 The method reporting limit is volume dependent. A prep batch must be utilized to account for changes in sample volume.

2) Summary of Procedure

2.1 Total Suspended Solids

2.1.1 A well-mixed sample is filtered through a standard glass fiber filter. The residue retained on the filter is dried to a constant weight at 104±1°C. The increase in the weight of the filter represents the total suspended solids, adjusted for the volume filtered.

3) Definitions

3.1 Solids: The term "solid" refers to matter that is suspended or dissolved in water or wastewater.

3.2 Fixed Solids: Fixed solid is the term applied to the residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature.

3.3 Analytical Batch: A group of 20 or fewer field samples of the same matrix processed during the same working shift.

3.4 Laboratory Control Sample (LCS): An analyte-free matrix spiked with known concentrations of all target analytes. This is used to evaluate and document laboratory method performance.

3.5 Matrix: The component or substrate (e.g., surface water, groundwater, soil) which contains the analyte of interest.

3.6 Matrix Spike (MS): An aliquot of background sample spiked with a known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.

3.7 Matrix Spike Duplicate (MSD): A duplicate aliquot of the background sample spiked with a known concentrations of all target analytes. Spiking occurs prior to sample preparation and analysis. The MS/MSD pair are used to assess precision and bias of a method in a given sample matrix.

3.8 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.9 Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ is also referred to as the method quantitation limit (MQL) or the

reporting limit (RL).

3.10 Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

3.11 Method Detection Limit (MDL) study: the procedure, as described in 40CFR part 136, for determining the LOD based on statistical analysis of 7 low-level replicate spikes.

4) Health and Safety Warnings

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons must be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Management: The principal wastes generated by this procedure are the method-required chemicals and standards. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required. Laboratory procedures in SOP VAL-SAF-001, Waste Disposal Procedures, must be followed.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be disposed.

5) Cautions

5.1 Use caution when removing samples from the ovens, as they will be hot.

5.2 Be knowledgeable of the MSDS information for each chemical used in this procedure.

6) Interferences

6.1 Large floating particles or other non-homogeneous materials should be excluded if they are not representative of the sample.

6.2 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material.

7) Personnel Qualifications and Responsibilities

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Each must read and understand this SOP and follow it as written. Any

deviations or non-conformances must be documented and submitted to the QA Manager for approval.

7.2.2 Produce method compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP (VAL-QS-009).

7.2.3 Complete the required initial demonstration of proficiency before performing this procedure without supervision.

7.2.4 Complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.2.5 The analysts must submit data for peer or supervisor review.

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required initial demonstration of proficiency before performing this procedure without supervision.

7.3.3 Ensure analysts complete an ongoing demonstration of proficiency annually when continuing to perform the procedure.

7.3.4 Ensure analysts produce method compliant data that meet all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.4 Project Manager - It is the responsibility of the Project Manager to ensure that all method requirements for a client requesting this procedure are understood by the laboratory prior to initiating this procedure for a given set of samples.

7.5 QA Manager: The QA Manager is responsible for

7.5.1 Approving deviations and non-conformances

7.5.2 Ensuring that this procedure is compliant with method and regulatory requirements,

7.5.3 Ensuring that the analytical method and SOP are followed as written through internal method and system audits.

8) Sample Collection, Handling, and Preservation

8.1 Sample collection bottles – plastic or glass, approximately 1L. These are purchased by the laboratory and meet EPA specifications for sample containers.

8.2 Preserve the samples with refrigeration at 4±2°C from the time of collection until analysis is performed.

8.3 Samples must be analyzed within 7 days from the time of collection.

9) Equipment and Supplies

9.1 Desiccator – with indicating desiccant

9.2 Analytical balance – capable of weighing to the nearest 0.0001g

9.3 Oven at 104 ± 1°C

9.4 Aluminum weighing dishes

9.5 Graduated cylinders, 10mL to 1L, Class A

9.6 Magnetic filter holder funnel

9.7 Glass microfiber 1.5 um pre-washed/double-weighed filters – ProWeigh Double from

Environmental Express

9.8 Vacuum pump

9.9 Tubing

9.10 Wide bore pipet tips

10) Standards and Reagents

10.1 DI water – ASTM Type II or better

10.2 TSS Mid-level Standard @ 100 mg/L – purchased from a commercial vendor.

11) Method Calibration

11.1 Balances must be checked daily with Class "S" weights using 100g, 1g, and 0.1g weights and recorded in the balance calibration logbook for each balance.

11.2 Oven thermometers must be calibrated annually against an NIST certified thermometer in the range of interest. Annual calibrations are recorded in an electronic logbook.

11.3 Daily oven temperatures must be recorded on the temperature monitoring logs as read from the thermometer and with any applicable correction factors applied.

12) Sample Preparation/Analysis

12.1 Preparation of glass fiber filters (TSS):

12.1.1 Pre-washed and pre-dried filters are purchased from an outside vendor (Environmental Express).

12.2 Sample Analysis – TSS

12.2.1 Record the weight of the pre-weighed filter in the appropriate computer-generated worksheet.
12.2.2 Assemble the filtering apparatus.
12.2.3 Place the filter, wrinkle side up into the filtration apparatus and turn on the vacuum.
12.2.4 Shake the sample vigorously and transfer a portion (*see note*) to the funnel using a graduated cylinder. The sample volume should be such that 2.5-200 mg of residue can be obtained. Record the sample volume used in the LIMS prep log and on the Excel spreadsheet.

Note: The analyst must vary the sample volume used in order to obtain 2.5-200 mg of residue. The volume should be maximized to obtain sufficient residue, up to 1L of sample.

12.2.4.1 For the TSS test, a Method Blank and LCS must be run.
12.2.4.2 Method Blank: 1000 mL of DI water.
12.2.4.3 LCS Preparation: Pour off 100 mL of the 100 mg/L TSS standard (10.2) and filter. Theoretical concentration = 100 mg/L.

12.2.5 Filter the sample through the glass fiber filter and rinse the graduated cylinder and funnel with 3 aliquots of DI water. Continue to apply the vacuum after the filtration is complete to remove as much water as possible. If the filtration takes >10 minutes, do not add anymore volume and document ">10 min" on the spreadsheet. Replace with new filter and use less sample volume to complete in less than 10 minutes.

12.2.6 Turn off the vacuum and carefully remove the filter. Replace filter in the aluminum weighing dish and place in the oven at 104±1°C.

12.2.7 Drying Cycles and Weigh Backs:

12.2.7.1 First drying cycle: into oven at 104±1°C for 90 minutes; remove and place in desiccator for 30 minutes; weigh.

12.2.7.2 Second drying cycle: into oven at 104±1°C for 45 minutes; remove and place in desiccator for 15-30 minutes; weigh.

12.2.7.3 Third drying cycle: the weight change must be < 4% or 0.5mg (whichever is smaller) of the previous reading. If this is not obtained, a third drying cycle will need to be performed.

12.2.8 If samples must be dried additional times, a 2nd spreadsheet shall be used to document the re-dry times and analyst performing the re-dry.

13) Troubleshooting

13.1 N/A

14) Data Acquisition

14.1 If the computer-generated worksheets are used, they must be initialed and dated.

14.2 Each batch must contain the analyst, sample identity, concentration and comments.

14.3 A peer analyst must review all data prior to entering into LIMS. The reviewer must initial and date each batch following review.

15) Calculation, and Data Reduction Requirements

15.1 Total Suspended Solids (mg/L) =

$$\frac{\text{suspended solids (g) x 1,000,000}}{\text{volume used (ml)}}$$

15.2 QC Calculations: LIMS calculates the percent recovery for various QC samples (MS, MSD, LCS) according to the following equations:

15.2.1 % Recovery, %R (for MS and MSD Samples)

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SR = Sample Result (unspiked).
SA = Spike Amount Added (mg/L or mg/kg).

15.2.2 % Recovery, %R (for standards and LCS)

$$\%R = \frac{(SSR)}{SA} \times 100$$

Where:
SSR = Spiked Sample Result (mg/L or mg/kg).
SA = Spike Amount Added (mg/L or mg/kg).

15.2.3 RPD (for precision or duplicate evaluation)

$$\text{RPD} = \frac{|\text{SR}_1 - \text{SR}_2|}{½(\text{SR}_1 + \text{SR}_2)} \times 100$$

Where:
SR_1 = Sample result for duplicate 1.
SR_2 = Sample result for duplicate 2.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Method Blank

16.1.1 A method blank must be processed with each analytical batch or every 20 samples, whichever occurs first.
16.1.2 The method blank must be:

16.1.2.1 < ½ the PQL, or
16.1.2.2 < 5% the sample concentration, or
16.1.2.3 < 5% of the regulatory limit.

16.1.3 All samples associated with a failed method blank must be re-processed. If the samples cannot be re-processed, all associated analytical results must be flagged and narrated as to possible bias.
16.1.4 Preparation:

16.1.4.1 For TSS: filter 1000 mL of DI water.

16.2 Laboratory Control Sample (LCS)

16.2.1 A LCS must be processed with each analytical batch or every 20 samples, whichever occurs first.
16.2.2 Recovery of the LCS must meet accuracy performance criteria as outlined in the applicable LIMS test code.
16.2.3 All samples associated with a failed LCS must be re-processed. If samples cannot be re-processed, all associated samples must be flagged and narrated as to possible bias.
16.2.4 Preparation:

16.2.4.1 For TSS: filter 100 ml of the 100 mg/L TSS standard (10.2).

16.3 Sample Duplicate

16.3.1 Analyze a sample duplicate for every 10 field samples analyzed.
16.3.2 Must meet precision performance criteria as outlined in the applicable LIMS test code.
16.3.3 If the sample result is <5x the reporting limit, the sample is narrated. If the samples cannot be reprocessed due to insufficient sample, all associated samples must be qualified and narrated as to possible bias.

16.4 Deviations and non-conforming events must be documented using a Nonconformance Corrective Action Report (NCAR) or as an Exception Report item on the laboratory

review checklist. For mandatory QC failures (e.g. LCS), the NCAR must be submitted to the QA Manager via the NCAR database.

17) Data Records Management

17.1 All data is stored both electronically and hard copy for 10 years.

17.2 All analytical sequence IDs and standard preparation information must be recorded in the appropriate logbook. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard tracking, analyst must attempt to use one lot of reagents and standards with each batch.

17.3 The effective date of this SOP is the date in the header or last signature date, whichever is most recent.

18) Contingencies for Handling Out of Control Data

18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.

18.2 When affected sample analysis can not be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).

18.3 All analysts must report sufficient comments for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.

18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Initial Demonstration of Proficiency- Each analyst must perform an initial demonstration of proficiency on a method and matrix basis with a successful analysis of four LCS where acceptable precision and accuracy are generated. The accuracy component must fall within LCS criteria. The precision component must be less than 20% for duplicate RPD data.

19.2 Method Detection Limits (MDLs) are evaluated on an annual basis based entirely from Method Blank data.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R00	9/15/15	CES	New SOP
R01	9/15/17	LC	Revised section 12.2.7 – updated weighing procedure
R02	9/25/19	LC	Updated to correct method references Added maximum filter time
R03	7/31/21	PAL	Revised sec. -12.2.5, 14.1,16.3.3, 18.3, 19.2

21) References and Related Documents

21.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, EPA/600/4-79-020, Methods 160.2.

21.2 40 CFR, Chapter 1, Section 63.7325.

21.3 Standard Methods for the Examination of Water and Wastewater, Online Edition, Method 2540D, 2011.

21.4 ALS Environmental Quality Assurance Manual, Version (most current).

Importance of Microcystins/Nodularins Determination

Most of the world's population relies on surface freshwaters as its primary source for drinking water. The drinking water industry is constantly challenged with surface water contaminants that must be removed to protect human health. Toxic cyanobacterial blooms are an emerging issue worldwide due to increased source water nutrient pollution caused by eutrophication. Microcystins and Nodularins are cyclic toxin peptides. Microcystins (of which there are many structural variants, or congeners) have been found in fresh water throughout the world. To date, approximately 80 variants of Microcystin have been isolated. The most common variant is Microcystin-LR. Other common Microcystin variants include YR, RR, and LW. These toxins are produced by many types of cyanobacteria (blue-green algae), including *Microcystis, Anabaena, Oscillatoria, Nostoc, Anabaenopsis,* and terrestrial *Hapalosiphon.* Nodularins are produced by the genus Nodularia and they are found in marine and brackish water

Acute poisoning of humans and animals constitutes the most obvious problem from toxic cyanobacterial blooms and, in several cases, has led to death. Human and animal exposure to these toxins occurs most frequently through the ingestion of water, through drinking or during recreational activities in which water is swallowed. These toxins mediate their toxicity by inhibiting liver function and are potent inhibitors of the serine/threonine protein phosphatases, and therefore they may act as tumor promoters.

To protect consumers from adverse health effects caused by these toxins, the World Health Organization (WHO) has proposed a provisional upper limit for Microcystin-LR of 1.0 ppb (µg/L) in drinking water. For recreational bathing waters, the WHO has established the following guidelines:

-Relatively low risk of exposure effect at 4 ng/mL (ppb)
-Moderate probability of exposure effect at 20 ng/mL
-High probability of exposure effect – scums

The U.S. Environmental Protection Agency (EPA) has also established guidelines for Microcystins in drinking water:

-For children below school age, 0.3 µg/L (ppb)
-For all other age groups, 1.6 µg/L (ppb)

Performance Data

Test sensitivity: The Eurofins Abraxis Microcystins Strip Test for Recreational Water will detect Microcystins and Nodularins at 1 ng/mL or higher. At this level, the test line exhibits moderate intensity. At levels greater than 10 ng/mL the test line is not visible. When compared with samples of known Microcystins concentration, it is possible to obtain a semi-quantitative result.

Selectivity: The assay exhibits very good cross-reactivity with all Microcystin cyclic peptide toxin congeners tested to date.

Cell Lysing: When comparing samples lysed using the QuikLyse™ reagents and the 3 cycle freeze/ thaw method, average recovery obtained was 94%, SD = 16.7%.

Samples: A sample correlation between the Eurofins Abraxis Strip Test and ELISA methods showed a good correlation.

General Limited Warranty/Disclaimer: Eurofins Abraxis warrants the products manufactured by the Company, against defects and workmanship when used in accordance with the applicable instructions for a period not to extend beyond the product's printed expiration date**. Eurofins Abraxis makes no other warranty, expressed or implied. There is no warranty of merchantability or fitness for a particular purpose.** The ETV verifies the performance of commercial ready technologies under specific criteria, testing conditions, and quality assurance. ETV does not imply approval or certification of this product, nor does it make any explicit or implied warranties or guarantees as to product performance. www.epa.gov/etv.

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R080919

Microcystins Strip Test

eurofins | Abraxis

Immunochromatographic Strip Test for the Detection
of Microcystins and Nodularins in Recreational Water at 10 ppb

QuikLyse™ reagents may be used in a method of U.S. Patent 9,739,777

Product No. 520023 (5 Test), 520022 (20 Test)

1. General Description

The Eurofins Abraxis Microcystins Strip Test for Recreational Water is a rapid immunochromatographic test, designed solely for the use in the qualitative screening of Microcystins and Nodularins in recreational water (freshwater samples only; please see the Brackish or Sea Water Sample Preparation technical bulletin for information on the screening of marine water samples). A rapid cell lysis step (QuikLyse™) performed prior to testing is required to measure total Microcystins (dissolved, or free, plus cell-bound). The Eurofins Abraxis Microcystins Strip Test provides only preliminary qualitative test results. If necessary, positive samples can be confirmed by ELISA, HPLC or other conventional methods.

2. Safety Instructions

Discard samples according to local, state and federal regulations.

3. Storage and Stability

The Microcystins Strip Kit should be stored between 4–30°C. The test strips, test vials and water samples to be analyzed should be at room temperature before use.

4. Test Principle

The test is based on the recognition of Microcystins, Nodularins, and their congeners by specific antibodies. The toxin conjugate competes for antibody binding sites with Microcystins/Nodularins that may be present in the water sample. The test device consists of a vial containing specific antibodies for Microcystins and Nodularins labeled with a gold colloid and a membrane strip to which a conjugate of the toxin is attached. A control line, produced by a different antibody/antigen reaction, is also present on the membrane strip. The control line is not influenced by the presence or absence of Microcystins in the water sample and, therefore, should be present in all reactions.

In the absence of toxin in the water sample, the colloidal gold labeled antibody complex moves with the water sample by capillary action to contact the immobilized Microcystins conjugate. An antibody-antigen reaction occurs forming a visible line in the 'test' area. The formation of two visible lines of similar intensity indicates a negative test result, meaning the test did not detect the toxin at or above the cut-off point established for the toxin. If Microcystins are present in the water sample, they compete with the immobilized toxin conjugate in the test area for the antibody binding sites on the colloidal gold labeled complex. If a sufficient amount of toxin is present, it will fill all of the available binding sites, thus preventing attachment of the labeled antibody to the toxin conjugate, therefore preventing the development of a colored line. If a colored line is not visible in the test line region, or if the test line is lighter than the control line, Microcystins are present at a level > 2.5 ppb. Semi-quantitative results in the range of 0-10 ppb can be obtained by comparing the sample test strip appearance to the appearance of test strips from solutions of known Microcystins concentrations (control solutions). Microcystins controls are available through Eurofins Abraxis (PN 422011).

5. Limitations of the Microcystins Strip Test, Possible Test Interference

Numerous organic and inorganic compounds commonly found in water samples have been tested and found not to interfere with this test. However, due to the high variability of compounds that might be found in water samples, test interferences caused by matrix effects can't be completely excluded.

Mistakes in handling the test can also cause errors. Possible sources for such errors include:
Inadequate storage conditions of the test strip, too long or too short incubation times, extreme temperatures during the test performance (lower than 10°C or higher than 30°C).

The test is designed for use with freshwater recreational waters. The use of the test with brackish or seawater samples will produce inaccurate results. Please see the Brackish or Sea Water Sample Preparation technical bulletin for information on the preparation and screening of marine water samples using the Microcystins Strip Test for Finished Drinking Water. The Microcystins Strip Test provides only a preliminary qualitative test result. Use another more quantitative analytical method such as ELISA or instrumental analysis to obtain a confirmed quantitative analytical result. Apply good judgement to any test result, particularly when preliminary positive results are observed.

6. Warnings and Precautions

-The Microcystins Strip Test for Recreational Water is for the screening of freshwater recreational water samples for total Microcystins (free and cell-bound). Please see the Brackish or Sea Water Sample Preparation technical bulletin for the preparation and screening of marine water samples using the Microcystins Strip Test for Finished Drinking Water.

-Use of the Microcystins Test Strips **without** the QuikLyse™ reagents will adversely affect the performance of the test, producing inaccurate results. To test samples without using QuikLyse™ reagents for cell lysis, such as when testing for free Microcystins only or when testing samples which have been previously lysed (such as those which have undergone the freeze/thaw method), please use the Eurofins Abraxis Microcystins Strip Test for Finished Drinking Water at 1 ppb, PN 520016 (5 Test) or PN 520017 (20 Test).

-Use only the Microcystins test strips and QuikLyse™ reagents from one kit lot, as they have been adjusted in combination.

-All reagents and samples should be allowed to reach room temperature before testing.

-Prior to use, ensure that the product has not expired by verifying that the date of use is prior to the expiration date on the label.

-For test strips packaged in a desiccated vial, the vial should be kept completely closed except for opening to remove test strips. When re-closing, snap lid firmly.

-Avoid cross-contamination of water samples by using a new sample vial and disposable pipette for each sample.

-Samples containing unusually large amounts of algal blooms or very thick algal scums should be diluted 1:1 with deionized or distilled water prior to lysis, as overly viscous samples may not allow for uniform cell lysis or proper capillary flow up the test strip. Diluted samples will have a cut-off of 20 ppb.

-Use reasonable judgment when interpreting the test results.

-Results should be interpreted within 5-10 minutes after completion of the test.

7. Sample Collection and Handling

-Collect water samples in glass or polyethylene terephthalate (PETG) containers only. The use of other types of plastic containers may result in adsorptive loss of Microcystins, producing inaccurate (falsely low) results.

-Samples can be stored refrigerated for up to 5 days. If samples must be held for greater than 5 days, samples should be stored frozen.

A. Materials Provided

1. Microcystins test strips in a desiccated container
2. Sample collection vials
3. Lysis vials
4. Graduated disposable pipettes (calibrated at 1 mL)
5. Forceps
6. Reagent papers
7. Conical test vials
8. Disposable transfer pipettes
9. User's guide

B. Additional Materials (not provided with the test)

1. Timer
2. Microcystins Check Samples, Eurofins Abraxis PN 422011, for the preparation of control solutions which can be analyzed with samples, to obtain semi-quantitative sample results (see Section C, Controls, below).

C. Controls

It is a good laboratory practice to use positive and negative controls to ensure proper test performance. Water samples containing known quantities of Microcystins (positive and negative controls) should be analyzed with each lot of test strips to provide a reference for line intensity to be expected.

D. Test Preparation

1. Allow the reagents and water sample to reach room temperature before use.
2. Remove the number of test strips required from the package. The remaining strips are stored in the tightly closed desiccated container.

E. Procedure

When analyzing for total Microcystins (dissolved, or free, and cell-bound), which may be present in recreational waters, a sample lysis is necessary before analysis. The Eurofins Abraxis QuikLyse™ reagents provide a rapid option for cell lysis.

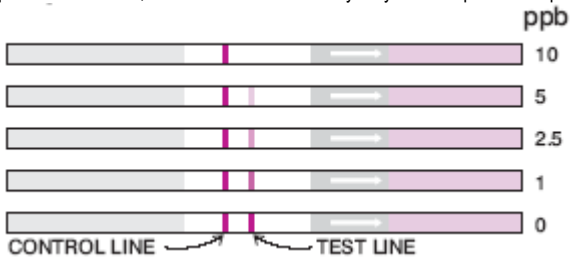
1. Using a new graduated disposable pipette for each sample, draw the sample to the 1 mL line (graduation mark slightly below bulb) and add 1 mL of sample to the lysis vial.
2. Cap the vial and shake for 2 minutes, then allow the sample in the vial to incubate at room temperature for 8 minutes, to begin the cell lysis.
3. Using the forceps provided, add 1 reagent paper to the lysis vial.
4. Cap the vial and shake for 2 minutes, then allow the sample in the vial to incubate at room temperature for 8 minutes.
5. Label conical test vials for each sample to be tested.
6. Using a new disposable transfer pipette for each sample, transfer 7 drops (approximately 200 µL) of the previously lysed water sample (Steps 1-4 above) to the appropriately labeled conical test vial.
7. Close the conical test vial and shake for 30 seconds. Examine the vial to ensure all dried reagents are completely dissolved (dried reagents will dissolve, turning the sample purple).
8. Insert test strip (arrows down) into the conical vial.
9. Allow the test to develop for 10 minutes.
10. Remove the test strip. Lay the strip flat and allow to continue developing for 5 minutes.
11. Read the results visually, as explained below in Section F, Interpretation of Results.

F. Interpretation of Results

Sample concentrations are determined by comparison of the intensity of the test line to the intensity of the control line on the same test strip. Although control line intensity may vary, a visible control line must be present for results to be considered valid. Test strips with a test line which is darker than or of equal intensity to the control line indicates a result which is below the limit of detection of the test. Test strips with a test line which is lighter than the control line indicates a result which is <10 ppb. Test strips with no test line visible (only the control line is visible) indicates a result which is ≥ 10 ppb. Results should be determined within 5-10 minutes after completion of the strip test procedure. Determination made using strips which have dried for more or less than the required time may be inaccurate, as line intensities may vary with drying time.

Control Line	***Test Line***	***Interpretation***
No control line present	No test line present	Invalid result
Control line present	No test line present	>10 ng/mL (ppb)
Control line present	Moderate to equal intensity test line present	Between 0 and 10 ng/mL (ppb)

The appearance of test strips may also be compared to the illustration below to determine approximate sample concentration ranges. Please note that the illustration is intended for the demonstration of test line to control line intensity only. Results should not be determined by comparing the intensity of test lines from test strips to the test line intensity of the illustration, as the overall intensity of test strips may vary slightly with different lots of reagents. To obtain semi-quantitative results in the range of 0-10 ppb, solutions of known Microcystins concentration (control solutions) must be tested concurrently with samples. Sample test line intensities can then be compared with control solution test line intensities, yielding approximate sample concentrations. Do not use strips run previously to determine semi-quantitative sample concentrations, as test line intensities may vary once strips are completely dry.



Alternately, test strips can also be interpreted using the AbraScan test strip reader (PN 475025), which provides objective determination of line intensities for consistent interpretation of results as well as a digital photographic record of all test strips.

G. Additional Analysis

If necessary, positive samples can be confirmed by ELISA, HPLC or other conventional methods. These services are available from commercial analytical laboratories such as Green Water Labs (www.greenwaterlab.com).

H. References

(1) W. J. Fischer, I. Garthwaite, C.O. Miles, K.M. Ross, J.B. Aggen, A.R. Chamberlain, N.A. Towers, and D.R. Dietrich, Congener-Independent Immunoassay for Microcystins and Nodularins. Environ. Sci. Technol. 35, 2002, 4849-4858.
(2) Worldwide Patenting PCT WO 01/18059 A2.
(3) U.S. Patent Number 6,967,240.
(4) U.S. Patent Number 9,739,777.

Standard Operating Procedure
for the Analysis of
Total and Fecal Coliform Enumeration Quanti-Tray® Method
SOP No. USC215

Utility Services Corporation

Laboratory Division

Standard Operating Procedure

For Total and Fecal Coliform Enumeration

Using the Quanti-Tray® Method
Certified Laboratory ID Number:
M-64-05

SOP No. USC215

Approved by ISDH

Revised 7/18/18

Utility Services Corporation

Laboratory Division

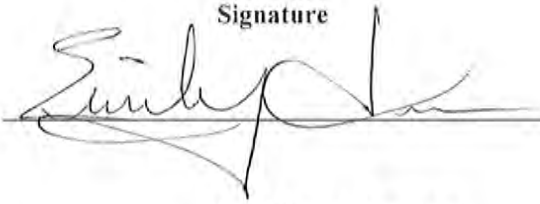
Standard Operating Procedure

Signature Sheet

I acknowledge that I have read the contents of this manual.
I shall follow the policies and procedures set forth relevant to my testing activities and capabilities for the analysis of

SM9223B, Enzyme Substrate Multi-well, Colilert Quanti-Tray Method

- Quanti-Tray sealer type updated

Signature	Date
	7-18-18
QC Officer Signature	**Date**
	7/18/18

Standard Operating Procedure
for the Analysis of
Total and Fecal Coliform Enumeration Quanti-Tray® Method
SOP No. USC215

Scope and Application

The coliform group of bacteria, as defined by Standard Methods, is a principal indicator of the suitability of drinking water, ambient waters, and wastewater. Specifically, the degree of pollution is interpreted by the fecal coliform microorganism, *Escherichia coli*, presence and density as potential health hazards. This method describes an enumeration test for compliance monitoring of total coliforms and *Escherichia coli* coliforms using a multi-well procedure. Colilert® is a chromogenic-fluorogenic substrate test used to detect certain enzyme activity processed by the total coliform group that possesses the enzyme β-D-galactosidase, which cleaves the chromogneic substrate and turns the sample yellow. The Colilert® substrate also identifies fecal coliforms that possess β-glucuronidase, which cleaves the fluorogenic substrate that will fluoresce under UV light.

Summary of Method

The Quanti-Tray® method is based on IDEXX's patented Multiple Enzyme Technology ™, which detects viable bacteria by testing for the presence of key enzymes known to be present in these organisms. Samples are collected in sterile, 100ml vials. The Colilert® media is aseptically added to the vial and shaken. The sample is then poured into a Quanti-Tray (1-200 MPN tray or 1-2419 MPN tray), put through the Quanti-Tray Sealer, and incubated at 35°C +/- 0.5°C for 24 hours. The number of yellow wells that are darker than the comparator are counted, compared to the MPN chart, and reported as Total Coliforms MPN/100mls. The wells that fluoresce under UV are counted, compared to the MPN chart and reported as *E. coli* MPN/100mls.

Apparatus

IDEXX 100ml sample bottles, with Dechlorinating agent, WV120BST-200
IDEXX Quanti-Trays, 1-200CFU, WQT100
IDEXX Quanti-Trays, 1-2419CFU, WCMB-100-2K
IDEXX Quanti-Tray Sealer Plus
Quanti-Tray rubber inserts, 51 wells & 97 wells
UV Hand Lamp and bulbs, long wavelength 365nm, NCL Cat. No. UV-56
Idexx MPN Table
Incubator, Precision 4E11
Autoclave, Pelton & Crane, Validator Plus
Pipets, reusable borosilicate glass
100ml Class A cylinders, autoclavable glass

Reagents

IDEXX Colilert® reagent, WP200I. Store the media at 2-30°C away from light.
IDEXX Comparator, 1-200CFU tray, WQTC
IDEXX Comparator, 1-2419CFU tray, WQT2KC
IDEXX Quanti-Cult® kit, WKIT1001. Store the Quanti-Cult® at 2-30°C, away from light.

QC IDEXX Quanti-Cult cultures are used to test each new lot of media, methodology as described by the manufacturer. Three different control cultures are analyzed, Table 1 below, illustrates the expected results. Sterile distilled water in sterile sample bottles is used for control cultures and then warmed in the incubator prior to inoculation.

QC New media lots are spot checked under UV for autofluorescence, results are logged in the 'Media & Supplies' book under "QC lot checks".

QC Quanti-Tray lots are checked for sterility, 100mls of sterile DI and add reagent. Results are logged in the 'Media and Supplies' book under "QC lot checks".

Packing slips from media shipments are kept on file by the log books in the Drinking Water lab area.

Table 1 QC Culture Results

	E. coli	*Kleb. Pneumoniae*	*PSA*
Expected Results	ONPG + / MUG +	ONPG + / MUG -	ONPG - / MUG -

Standard Operating Procedure
for the Analysis of
Total and Fecal Coliform Enumeration Quanti-Tray® Method
SOP No. USC215

Sampling and Storage

The laboratories' sample collector must be trained in aseptic sampling procedures according to SM 9060A. Refer to the Quality Assurance Manual for detailed discussion on Quality Control for equipment, lot checks, sample forms, and log in procedures. Samples not collected by company personnel must be collected in bottles provided by our laboratory or are otherwise noted on the COC. A disclaimer about the sample container in the analysis report narrative should discuss this. After collection, the sampler must fill out the USC Sample Information Form (SIF), Chain of Custody, or State Form 53297.

QC The sample bottles are purchased pre-sealed with Sodium Thiosulfate in them, and are lot checked for sterility, auto-fluorescence, and volume. Quality Control results are recorded in log books kept in the laboratory.

QC According to SM9060B, hold source water, stream pollution, recreational water, and wastewater samples at <8.0 °C from time of collection to analysis. Samples should not exceed transit time of 6 hours. Samples over 6 hours are rejected and a new sample requested. Samples that are rejected are indicated on Sample Information Form or the Chain of Custody form and why.

Procedure

Refer to the Quality Assurance Manual for sample log in procedures, equipment calibration and maintenance QC, and temperature monitoring procedures. Samples are assigned a laboratory number or facility name and date. The sample ID, date and time of analysis, are written on back of the Quanti-tray.

Turn on Sealer and wait for the ready light to turn green. Sample should measure 100mls, mix sample by inverting several times and then decant carefully to the 100ml mark on the sample bottle. For unmarked sample containers, use a Class A sterilized cylinder, mix sample by inverting several times, pour off sample in cylinder. Waste any remaining sample in bottle, then pour sample from the cylinder to the bottle.

Aseptically add one snap pack of media to the vessel and shake the sample to dissolve media. Allow the foam to settle from the sample. Take the cap off the vessel. Using one hand, hold a tray upright, well side facing the palm. Fold the sides of the tray away from palm and gently pull the foil tab to separate the foil from the wells. Aseptically pour the sample into the tray. Allow the foil to close on the tray and tap the bottom of the tray (or small wells) until all trapped air bubbles are released. Place the tray in the appropriate rubber insert. Put the rubber insert and tray, well side facing down, on the sealer loading shelf. Gently push rubber insert into sealer until the mechanism catches it and pulls the tray through. Tray will come out of the sealer and be ready for incubation. Lay the tray well side down, in stacks up to 10 trays, in a 35°C +/- 0.5°C incubator for 24 hours.

QC A single, *positive,* sample tray must be counted by both analysts once a month. The counts must agree within %10 and are logged in the 'HPC Log Book' under Quanti-Tray Analyst Duplicates.

Interpretation of Results

Count the number of wells that are more yellow than the comparator for Total Coliforms. Count the large wells and small wells separately on the 1-2419CFU trays. Refer to the MPN chart provided by IDEXX that corresponds to the tray. Report results as MPN/100mls Total Coliform.

To numerate *Escherichia coli*, hold the UV light 2-3" above the tray in a dim lit room, if the well fluoresces, then the results are interpreted as 'Present' for *E. coli*. Count all fluorescing wells, large and small wells separately. Refer to the appropriate MPN chart to interpret the results. Report the results as MPN/100mls *E. coli*.

Always use the comparator when reading results. Read the results according to the Result Interpretation Table 1, below:

Table 1 Result Interpretation

Well Appearance	Results: MPN	
Less yellow than the comparator	Negative –Total coliforms Negative –*E. coli*	 ONPG - / MUG -
Yellow equal to or greater than the comparator	Positive –Total coliforms Negative –*E. coli*	 ONPG + / MUG -
Yellow equal to or greater than the comparator and fluorescence	Positive –Total coliforms Positive –*E. coli*	 ONPG + / MUG +

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Results are definitive at 24-28 hours. Positive results *before* 24 hours are valid. Samples are negative if at any time after 24 hours there is no color change greater than the comparator. Only negative results are valid if the incubation exceeds 28 hours.
Data sheets are kept on file in the lab office.

Action Response to Laboratory Results
The Utility Services SIF or State Form 53297 is filled out according to the results and a copy is returned to the water source authority, private party, or operator within 4 days. A scanned copy of the form can be emailed or faxed to the county Health Department that the sample was collected in, on behalf of the client. Samples with a State ID Number will be faxed to IDEM Water Quality Department. Results that do not comply with state regulations according to 410 IAC 6-7.1-27 are reported to the client by the end of the business day, or within 24 hours. Wastewater permit holders that fail compliance monitoring are also notified as soon as possible.

Procedural Quality Control
Each new lot of reagent, bottles, and supplies that come into the lab are recorded in the 'Media & Supplies' log book kept in laboratory and given a laboratory number.
If a sample has some background color, compare inoculated Colilert sample to a control blank of the same water sample. If the background color is darker than the comparator, the sample is rejected.
If a sample has excessive chlorine in it, a blue flash may been seen when adding Colilert. If this is seen, consider the sample invalid and request a new sample.
Do not pre-filter sample and culture the filter in Colilert media.
Do not use buffered water for dilution, only use sterile water.
Avoid prolonged exposure to light.

Glassware
A complete discussion of glassware quality control is detailed in the Quality Assurance Manual.

QC Pipets and cylinders are washed using laboratory grade detergent, Liquinox. The Inhibitory Residue Test is kept on file in the laboratory office, per new lot of Liquinox.

QC One random pipet is spot checked for pH reaction using Bromthymol Blue, per batch of washed pipets. The results must be a 'blue-green' color or the pipets are washed again until it does.

QC The pipets are put in the stainless steel container and sterilized according to the QA Manual procedures. Sterilization records are maintained according to the QA Manual. The sterilized container is then given an expiration date of three weeks from the date it was autoclaved and labeled with tape on the container.

Disposal
Cultured media is destroyed by autoclaving. Place the trays inside an autoclavable bag and seal, do not over crown the bags. Double bag if necessary. The bags are put on a stainless-steel tray lined with foil and autoclaved at 121°C, 15 psi, for 30 minutes.

References
Standard Methods for the Examination of Water and Wastewater, 22nd Edition 2012; Method 9060A&B Sample Collection and Storage, 9213D Natural Bathing Beaches, Method 9223A&B, b. Multi-well procedure.

Indiana Certification Standards For Drinking Water Microbiology Laboratories, July 2006, Indiana State Department of Health, Environmental Laboratory Division, Indianapolis, Indiana 46202.

Quality Control Policies and Procedure Manual, Indiana State Department of Health, Environmental Microbiology Laboratory, REV2006, Indianapolis, Indiana 46202.

IDEXX Quanti-Tray/2000 method & IDEXX Quanti-Tray Sealer manual 2X, IDEXX Laboratories, Inc.

Colilert® Procedure Document, MMO-MUG Test, Indiana State Department of Health, Environmental Microbiology Laboratory, REV2006, Indianapolis, Indiana 46202.

Colilert® Procedure Manual, 2007 IDEXX Laboratories, Inc.

APPENDIX 6
EXAMPLE CHAIN OF CUSTODY FORM

Chain of Custody Form

Page _____ of _____

☑ ALS Environmental
2400 Cumberland Ave
Valparaiso, IN 46383
(Tel) 219.299.8127
(Fax) 616.399.6185

ALS Project Manager:		ALS Work Order #:	

Customer Information		Project Information		Parameter/Method Request for Analysis	
Purchase Order		Project Name		A	
Work Order		Project Number		B	
Company Name		Bill To Company		C	
Send Report To		Invoice Attn.		D	
Address		Address		E	
				F	
City/State/Zip		City/State/Zip		G	
Phone		Phone		H	
Fax		Fax		I	
e-Mail Address				J	

No.	Sample Description	Date	Time	Matrix	Pres. Key Numbers	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold

Sampler(s): Please Print & Sign

Shipment Method:

Turnaround Time: (Business Days)
☐ 10 BD ☐ 5 BD ☐ 3 BD ☐ 2 BD ☐ 1 BD ☐ Other _____

Results Due Date:

Relinquished by:	Date:	Time:	Received by:	Date:	Time:	Notes:
Relinquished by:	Date:	Time:	Received by (Laboratory):	Date:	Time:	
Logged by (Laboratory):	Date:	Time:	Checked by (Laboratory):			

Cooler Temp °C	pH Verified	QC Package: (Check Box Below)	
		☐ Level II: Standard QC	☐ Level III: Raw Data
		☐ TRRP LRC	☐ TRRP Level IV
		☐ Level IV: SW846 Methods/CLP like	
		☐ Other: _____	

Preservative Key: **1**-HCl **2**-HNO_3 **3**-H_2SO_4 **4**-NaOH **5**-$Na_2S_2O_3$ **6**-$NaHSO_4$ **7**-Other **8**-4°C **9**-5035A

Note: Any changes must be made in writing once samples and COC Form have been submitted to ALS.

APPENDIX 7
QUALITY ASSURANCE INFORMATION REQUIRED FOR DQA LEVEL 2 AND DQA LEVEL 3 DATA SUBMISSION FOR OWQ USES

Quality Assurance Information Required for DQA Level 2 and DQA Level 3 Data Submissions for OWQ Uses					
Item	**Included with Data Submittal**	**Available to OWQ upon Request**	**Not Available**	**N/A**	**Comments**
	DQA Level 3	**DQA Level 2**	**DQA Level 1**		
Sample Information					
Sampling and Analysis Work Plan or Quality Assurance Project Plan was submitted as part of the Data Package.					
General Sample Information and Field Parameters					
Dates of sample collection were recorded.					
Times of sample collection were recorded.					
Physical locations of sample collection were recorded.					
Analytical methods used with this data set were recorded.					
Approved detection limits were recorded.					
Field calibration checks were recorded.					
Field duplicates were collected as appropriate.					
Data Package included detailed listing of the preservatives used in the samples, per each individual container.					
General Chemistry and Nutrients Data					
Sample Prep Dates were recorded.					
Date of analysis was recorded for each result.					
Analytical method was recorded for each result.					
Detection limits were recorded for each parameter.					
Quantitation (Reporting) Limits were recorded.					
Blank, Field Duplicate and MS/MSD results were recorded.					
Instrument calibrations were recorded.					
Laboratory control standards results were recorded.					
Initial and continuing calibration results were recorded.					
Metals Data					
ICP Serial Dilution information was recorded.					
ICP Linear Range Studies information was recorded.					
ICP Interelement Correction Study information was recorded.					
ICP Interference Check Standard information was recorded.					
ICP CRQL Standard information was recorded.					
ICP/MS Mode used in the analysis was recorded.					
ICP/MS Stability Check with Tuning Solution information was recorded.					

Quality Assurance Information Required for DQA Level 2 and DQA Level 3 Data Submissions for OWQ Uses					
Item	**Included with Data Submittal**	**Available to OWQ upon Request**	**Not Available**	**N/A**	**Comments**
	DQA Level 3	**DQA Level 2**	**DQA Level 1**		
Organics Data					
Surrogates information was recorded.					
Internal Standards information was recorded.					
System Performance information was recorded.					
Bacteriological Data					
Summary Data Package was compiled.					
Sample Prep Dates and Times were recorded.					
Sample Analysis Dates and Times were recorded.					
Holding Times were recorded.					
Incubation Parameters were recorded.					
Temperature Evaluation was conducted.					
Analytical Methods were recorded.					
Detection Limits were recorded.					
Quantitation (Reporting) Limits were recorded.					
Blank, Field Duplicate and MS/MSD results were recorded.					
Field and Method Duplicates were collected.					
Colilert Quality Control Report(s) were collated.					
Positive Control results were recorded.					
Beginning and Ending Sterility Control results were recorded.					
KP, PA, EC, Media Control Standards results were recorded.					
Chain of Custody					
Chain of Custody form was used.					
Chain of Custody Form included the signature of the person who collected the samples.					
Chain of Custody Form included the signature of the person accepting custody of the samples.					
Chain of Custody Form included the date that the samples were collected.					
Chain of Custody Form included the time that the samples were collected.					
Chain of Custody Form included the date that the samples were received by the Testing Laboratory.					
Chain of Custody Form included the time that the samples were received by the Testing Laboratory.					
Chain of Custody Form included the type and number of containers that were used for each sample.					

Quality Assurance Information Required for DQA Level 2 and DQA Level 3 Data Submissions for OWQ Uses					
Item	Included with Data Submittal	Available to OWQ upon Request	Not Available	N/A	Comments
	DQA Level 3	DQA Level 2	DQA Level 1		
Testing Laboratory					
Name and address of the Testing Laboratory was recorded.					
Telephone number and e-mail of the Contact Person at the Testing Laboratory was recorded.					
Sample delivery date and time was recorded by the laboratory.					
Testing Laboratory Job Number was recorded.					
Date that the Lab Report was prepared was recorded.					
Date that the Lab Report was received from the laboratory was recorded.					

APPENDIX 8
DATA REVIEW AND REPORTING STANDARD OPERATING PROCEDURES

DATA REDUCTION, REVIEW, AND VALIDATION

SOPID:	VAL-QS-009	Rev. Number:	R01	Effective Date:	10/15/2021

Approved By: _____ Date: 10/7/21
QA Manager

Approved By: _____ Date: 10/7/2021
Laboratory Director

Archival Date:	_____	Doc Control ID#:	_____	Editor:	_____

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

DATA REDUCTION, REVIEW, AND VALIDATION

1) Scope and Applicability

1.1 This SOP provides the procedures for data reduction, review, and validation of analytical results produced by ALS Environmental. These procedures are performed either manually or through the use of computer programs associated with the analytical instruments and/or the LIMS system. Checklists are employed to ensure processes are carried out to meet project and/or method QC requirements and to ensure the validity of the data for its intended use.

1.2 The data validation process consists of the following reviews:

1.2.1 Preliminary review of information regarding samples (e.g., sample collection, preservation, holding time requirements, condition upon arrival).
1.2.2 Evaluation of QC analysis data (e.g., calibrations, blanks, spikes, replicates, etc.) against method and/or project specified QC limits
1.2.3 Evaluation of compliance with project requirements.
1.2.4 Comparison of sample results against raw data to identify and correct transcription errors.
1.2.5 A peer review of all analytical data.
1.2.6 A review for reasonableness as part of an independent assessment of the data prior to being reported to the client.

2) Summary of Procedure

2.1 Prior to release of laboratory data, it is taken through reduction, review, and validation steps, beginning with the analyst review (Level I), followed by a peer (Level II) and a final administrative (Level III) review.

2.2 Level I Review: The initial data review is performed by the analyst who generated the data and compares data against a set of prescribed data quality objectives (DQOs) from an SOP or project plan. LIMS and other computer software programs are utilized for data reduction and review. Programmed QC limits are used to compare the data (calibration, client sample, batch QC, etc) generated and entered against method or project QC limits prescribed in analytical SOPs and/or client project DQOs. Data review checklists are used to ensure all steps are performed. The analyst dates and signs the review checklist upon completion and documents any data anomalies and/or non-conformances.

2.3 Level II Review: A peer review is performed by a supervisor or another designated analyst. This review is performed in a manner similar to the Level 1 review using the same approach and checklist. The objective in this review is to confirm the original Level I review and correct any errors or interpretations in the initial review. The peer reviewer also dates and signs the review checklist upon completion.

2.4 Level III Review: This final administrative review, performed by the Project Manager proceeds with review of the entire data package to ensure consistency and compliance with project specific requirements. This review includes checking that all data is included in the report, verifying that all QC and project specific requirements have been met, and screening for logical inconsistencies in the data; a list of common checks is included in Figure 1. Additional comments regarding sample handling and quality control issues may be added at this time within the report case narrative. The Project Manager or designee ensures that all required reviews have been performed

before signing the final report and releasing the data to the client. Level III reviews are documented through release of the final data.

2.5 QA Review: This review will be done in the same manner as the Level III review and is performed by the QA Department. The QA Department randomly selects Work Orders to be reviewed during internal audits. Corrective Action/ Non-conformance Reports are required for any errors noted.

2.6 All information used in the calculations (e.g., raw data, calibration files, tuning and standardization records, results of standard additions, interference check results, and background-correction protocols) must be recorded and archived in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent moisture for solids, actual extraction and dilution volumes, extract volume, dilution factor used) must also be maintained in order to enable reconstruction of the final result at a later date.

3) Definitions

3.1 Data Quality Assessment (DQA): The scientific and statistical evaluation of data to determine if data obtained from environmental operations is of the right type, quality, and quantity to support their intended use. The five steps of the DQA Process include:

3.1.1 Reviewing DQOs and sampling design,
3.1.2 Conducting a preliminary data review,
3.1.3 Selecting the statistical test,
3.1.4 Verifying the assumptions of the statistical test,
3.1.5 Drawing conclusions from the data.

3.2 Data Quality Indicators (DQIs): The quantitative statistics and qualitative descriptors that are used to interpret the degree of acceptability or utility of data to the user. The principal data quality indicators are bias, precision, accuracy, comparability, completeness and representativeness.

3.3 Data Quality Objectives (DQOs): The qualitative and quantitative statements derived from project specifications that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be required of data needed to support decisions.

3.4 Data reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors into a useful form.

3.5 Precision: The agreement among a set of replicate measurements

3.6 Accuracy: The closeness of agreement between an observed value and an accepted reference value.

3.7 Comparability: A measure of the confidence with which one data set or method can be compared to another.

3.8 Completeness: A measure of the amount of valid data obtained from a measurement system relative to the amount expected under correct, normal conditions.

3.9 Representativeness: A measure of the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition.

3.10 Batch: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit

3.11 Matrix Spike: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

3.12 Duplicates: an inter-laboratory split of a sample used to document the precision of a method in a given sample matrix.

3.13 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank must be carried through the entire sample preparation and analytical procedure. The method blank is used to document the presence/absence of contamination resulting from the analytical process.

3.14 Standard Curve: A plot of concentrations of known analytes (standards) versus the instrument response. Calibration standards are purchased or prepared by successively diluting a standard solution to produce working standards that cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate method. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

3.15 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

3.16 Relative Percent Difference (RPD): The percentage representation of the absolute difference between two values relative to the mean of the two values.

4) Health and Safety Warnings

4.1 None

5) Personnel Qualifications and Responsibilities

5.1 General Responsibilities - These procedures are restricted to use by or under the supervision of laboratory personnel experienced in the data reduction, review, and validation of data produced from the various analytical methods performed at ALS Environmental.

5.2 Analyst - It is the responsibility of the analyst(s) to:

5.2.1 Produce contractually compliant data relative to the applicable quality requirements as documented in ALS Environmental SOPs and related client QC criteria.

5.3 Peer Reviewer – It is the responsibility of the peer reviewer to:

5.3.1 Review, confirm, and/or note for correction (as necessary) the findings of the primary analyst as specified in §5.2.1.

5.4 Supervisor - It is the responsibility of the department supervisor to:

5.4.1 Ensure that all department analysts have the technical ability and the necessary training to perform the analytical methods.

5.4.2 Ensure that sufficient redundancy is available to complete peer review.

5.5 Project Manager - It is the responsibility of the Project Manager to ensure that all contractual requirements for client projects are understood prior to initiating a project, and that final reports are reviewed/validated according to these procedures prior to release.

5.6 QA Department – It is the responsibility of the QA Department to have sufficient technical understanding of data generated to perform data reduction, review, and validation of client data generated at ALS Environmental using the client and /or method specified criteria and review checklists associated with this SOP.

6) Procedure

6.1 Level I Data Reduction: The analyst is responsible for reducing data from analytical testing to a format that allows for easy review and reporting of the data. Data is to be summarized onto LIMS generated data summary sheets that contain but are not limited to the following information:

Analysis/Method Number	Lab Sample ID
Client Sample ID	Matrix
Collection Date	Extraction/Digestion Date
Batch ID	Analysis Date
Analyst	Instrument ID
pH, if applicable	% Moisture, if applicable
Dilution Factor(s)	Units
Result(s)	Qualifiers
Method Detection Limit(s)	Batch QC Data
Initial/Continuing Calibration data (ICV, ICB, CCV, CCB)	Raw Data (chromatograms, RF values, etc)
Analyte/CAS Number	

(Batch QC data includes results from the method blank, spike recoveries (LCS, surrogate, and MS/MSD), and relative percent differences (RPD) between replicates (sample and duplicate, and/or MS/MSD.)

6.2 Data reduction is accomplished in one of the following ways:

6.2.1 Manual computation of results (general chemistry areas)

6.2.2 Input of raw data for computer processing (general chemistry and sample preparation areas) into LIMS.

6.2.3 Direct acquisition and processing of raw data by a computer (instrumental methods- ICP/MS, Lachat, etc.)

6.2.4 When analysts make use of manual computation methods, all steps in the computation process shall be shown including the equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. These calculations shall be maintained with the original raw data (e.g., chromatograms). The equations for calculations must be defined in the SOP and be defined in the laboratory logbooks (e.g. titrimetric or gravimetric method calculations).

6.2.5 If the analyst enters raw data for computer processing, a copy of the input shall be kept and uniquely identified with the analytical batch number and other information as needed (laboratory sample IDs and their corresponding raw data for input shall be easily identifiable).

6.2.6 Computer algorithms shall be tested with a set of known data and maintained on file.

6.3 Level I Data Review

6.3.1 The analyst who performed the analysis must review the reduced data. If this individual is unavailable, a designated person with method-specific data interpretation skills must perform the review. This is a 100% review of the raw data. Using a pre-designed (method specific) checklist, the analyst shall systematically review the data (see appendix for an example method checklist).

6.3.2 Blank pre-designed review checklists are available on the server stored under each department's folder. The Level I data review analyst is responsible for signing and dating the review checklist.

6.3.3 The following are examples of what the analyst should be looking for after the analysis has been completed:

- Evaluate any technical problems not previously addressed.
- Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbance, omissions, legibility, etc.).
- Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights, stock and working standards, weighing and dilution data).
- Verify that results fall within the linear range of the analytical procedures.
- Verify that all calibration requirements are met.
- Determine whether QC results meet established criteria. This includes the comparison of QC data against control chart limits.
- Initiate any case narrative discussions relative to that particular analysis, if necessary.
- Document all non-conformance reports upon discovery of errors at this point in the review process and take the appropriate corrective action. The analyst must use experience and judgment to determine the proper corrective actions. Corrective actions for many types of errors are described in the method SOP.

6.4 Level II Data Review - The second stage of data review is an independent review of the batch QC and raw data by the section supervisor. Alternatively, the Section Supervisor may designate a second peer analyst to perform this review. Raw data is to be reviewed to evaluate whether data reductions steps are performed correctly. If errors in data reduction are revealed, all raw data must be re-reviewed and a non-conformance report initiated. This review of the data includes example items such as:

6.4.1 Logbook entries are complete with no spaces left blank.

- If blank fields or incorrect entries are found, the logbook shall be sent back to the primary/initial analyst for correcting prior to continuing level II data review.

6.4.2 Correctness of all calculations (these calculations shall be performed by the checker).

6.4.3 Correct interpretation of spectra, proper integration of chromatograms, strip charts, etc.

6.4.4 Acceptability of QC results, verification that results are within limits generated by current control charts.

6.4.5 Correct qualifiers used.

6.4.6 Spot-checks of computer calculations to verify program validity.

- Any disagreements shall be noted in the comments section of the review checklist. For data processed manually (no computer assistance), the reviewer shall check the data calculations and check final data entry input for possible error.

6.4.7 Review of any comments required for case narrative discussions.

6.4.8 Errors detected in the review process shall be referred to the analyst(s) for corrective action. The original analyst must confirm any errors noted by the reviewer. If the original analyst disagrees with the change, the reviewer and the original analyst must resolve the difference. The reviewer and the analyst must agree on the result that is reported.

6.4.9 The reviewer must sign and date in indelible ink the data checked (except for groups of printouts such as chromatograms).

6.4.10 All exceptions noted in the review must be entered into designated LIMS data comments fields for later inclusion into the project narrative.

6.5 Level III Data Review - The assembled draft report is reviewed by the Project Manager prior to reporting

6.5.1 Level III data review is not intended to review raw data but should assess the usability of the data to assist the client in avoiding inappropriate use of the data. All appropriate information should be used for this review, including the Lab QAPP and the Project QAPP, if available, and communication with the client concerning the intended use and desired quality of the data. If deemed necessary by the PM, data check requests may be requested to verify any anomalies.

6.5.2 This review includes checking that all data is included in the report, verifying that all QC and project specific requirements have been met, and screening for logical inconsistencies in the data; a list of common checks is included in Figure 1.

6.5.3 After completing the Level III review, the Project Manager shall issue a final report to the client. This issuance documents the acceptable Level III review by the PM.

6.6 Review of Subcontractor Data

6.6.1 Contractor data is not subject to Level I and Level II review. These levels of review shall be replaced with a contract compliance screening and shall be performed by the Project Management.

6.6.2 The following are examples of what the reviewer should be looking for in the subcontractor's data package:

- Verify that all samples are accounted for and all the assigned tests have been performed.
- Verify that all contract required calibrations and QC samples are included.
- Verify that adequate explanations are given for data when QC results depart from acceptable criteria.
- Errors detected in the review process shall be referred back to the contract laboratory for corrective action. The contract laboratory must confirm errors noted by the reviewer. If the contract laboratory disagrees with the errors noted, the reviewer and contract laboratory must resolve the difference. The reviewer and the contract laboratory

must agree on all data reported. If necessary, the contract laboratory should submit a revised report to correct any errors noted.

6.6.3 After completing the review, the Project Manager shall issue a final report.

6.7 QAD Review - Client reports are reviewed by the QAD during internal audits.

6.7.1 Any nonconformance discovered during the course of the audit will have an applicable corrective action issued.

6.7.2 Any report revisions recommended as part of a corrective action investigation will be carried out with the cooperation of the Project Manager.

6.7.3 Corrective actions investigated will follow the procedures in VAL-QS-003, *Corrective Action Procedures*.

7) Equipment and Supplies

7.1 Analytical Data Checklists

7.2 LIMS

7.3 Instrument Specific Data Processing software (Target, Atlas, HP Chemstation®, etc.)

8) Quality Assurance and Quality Control

8.1 Refer to Section 6.7 of this SOP

8.2 Refer to VAL-QS-011, Records Archival Procedures.

8.3 Refer to VAL-ADM-003, *Work Order Reporting*

9) Summary of Changes

Table 9.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R00	9/1/15	CES	New SOP
R01	10/15/21	LC	Removed cover page graphics 1.2.6, 2.4 & 6.5.2: revised to include review for reasonableness Added (new) section 6.4.1 for missing entries Figure 1 added – Logical data checks

10) References and Related Documents

10.1 Test Methods for Evaluating Solid Waste. U.S. EPA, SW-846, Third Edition, through Update III, June 1997.

10.2 EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/600/R-98/018, revised February 1998

10.3 ALS Environmental Quality Assurance Manual, Revision (most current)

Figure 1
Logical Data Checks

Test(s)	**Relationship**
Solids (in waters)	Total solids ≥ TSS
	Total solids ≥ TDS
Nutrients	Total phosphorus ≥ ortho-PO_4
	Total phosphorus ≥ organic phosphorus
	TKN ≥ NH_3
	TKN ≥ Total organic nitrogen
	NO_3/NO_2 ≥ NO_3
	NO_3/NO_2 ≥ NO_2
Cyanide	Total cyanide ≥ Free cyanide
	Total cyanide ≥ Amenable cyanide
	Total cyanide ≥ Reactive cyanide
Demands	COD ≥ 1.5 x BOD
	COD ≥ Oil and Grease
	COD ≥ TOC (COD @ approximately TOC x 2.5)
Minerals	Total alkalinity ≥ carbonate alkalinity
	Total alkalinity ≥ bicarbonate alkalinity
	Total alkalinity ≥ hydroxide alkalinity
Others	Total ≥ Dissolved
	Influent ≥ Effluent
	Total ≥ TCLP
	Total sulfide ≥ Reactive sulfide
	Cr ≥ Cr^{+6}
	TDS ÷ Conductivity = 0.55 to 0.81
	TDS ≥ Total alkalinity
	TDS ≥ Hardness
	TDS ≥ Chloride
	TDS ≥ Sulfate
	If TDS = ND, metals should be ND (or very low)
	Measured TDS ÷ Calculated TDS = 1.0 – 1.2 • TDS_{calc} = 0.6(alkalinity) + Na + K + Ca + Mg, + Cl +SO_4 + SiO_3 + NO_3 + F
	COD, BOD, TSS, and Oil and Grease are all high or all low
	If TOC = ND, organic results should be very low level
	If a sample flashes, BTU ≥ 1000
	If a sample flashes, VOC data should show detections for BTEX and/or other flammable compounds
	Cation check[1]: (Ca + Mg + Na) ÷ Conductivity = 0.009 to 0.0124 • If this does not check out, check for other cations at high levels or low pH
	Anion check[1]: (Bicarbonate + carbonate + Cl + SO_4) ÷ Conductivity = 0.009 to 0.0124
	Ion balance check[1]: (Anions – Cations) ÷ (Anions + Cations) < .075
Organics	Gasoline pattern by GC/FID is consistent with BTEX, alkylbenzenes, and naphthalene data
	Diesel pattern by GC/FID is consistent with PAH data
	Chlorinated degradation products (DCE, DCA, DCM, etc.) in the presence of high TCE or PCE results
	PCB Aroclor 1016 results require careful evaluation.
	Mixtures of Aroclor 1242, 1254, and 1260 require careful evaluation and possible flagging of data due to a potential for double counting PCBs
	DDD and DDE in the presence of DDT

[1] Applicable to clean samples with conductivity readings between 200 – 5,000 umhos/cm. Low conductivity, high viscosity, colored samples with high NH_3 content may not work for the cation, anion and ion balance checks.

[1] Applicable to clean samples with conductivity readings between 200 – 5,000 umhos/cm. Low conductivity, high viscosity, colored samples with high NH_3 content may not work for the cation, anion and ion balance checks.

Dept:	ICP-MS	Batch ID:	ICPMS2_150515A
Method:	6020W/200.8W	Analyst:	rh
		Review:	

	Analytical Procedures		***Status***		***ER #***	***NCAR***
	(1) Were all samples prepared & analyzed w/i holding time?		Yes			
	(2) Other than results < PQL, were all raw values bracketed by calibration standards?		Yes			
	(3) Were calculations peer reviewed?		Yes			
	(4) Were analyte identifications peer reviewed?		Yes			
	(5) Were MDL/PQLs reported for all analytes not detected?		Yes			
	Blank Samples					
	(1) Were appropriate types of blanks analyzed?		Yes			
	(2) Were blanks analyzed at the appropriate frequency?		Yes			
	(3) Were method blanks taken through the entire prep/analytical process?		Yes			
	(4) Were blank concentrations < 1/2 PQL?		Yes			
	(5) Were blank concentrations < PQL?		Yes			
	Blank Spike (LCS)					
	(1) Were all reported analytes included in the LCS?		Yes			
	(2) Was the LCS taken through the entire prep/analytical process?		Yes			
	(3) Was the LCS analyzed at the required frequency?		Yes			
	(4) Was the LCS % recovery within the LIMS specified QC limit?		Yes			
	Matrix Spikes (MS/MSD)					
	(1) Were all reported analytes included in the MS/MSD pair?		Yes			
	(2) Was the MS/MSD pair taken through the entire prep/analytical process?		Yes			
	(3) Was the MS/MSD pair analyzed at the required frequency?		Yes			
	(4) Was the MS/MSD accuracy within the LIMS specified QC limit?		No			
	(5) Was the MS/MSD precision within the LIMS specified QC limit?		Yes			
	(6) Were appropriate flags notated in LIMS for MS/MSD excursions?		Yes			
	Duplicates (if required)					
	(1) Were appropriate duplicates analyzed for each matrix?		NA			
	(2) Were duplicates analyzed at the appropriate frequency?		NA			
	(3) Was duplicate precision within the LIMS specified QC limit?		NA			
	(4) Were appropriate flags in LIMS notated for duplicate excursions?		NA			
	Practical/Method Quantitation Limit (PQL/MQL)					
	(1) Are PQL/MQLs for each analyte listed in the data entry batch?		Yes			
	(2) Are the listed PQL/MQLs ≥ than the lowest non-zero calibration standard?		Yes			
	Other					
	(1) Are all known excursions noted by an ER# or NCAR entry?		Yes			
	(2) Were all SOP related corrective actions taken prior to reporting data?		Yes			

	Initial Calibration (ICAL)					
	(1) Was ICAL criteria achieved for reported analytes?		Yes			
	(2) Was the SOP (or method) specifed number of standards utilized for all analytes?		Yes			
	(3) Were all data points between the lowest & highest utilized for curve generation?		Yes			
	(4) Is the initial calibration curve verified with a second-source ICV standard?		Yes			
	(5) Is data traceable to the instrument & the standards utilized?		Yes			
	Calibration Verification (CCV)					
	(1) Were low/high standards (if required) processed at the required frequency?		Yes			
	(2) Were the low/high standard verifications within SOP specified criteria?		Yes			
	(3) Were CCVs processed at the SOP specified frequency?		Yes			
	(4) Was CCV % recovery within SOP specified criteria?		Yes			
	(5) Is data traceable to the instrument & the standards utilized?		Yes			
	(6) Was the absolute value of the analyte concentration in the organic CCB<MDL?		Yes			

	Instrument Tuning					
	(1) Was the instrument tuned according to SOP specifications?		Yes			
	(2) Were tuning criteria within required specifications?		Yes			
	Internal Standard (IS) Analytes					
	(1) Were IS analytes added to all extracts prior to analysis?		Yes			
	(2) Were IS analyte counts within required criteria?		Yes			
	Raw Data					
	(1) Were raw data reviewed by the analyst?		Yes			
	(2) Are all manual integrations notated properly?		NA			
	(3) Is "Before/After" documentation maintained for all manual integrations?		NA			
	Interference Check Samples (ICS)					
	(1) Were % recoveries within SOP specified QC limits?		Yes			
	Serial Dilutions, Post Digestion Spikes, & Standard Additions					
	(1) Were % differences, recoveries, and linearity within SOP specified criteria?		Yes			
	Method Detection Limit (MDL)					
	(1) Was a current MDL study performed for all reported analytes?		Yes			
	(2) Do raw MDLs agree between field samples, QC samples, & primary test code?		Yes			
	(3) If required, is the MDL supported by analysis of a DCS?		NA			
	Demonstration of Capability (IDC/ODC)					
	(1) Is analyst IDC/ODC up-to-date and on file?		Yes			
	Standard Documentation					
	(1) Are all primary standards traceable?		Yes			
	(2) Are all secondary standards verified with applicable traceability?		Yes			

ER#	***Description***	

A NCAR or ER must be completed for any item where "No" has been documented on the checklist.

REPORT FORMATTING

SOPID:	VAL-ADM-005	Rev. Number:	R01	Effective Date:	01/27/2021

Approved By: Jennifer Hall — Date: 1/27/21
Laboratory Supervisor

Approved By: Chad Storke — Date: 1/27/2021
Technical / Quality Manager

Archival Date:		Doc Control ID#:		Editor:	

PROCEDURAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR 24 ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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REPORT FORMATTING

1) Scope and Applicability

1.1 This SOP provides guidance in the formatting and reporting of analytical results. The results of all reported tests must be presented accurately and in a clear, concise, and unambiguous manner. Following client submittal, copies of the final report are archived. The Project Manager (PM), as directed by the Laboratory Director, is responsible for work order reporting.

2) Summary of Procedure

2.1 Upon the receipt of client's samples, a numeric work order is assigned in LIMS (VAL-SM-001, *Sample Log-in Procedures*). Information for the work order is provided from the Chain of Custody (COC) and associated project information on file with the PM. When the work order is complete, a final report with associated QC is generated, reviewed by the Project Manager, and submitted to the client (VAL-ADM-003). An electronic record of the final report is archived in LIMS and maintained for a period of no less than seven years.

3) Definitions

3.1 LIMS: Laboratory Information Management System

3.2 QA/QC: Quality Assurance/Quality Control

3.3 COC: Chain of Custody

3.4 PM: Project Manager: Individual managing client projects.

4) Health and Safety Warnings

4.1 None

5) Personnel Qualifications and Responsibilities

5.1 The Project Manager is responsible for management of clients' analytical projects, and is therefore responsible for the reporting/formatting of work orders.

5.2 A Project Manager must have a bachelor's degree in Chemistry, Biology, Engineering, Management, or related science and have at least five years environmental laboratory experience.

6) Procedure

6.1 The following items must be included in all analytical reports submitted to the client.

6.1.1 Title of the report
6.1.2 Name and address of the laboratory.
6.1.3 Name and telephone number of laboratory contact
6.1.4 Total number of pages and unique identification of each page
6.1.5 Name and address of the client
6.1.6 Project identification (if available)
6.1.7 Client sample identification
6.1.8 Laboratory sample identification
6.1.9 Date and time of sample collection
6.1.10 Date of sample receipt

6.1.11 Date of analysis
6.1.12 Time of preparation and/or analysis if either holding time is ≤ 72 hours
6.1.13 Identification of test method
6.1.14 Method Detection Limits (47CSR32 compliant samples)
6.1.15 Identification of sampling procedure (if laboratory collected sample)
6.1.16 Any deviations or anomalies that affect the quality of the reported results. These excursions may be indicated by flags or qualifiers and include, but are not restricted to, improper sampling, improper sample containers, temperature and/or preservation anomalies upon receipt, violation of shipping and/or storage requirements, holding times, and failed QC. Narrative text may also be used to define the deviations and/or anomalies (if present).
6.1.17 Definitions of flags or qualifiers (see item 6.1.16).
6.1.18 Measurements or derived results along with the appropriate reporting units. Where applicable, the report must indicate if results are based upon dry or wet weight measurements.
6.1.19 Clear identification of results provided by outside (sub-contracted) sources.
6.1.20 Signature and title of the PM accepting responsibility for issuing of the final report.
6.1.21 Date of issue.
6.1.22 Statement that results relate only to items tested and/or as received by the laboratory.
6.1.23 Statement that the report shall not be reproduced except in entirety without written approval of the laboratory.

6.2 If the report contains test results performed by subcontractors, the reported values must be clearly identified (see Item 6.1.19) along with the subcontractor's name or accreditation link.

6.3 All results reported under NELAC accreditation must be certified to be in compliance with applicable requirements. If such certification is not appropriate, the results must be indicated as such and the reason(s) clearly stated.

6.4 Upon issuance, the final report must remain unchanged. Any previously issued report requiring changes and/or revisions must be supplied to the client clearly identified as a supplement or revision and must provide all items documented in Section 5 of this document.

6.5 After issuance, the client must be immediately informed of any events discovered which affect or cast doubt upon the integrity of previously reported values.

6.6 Reports issued electronically must meet all specifications documented in Section 5 of this document.

6.7 Reports issued electronically must provide for protection of client confidentiality (see VAL-GEN-004, *Client Confidentiality*).

7) Equipment and Supplies

7.1 LIMS

8) Quality Assurance and Quality Control

8.1 An electronic copy of all issued reports (and amended reports if applicable) shall be maintained in LIMS for a period of no less than seven years.

8.2 Copies of any client correspondence pertaining to previously issued reports shall be

maintained in the appropriate Work Order folder.

8.3 Redundant checking must be performed during the PM review of Work Order log in (VAL-SM-003, *Log-In Procedure*).

8.4 Project Management personnel must be trained in the required parts of the final documents and be familiar with this SOP.

8.5 Final Reports are reviewed through QA System Audits and as part of the QA Review process described in VAL-QS-009, *Data Reduction, Review and Reporting*.

9) Summary of Changes

Table 9.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
R00	9/1/2015	CES	New SOP
R01	1/27/2021	LC	Added section 6.1.14 to include MDL in final report for 47CSR32 samples
R01	1/27/2021	LC	Revised section 6.1.16 to include narration of any temperature or preservation anomalies observed during sample receipt.
R01	1/27/2021	CES	Updated cascading references due to addition of section 6.1.14

10) References and Related Documents

10.1 TNI Standard, Quality Systems, Volume 1/Module 2/ Section 5.10, (Adopted September 9, 2009 & Implemented July 1, 2011)

10.2 ALS Environmental Quality Assurance Manual, Revision (most current)

10.3 Table 1 – Flags and Qualifiers

Table 1:

Flags and Qualifiers

a	-	Not Accredited
ND	-	Not Detected at the Reporting Limit
U	-	Analyzed but not detected above the Method Detection Limit
J	-	Analyte is present at an estimated concentration between the MDL and Reporting Limit
B	-	Detected in the associated method blank above the reporting limit
S	-	Spike recovery outside laboratory control limits
R	-	RPD above laboratory control limit
P	-	Dual column results percent difference > 40%
E	-	Value above quantitation range
H	-	Analyzed outside of Holding Time
O	-	Sample amount is > 4 times amount spiked
*****	-	Value exceeded Regulatory Limit
Z	-	Laboratory defined as explained in narrative
X	-	Analyte was detected in the Method Blank between the MDL and PQL, sample results may exhibit background or reagent contamination at the observed level