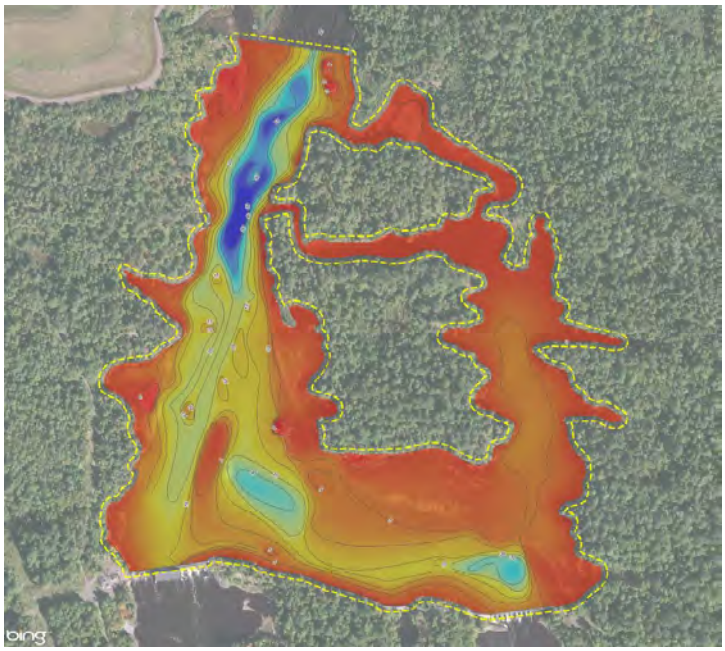
FOCUSED FEASIBILITY STUDY
Scanlon Reservoir

SR#1374
Scanlon, Minnesota
MPCA Work Order #3000026021



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CLEAN WATER LAND & LEGACY AMENDMENT

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June 2020
Revision 00
BWJ200100

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Executive Summary

This Focused Feasibility Study (FFS) for the Scanlon Reservoir (Site) presents a summary of current Site conditions; a discussion of remedial action objectives (RAOs); and the identification, screening, evaluation, and comparison of potential alternatives. This report was prepared by Bay West LLC (Bay West) in accordance with the Minnesota Pollution Control Agency (MPCA) Contract Work Order No. 3000026021.

The Site was studied as a part of the St. Louis River (SLR) area of concern (AOC; see **Section 1.2**). Funding to complete an FFS was obtained through the United States Environmental Protection Agency, Great Lakes Legacy Act, and state funding through the Minnesota Legacy Fund .

Investigations previously completed at the Site identified sediments with elevated concentrations of polychlorinated dibenzo-p-dioxins/dibenzofurans (dioxins) and mercury. Since a remedial investigation (RI) was never conducted for the Site, historical data was reviewed during this FFS process for contaminant of concern (COC) determination. Dioxins were determined to be a COC for sediments at the Site. Mercury was identified and listed as a COC in previous versions of this FFS; however, mercury (as well as methylmercury) has been determined not to be a COC based on bioaccumulation and fish tissue data collected in 2016. Dioxins are determined to be of greater hazard to aquatic life and associated bioaccumulation because contaminated sediments are the primary source of dioxins into the downstream SLR AOC system.

In 2016, data was collected to support previous investigations by addressing data gaps identified by the MPCA to investigate the extent and volume of contaminated sediment within Scanlon Reservoir, and to evaluate risks to human health and the environment due to potential impacts by the benthic community. Sediment samples were collected and analyzed for Site COCs to gather additional chemical data for delineation of the extent and depth of contamination at the Site. Dioxin sediment concentrations exceeded Level II SQTs in 60% of the samples, focused within the back bay of the eastern half of Scanlon Reservoir in both the 0.0 to 0.15 and 0.15 to 0.50-meter intervals. Benthic macroinvertebrates appear to bioaccumulate dioxins due to exposure to Site sediments significantly more compared to reference samples collected from an area reference lake (Boulder Lake). Fish tissue collection and testing of seven fish species indicate that dioxins concentrations are greater than the reference sample and have a statistically significant difference between fish collected from the Site and the reference site. Based on the sediment and tissue testing results, dioxins should be retained as a COC for the Site.

As identified in the SLR Remedial Action Plans (RAPs): RAP Stage I, MPCA and Wisconsin Department of Natural Resources (WDNR), 1992; and RAP Stage II, and MPCA and WDNR, 1995; and later proven with testing, Scanlon Reservoir, SR#1374, Scanlon, Minnesota (**Figure 1**), is potentially contributing to several impairments in the SLR AOC:

- Restrictions on dredging; and
- Degradation of the benthos.

Areas that are contributing to river sediment impairments should be addressed through remedial activities, as recommended by the RAP. In addition, addressing the contaminated sediments within the Site would assist in the reduction of impaired waters resulting from bioaccumulative toxins in the SLR.

Remedial Action Objectives Developed by the MPCA for the Site

RAOs for the Site were developed based on the requirements of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP; 40 Code of Federal Regulations [CFR]

§300.430[e][2][i]), which defines RAOs as a listing of the COCs and media of concern, potential exposure pathways, and remediation goals. Specific RAOs were developed from a review of the results of site characterization activities, site-specific risk and fate and transport evaluations, and an initial review of Applicable or Relevant and Appropriate Requirements.

The following RAOs for the Site include goals for the protection of ecological receptors:

- Minimize or remove exposure to sediment contaminants that bioaccumulate in the food chain.
- Minimize or remove exposure of the benthic organisms to contaminated sediments above sediment cleanup goals.
- Maintain current reservoir operating capacity and functionality.

Alternatives Developed for the Site

Alternatives were identified and screened to determine whether they could meet these RAOs. The following alternatives were evaluated in this FFS:

Alternative 1: No Action – The NCP at Title 40 CFR provides that a No Action Alternative should be considered at every site. The No Action Alternative should reflect the site conditions described in the baseline risk assessment and RI. The No Action Alternative included within this FFS does not include any treatment or engineering controls, ICs, or monitoring. There are no costs associated with the No Action Alternative.

Alternative 2: Enhanced Monitored Natural Recovery with Thin-Layer Amended Cover – This alternative incorporates construction of a 0.30-meter (1.0-foot) thick thin-layer amended sand cover over sediments with COC concentrations exceeding the preliminary cleanup levels (CULs; i.e., areas of the Site with exceedances of the Midpoint Sediment Quality Target [SQT] for dioxins), hereafter referred to as the remedial footprint, an area of approximately 17 acres in size. An amendment material such as activated carbon would be incorporated into the sand cover to facilitate sequestration of sediment contaminants. The objective of constructing an amended thin-layer cover over contaminated sediments would be to (1) reduce bioavailability of Site COCs in sediments and sediment pore water to aquatic organisms (primarily through contaminant sequestration) and thereby limit transfer of chemical contaminants to higher trophic organisms, and (2) provide some immediate isolation of contaminated sediments to aquatic life. Long-term mixing of cover materials into underlying in situ sediments from bioturbation processes could be anticipated and would result in delivery of amendment materials to deeper sediment depths.

Monitoring of chemical concentrations in sediment and cap material, sediment toxicity, and bioaccumulation of COCs in aquatic life would be conducted following the construction phase until sufficient contaminant sequestration, degradation, transformation, or other natural recovery processes reduce risks to acceptable levels. Institutional controls would be implemented to ensure that remedy integrity is maintained. The approximate present value cost associated with Alternative 2 is $8,219,000.

Alternative 3: Potential Bioactive Zone Cap – This alternative incorporates construction of a 0.5- to 1.2-meter-thick sand cap over the same 17-acre remedial footprint as Alternative 2. The constructed cap thickness would be equal in thickness to the Potentially Bioactive Zone, which is determined by the varying habitat areas at the Site (see **Section 1.4.4.2**) and, therefore, provide contaminant isolation from aquatic plant and animal life. Construction of a cap will also mitigate exposure to human receptors, although human health criteria are not being used as cleanup criteria at this time (see **Section 1.4.3.3**). Armoring consisting of gravel and/or cobble would be placed over the sand cap in areas that could experience scouring during abnormally high flow

events, such as the inlets immediately adjacent to the river's main channel. A monitoring period and implementation of ICs would be conducted following the construction phase as detailed for Alternative 2. Monitoring and enforcement of ICs would continue indefinitely until RAOs are achieved for the Site, but a period of 30 years was used for incorporation into each alternative's cost analysis. The approximate present value cost associated with Alternative 3 is $8,508,000.

Alternative 4: Sediment Dredging and Excavation – This alternative incorporates the removal of all sediments contained within the same 17-acre remedial footprint as Alternatives 2 and 3. Sediment removal would be conducted to a target depth ranging from 0.65 meter (2.1 feet) and 0.80 meter (2.6 feet) below the sediment surface. These target sediment removal depths include a Site-wide over dredge/excavation of 0.15 meter (0.5 feet) to increase the efficiency of sediment removal and limit the occurrence of dredge residuals. The total volume of sediment assumed for removal, including over dredge, is approximately 70,000 cubic yards. Some dredge residuals should be anticipated and would be addressed by constructing a 0.15-meter thick sand cover over the entire remedial footprint. In addition to addressing dredge residuals, the constructed sand cover would provide the additional benefit of providing clean substrate for benthic organisms, particularly if bedrock is encountered within the dredge areas.

The shallow eastern "arm" of the Site located outside the primary river channel would be contained within a cofferdam and dewatered so that excavation of contaminated sediments could be conducted "in the dry." All other areas of the Site located outside the cofferdam, which comprise approximately 4 of the 17-acre remedial footprint, would be dredged "in the wet." Monitoring and implementation of ICs was not incorporated into this alternative. The approximate present value cost associated with Alternative 4 is $10,101,000.

Alternative 5: Enhanced Monitored Natural Recovery with Broadcast Amendment – This enhanced monitored natural recovery (MNR) with broadcast amendment alternative would consist of applying a thin 0.01-meter layer of amendment material directly on top of the sediment surface in areas with sediment concentrations of COCs exceeding the preliminary CULs (i.e., areas of the Site with exceedances of the Midpoint SQT for dioxins), hereafter referred to as remedial areas. Amendment material would be mixed into the sediments over time through bioturbation. The chosen amendment would reduce exposure of aquatic life to COCs through sequestration the bioavailable fraction of sediment contaminants. Monitoring of sediment chemical concentrations, sediment toxicity, and bioaccumulation of COCs in aquatic life would be conducted until sufficient contaminant sequestration, degradation, transformation, or other natural recovery processes reduce risks to acceptable levels. A monitoring period and implementation of ICs would be conducted following the construction phase as detailed for Alternative 5. Monitoring and enforcement of ICs would continue indefinitely until RAOs are achieved for the Site, but a period of 30 years was used for incorporation into each alternative's cost analysis. The approximate present value cost associated with Alternative 5 is $3,355,000.

Comparative Analysis Summary

The comparative analysis of the alternatives is presented in **Section 5.0**. Alternatives 2, 3, 4, and 5 were all protective of ecological receptors. Alternative 1 was not protective and will not be considered as a viable remedial alternative for the Site. Based on the comparative analysis, Alternative 3 and Alternative 2 scored the highest and should be evaluated further for remedy selection.

Benchscale treatability testing was completed on sediments collected from the Site to evaluate the effectiveness of different AC amendments and doses to reduce the bioavailability of dioxins/furans in Site sediments using two AC particle size ranges; a silt-sized powdered activated carbon (PAC) and a fine sand-sized granular activated carbon (GAC). The results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4%

dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments. Application methods will be retained for further engineering and cost evaluations.

In order to evaluate how a remedial alternative involving AC might affect mercury, which is present in sediment at the Site but determined not to be a COC, a literature review was completed by the United States Army Research and Development Center. The review focused on how a remedy involving AC will impact the potential for mercury toxicity and bioaccumulation. Review of available research indicated either AC is a useful sorbent for reducing the potential for mercury bioaccumulation, or that AC is not effective at sorbing mercury; however, AC does not appear to increase the potential for mercury toxicity and bioaccumulation in water or sediments (U.S. Army Research and Development Center, 2020).

The MPCA will conduct further outreach activities with the public, resource managers, and local units of government. The modifying criteria, State/support agency acceptance and community acceptance, are assessed formally after the public comment period. These criteria may provide necessary information to assess the preferred alternative.

Further studies are recommended during the design phase of the selected alternative. These recommended studies, depending on the alternative selected, may include:

- Further delineation and determination of extent, thickness, and volume of contaminated sediment.
- Hydrodynamic study to understand natural processes such as depositional and scouring forces to inform design and placement of cover materials, and effectiveness of Monitored Natural Recovery (MNR).
- Updated bathymetric survey and mapping of substrate types.
- Investigation into the potential for ongoing sources related to diffuse regional source and potential upstream historical sources.

TABLE OF CONTENTS

List of Figures

List of Tables

List of Appendices

Acronyms and Abbreviations

% .....percent
µg/kg .....micrograms per kilogram
2,3,7,8-TCDD ...2,3,7,8-tetrachlorodi-benzo-p-dioxin
AOC .....area of concern
ARAR.....Applicable or Relevant and Appropriate Requirement
Bay West.....Bay West LLC
bss .....below the sediment surface
BUI.....beneficial use impairment
CAD .....confined aquatic disposal
CDF .....confined disposal facility
CERCLA .....Comprehensive Environmental Response, Compensation, and Liability Act
CFR .....Code of Federal Regulations
ch. .....chapter or chapters
COC.....contaminant of concern
CSM.....conceptual site model
CUL.....cleanup level
DGI .....Data Gap Investigation
dioxins.....polychlorinated dibenzo-p-dioxins/dibenzofurans
DRO.....diesel range organics
EA.....EA Engineering, Science, and Technology, Inc., PBC
EMNR .....enhanced monitored natural recovery
FERC .....Federal Energy Regulatory Commission
FFS.....Focused Feasibility Study
GHG.....Greenhouse Gas
GLI.....Great Lakes Initiative
GSR.....Green Sustainable Remediation
IC .....institutional control
ITRC .....Interstate Technology and Regulatory Council
IZ .....Isolation Zone
LTM.....long-term monitoring
MDH.....Minnesota Department of Health
MDNR .....Minnesota Department of Natural Resources
MERLA.....Minnesota Environmental Response and Liability Act
mg/kg .....milligrams per kilogram
MNR .....Monitored Natural Recovery
MPCA .....Minnesota Pollution Control Agency
NCP.....National Oil and Hazardous Substances Pollution Contingency Plan
ng TEQ/kg .....nanograms toxic equivalency per kilogram
ng/kg.....nanograms per kilogram
NPDES .....National Pollutant Discharge Elimination System
O&M .....operation and maintenance
OIRW.....Outstanding International Resource Water
ORO .....oil range organics
PAH.....polycyclic aromatic hydrocarbon
PBAZ.....Potentially Bioactive Zone
PCB.....polychlorinated biphenyl
RAO.....Remedial Action Objective
RAP.....Remedial Action Plan
RCRA .....Resource Conservation and Recovery Act
RI.....remedial investigation
RME .....reasonable maximal exposure
ROM.....rough order of magnitude
RRO .....residual range organics
SDS.....State Disposal System
SLR .....St. Louis River
SQT.....sediment quality target
SSV .....Sediment Screening Value
TBC .....to be considered
TEQ.....toxicity equivalent
U.S. .....United States
UECA .....Uniform Environmental Covenants Act
USACE .....United States Army Corps of Engineers
USC.....United States Code
USEPA .....United States Environmental Protection Agency
WCA.....Wetland Conservation Act
WDNR .....Wisconsin Department of Natural Resources
Weston .....Weston Solutions, Inc.
WLSSD.....Western Lake Superior Sanitary District

1.0 INTRODUCTION AND BACKGROUND

The St. Louis River (SLR), located on the border between Minnesota and Wisconsin, is the second-largest United States (U.S.) tributary to Lake Superior and has a special significance in the region. The lower estuary empties into the Duluth-Superior Harbor, the largest freshwater seaport in North America. It serves as a geographic boundary for Wisconsin and Minnesota and provides regional shipping access to Lake Superior.

Development along the SLR over the past 130 years has contributed to contaminated sediments. In 1987, concerns over environmental quality conditions prompted the designation of 73 miles of the lower SLR, which includes the segment from Cloquet, Minnesota, to the Duluth/Superior Harbor, as 1 of 43 Great Lakes Areas of Concern (AOCs). The Minnesota Pollution Control Agency (MPCA) and Wisconsin Department of Natural Resources (WDNR) worked together to divide the SLR AOC into Sediment Assessment Areas for the purposes of evaluation and prioritization of remediation and restoration activities. Contaminated sediments have been identified and characterized through several studies that included the collection and analysis of sediments and biota samples throughout the AOC.

Historical sediment contamination in the SLR AOC has resulted in impaired uses, including degradation of bottom-feeding invertebrate communities, increased incidence of fish tumors and other abnormalities, fish consumption advisories, and restrictions on dredging, resulting in nine beneficial use impairments (BUIs; MPCA, 2008). BUIs are a change in the chemical, physical or biological integrity of the Great Lakes system sufficient to cause any 1 of the 14 established use impairments, or other related uses, such as the microbial objective for waters used for body contact recreational activities (joint commission). The MPCA and WDNR are currently working together to implement a comprehensive long-term plan to restore beneficial use and delist BUIs in the SLR AOC. Many of the BUIs in the AOC are linked to the presence of sediment contaminants. Some sediment-derived contaminants also appear suspended in the water column and are carried by the SLR to Lake Superior.

As identified in the SLR Remedial Action Plans (RAPs): RAP Stage I, MPCA and WDNR, 1992; and RAP Stage II, MPCA and WDNR, 1995; and later proven with testing, the Site is potentially contributing to two impairments in the SLR AOC:

- Restrictions on Dredging; and,
- Degradation of the benthos.

Areas that are contributing to river sediment impairments should be addressed through remedial activities, as recommended by the RAP. In addition, addressing the contaminated sediments within the Site would assist in the reduction of impaired waters resulting from bioaccumulative toxins in the SLR.

This Focused Feasibility Study (FFS) was prepared to evaluate remedial alternatives for contaminated sediment at the Site. The scope of this FFS does not consider alternatives for any other matrix such as soil, surface water, or groundwater that may be impacted at the Site.

This report has been developed pursuant to the Bay West LLC (Bay West) Master Contract No. 142504 and MPCA Contract Work Order No. 3000026021, effective March 2, 2020, and accompanying the Scope of Work/budget for the Site. Funding to complete the FFS for the Site comes from the U.S. Environmental Protection Agency (USEPA), Great Lakes Legacy Act, and state funding through the Minnesota Legacy Fund.

This FFS was written in general accordance with the MPCA Site Response Section Guidance Document Draft Guidelines on Remedy Selection (MPCA, 1998), the Minnesota Environmental

Response and Liability Act (MERLA), the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 Code of Federal Regulations (CFR) Part 300, along with other Minnesota and Federal rules, statutes, and guidance.

1.1 Report Organization

Section 1.0 presents general background information including the Site history and a summary of current Site conditions. **Section 2.0** discusses Applicable or Relevant and Appropriate Requirements (ARARs) and summarizes Remedial Action Objectives (RAOs) to provide the framework for alternative evaluations for the Site. **Section 3.0** and **Section 4.0** present alternatives descriptions and the NCP remedy selection criteria used in this FFS. **Section 5.0** presents an evaluation of alternatives against standards and criteria. References are presented in **Section 6.0**.

1.2 Site Location and Current Use

The SLR estuary covers an area of approximately 12,000 acres and comprises numerous large bays, peninsulas, and islands (**Figure 1**). Upstream of the AOC, the river is characterized by shallow meanders and sandy gravel bars. The character of the river changes abruptly near Cloquet, Minnesota, as it starts its steep descent to Lake Superior. This portion of the watershed is characterized by deeply incised river channels and canyons. Five dams were constructed on this reach of the river, resulting in the creation of five reservoirs within the AOC that may significantly impact downstream flow and water levels. As the river approaches Lake Superior, the current dissipates and the SLR takes on the characteristics of a lake. Just prior to entering Lake Superior at the Duluth Ship Canal and the Superior Entry, the river forms a large embayment protected by two long sandbars (i.e., Minnesota and Wisconsin Points). These sandbars form the longest natural freshwater baymouth sandbars in the world. Two inner spits, Rice's Point and Conner's Point, divide the port into inner and outer harbors (Crane et al., 1996).

Scanlon Reservoir (the Site) is one of five reservoirs downstream of Cloquet, Minnesota, that regulates streamflow into the downstream portion of the SLR. The other four reservoirs are Northwest Paper (Potlatch), Thomson, Fond du Lac, and Knife Falls. A hydroelectric power station located at the southern (downstream) portion of the Site is owned and operated by Minnesota Power and has the capacity to generate up to 1.6 megawatts of electricity for the electrical grid. The power generating and dam infrastructure consists of four major components: a west channel dam; an east channel dam; and two non-overflow gravity dams that are located on the island that separates the east and west channels of the river. The west channel structures consist of two Tainter gated spillways flanking a powerhouse and a short concrete gravity non-overflow section keyed into the island bedrock (Schubauer-Berigan and Crane, 1996b). The Scanlon hydroelectric station is a "run of river" station, meaning it doesn't store water in a reservoir for future use. Rather, the station relies on the water that is currently available to generate electricity. As water flows downstream, some of it is diverted through turbines in a powerhouse and then is immediately released back into the river (Minnesota Power, 2014).

The Site is located immediately east of Scanlon, Minnesota, and downstream of the nearby city of Cloquet, Minnesota (**Figure 2**). The Site is approximately 40 acres in size and consists of the water body upriver of the dams. The Site is bounded on all sides by forested lands owned by Minnesota Power and Sappi Cloquet, LLC. The forested lands extend for nearly 1 mile to the east, northeast, and north, after which low-density residential neighborhoods and farmland are present. Upriver and to the northwest are the cities of Scanlon and Cloquet, and the location of former industries that discharged into the SLR up until 1979. The Site is bounded to the west by a small strip of forested land between the Site and State Highway 45. Beyond State Highway 45 are

residential neighborhoods associated with the City of Scanlon. The Site is bounded to the south by the Scanlon hydro station and associated dams. Beyond the hydro station are downstream portions of the SLR along with a mix of residential properties, commercial properties, and forested lands.

Wetlands at the Site are classified as freshwater emergent wetlands, classification code PEM1F. Wetland areas are located at the far northern portion of the Site and appear to have a combined size of approximately 1 acre. The remainder of the Site is classified as lake, classification code L1UBHh. The size of the lake area is approximately 43 acres (data retrieved from http://www.fws.gov/wetlands/Data/Mapper.html).

The section of the SLR between Scanlon and Thomson Reservoirs is a popular location for white water kayaking. This 5 mile stretch of river has class 2 through class 6 rapids and is publicized as a kayaking destination on the Minnesota Department of Natural Resources (MDNR) website (http://www.dnr.state.mn.us/watertrails/trips.html) and other public information documents (http://files.dnr.state.mn.us/maps/canoe_routes/stlouislower.pdf). There is public water access located approximately 0.2 miles downstream of the Site and the hydro station. The trail that leads to the public water access appears to continue north unimpeded past the hydro station and alongside the western border of the Site. Although no official recreational use of the Site is known, it is likely that the Site is used recreationally due to readily available access.

1.3 Site History (From Schubauer-Berigan and Crane, 1996a)

Little is known about the area of the Scanlon Reservoir prior to 1922; records only indicate that it was the site of the old Scanlon wagon bridge. In 1922, the St. Louis River Improvement Company began construction of the Stevens Dam, now referred to as the Scanlon Dam. The dam was designed by R. D. Thomas Consulting Engineers and was built by Siems, Helmers and Schaffner Contractors. The Site has been used for hydroelectric generation since construction was completed in 1923.

Historical discharge directly to the SLR, upriver of the Site, includes the following: municipalities, building materials manufacturing, paper manufacturing, and match manufacturing. There is little detail available on the chemical constituents of the waste streams of the abovementioned discharges. Research is being completed on historical industrial activities in the area; however, at the time of publication of this FFS, research has not been completed. Available data for two facilities (the building materials manufacturer and the paper manufacturer) are discussed below.

The building materials facility manufactured acoustical tile and a cushioning material in automobile dashboards, shoes, and other items. Select analytical data was available from the waste stream for 1975 and 1977. The 1975 effluent data showed elevated metals and phenols (sample was not analyzed for mercury) and 1977 effluent data showed elevated mercury (sample was not analyzed for polychlorinated biphenyls [PCBs] and polychlorinated dibenzo-p-dioxins/dibenzofurans [dioxins]), when compared to current water quality data.

The paper manufacturer began operation in 1928, located half a mile downstream of Cloquet. Wastewater was discharged from this facility for approximately 50 years. Wastewater over the course of the plants' operation went through various forms of treatment, some more protective than others. Effluent from the paper manufacturer was analyzed from two outfalls in 1975 and 1977 for heavy metals and phenols. For the 1975 and 1977 effluent analytical data, mercury and phenols were elevated when compared to current water quality standards. Mercury levels in the effluent from 1950 to 1960 were likely higher than levels observed in the 1975 and 1977 effluent based on the widespread use of mercury as a fungicide in the paper industry at the time.

Dioxins and PCBs were not measured in the paper manufacturer effluent during the 1975 and 1977 sampling events. In 1987, due to increased concern over dioxins contamination, effluent from the paper manufacturer (which had begun discharging to the Western Lake Superior Sanitary District [WLSSD] in 1979) and WLSSD influent were analyzed for dioxins. Dioxins refer to a broad class of compounds that vary in toxicity. To minimize sampling completed for the effluent and influent 2,3,7,8-tetrachlorodi-benzo-p-dioxin (2,3,7,8-TCDD), the most toxic dioxins congener, was analyzed. From the results of these analyses pre-1979 dioxins discharges to the SLR can be inferred. The concentration of 2,3,7,8-TCDD in suspended solids from WLSSD influent was 260 nanograms per kilogram (ng/kg) and effluent concentrations were 620 ng/kg, corresponding with estimates that the paper manufacturer contributes to nearly half of WLSSDs influent stream.

Potential nonpoint contaminant sources upgradient of the Site may include the following: landfills/hazardous waste sites, agricultural/forestry runoff, stormwater discharge, unsewered community discharge, recreation and commercial activity, spills, and atmospheric deposition.

Based on available analytical information and known contributor processes potential contaminates in the Site may include mercury, heavy metals, phenols, PCBs, and dioxins.

1.4 Site Characterization

1.4.1 Site Geology

Regional geology in the Duluth area consists primarily of materials deposited during the last glaciation, and more recently as river sediment, overlying Precambrian igneous and sedimentary bedrock. These materials consist of silts, sands, and gravels that were deposited as the glaciers retreated northward. Fine grained sediment, primarily red silt and clay, was deposited in the ancestral glacial Lake Duluth. This red silt and clay occurs over much of the lower elevations in the Duluth area.

Bedrock units underlying the region consist of olivine gabbro and anorthositic gabbro members of the Duluth Complex, and the sedimentary units of the Fond du Lac Formation. The Duluth Complex is lower Precambrian, and the Fond du Lac Formation is upper Precambrian in age. The gabbroic members of the Duluth Complex form the hills to the west of the SLR and Lake Superior shore (MPCA, 1995).

1.4.2 Site Hydrology

The Site is located in the SLR Watershed. Although a site-specific groundwater study has not been performed, local groundwater flow in surficial sediments and bedrock is generally depicted in the County Atlas Series, Atlas C-19, Part B, Plate 7 and Plate 9, respectively. Generally, groundwater in surficial sediments and bedrock within the vicinity of the Site flows towards the SLR, emptying into the Site.

Scanlon Reservoir along with other impoundments located on the SLR slow the flow of water and reduce the river's gradient, thus resulting in the deposition of sediments. Larger reservoirs such as the Thomson and Fond du Lac Reservoirs have greater surface areas, greater water column depths, and longer sediment settling times, which results in increased sediment deposition. MPCA staff were unable to locate deep sediments in the Scanlon areas during scouting of appropriate depositional locations for the preliminary contaminant assessment of the Thomson, Forbay, and Fond Du Lac Reservoirs (Schubauer-Berigan and Crane, 1996a), suggesting Scanlon Reservoir is a net erosional zone. Areas outside of the main river channel such as the eastern portion of the Site may be more prone to sediment deposition. This is evidenced by the increased concentrations of contaminants in these areas.

Maximum streamflow in the SLR normally occurs during spring snowmelt. Periods of low flow generally occur in late summer or late winter. The lowest annual flows usually occur in late winter when precipitation is normally in the form of snow. The median low flow measured at Scanlon from 1909 to 1978, was 20 percent (%) of average flow. The ordinary high flow is 6.4 times higher than the average flow. The river's mean annual discharge is 2,300 cubic feet per second (MPCA and MDNR, 1992).

The Site's hydro station is operated as a run-of-river facility whereby inflows at the dam match outflows to the greatest extent possible. To achieve this mode of operation, a programmable controller operating in conjunction with a pond water level sensor is utilized to bring the units on or offline as required. Under normal operating conditions, the pond level at the dam is maintained at Elevation 1119.8±0.5 feet (Schubauer-Berigan and Crane, 1996a).

1.4.3 Nature and Extent of Contamination

Several investigations have been conducted at or near the Site since 1970 and have included analysis of chemical compounds in sediments and fish. These studies are identified in **Sections 1.4.3.1** and **1.4.3.2** and available historical sediment data for the Site is summarized in **Table 1**. This section also presents a discussion of the contaminants of concern (COCs) and the known extent of sediment contamination at the Site.

1.4.3.1 Previous Investigations

- Table 2-4 of the Preliminary Contaminant Assessment of the Thomson, Forbay, and Fond Du Lac Reservoirs (Schubauer-Berigan and Crane, 1996a) indicates that mercury data was collected at the Scanlon Reservoir during a 1970 survey performed by the Federal Water Quality Association. The source location of this data could not be found.
- Tables 2-5 and 2-7 of this same report indicate that PCB and fish tissue data was collected at the Scanlon Dam/Reservoir during a comprehensive sediment survey performed by the MPCA in 1980. A reference to the source location of this data could not be found.
- Table 2-6 of this same report indicates that TCDD data was collected at the Scanlon Reservoir in 1987 and included within the Legislative Commission on Minnesota Resources Dioxin database, Version 1.1.
- Table 2-4 of this same report indicates that mercury data was collected at the Scanlon Dam in 1989 and included within a Report to the Legislative Commission on Minnesota Resources.
- A Fond du Lac Reservation study was conducted during the summer of 1994 and included collection of approximately 75 sediment cores in the Knife Falls, Potlatch Dam, Scanlon, Thomson, Forbay, and Fond du Lac Reservoirs for analysis of mercury, per the SLR RAP Progress Report (MPCA and WDNR, 1995). The source location of this data could not be found.

1.4.3.2 Previous Reports

The following is a list of previous reports and associated studies conducted at the Site that included the collection and analysis of sediment data:

- Weston Solutions, Inc. (Weston), 2012, "Sediment Assessment Report, Upper St. Louis River – St. Louis River AOC, Cloquet, Carlton and St. Louis County, Minnesota", July. Three locations were sampled up to 0.9 meter (2 feet 10 inches) below the sediment surface (bss) during summer of 2011. Analytical completed includes: semi-volatile organic compounds (SVOCs) including polycyclic aromatic hydrocarbons (PAHs), diesel range

organics (DRO)/oil range organics (ORO), dioxins, pesticides, metals, total organic carbon, and grain size.

- EA Engineering, Science, and Technology, Inc., PBC (EA), 2015, "Site Characterization Report, Assessment of Contaminated Sediment, St. Louis River Site Characterization, St. Louis River and Bay area of concern (AOC), Duluth, Minnesota"; USEPA, Great Lakes National Program Office, Chicago, Illinois, EP-R5-11-10. Thirteen locations were sampled up to 0.8 meters (2 feet 6 inches) bss during summer of 2014. Analytical completed includes PAHs, DRO/residual range organics (RRO), PCBs, dioxins, pesticides, and metals. The hand core sampler used was only capable of collecting samples in less than 5 feet of water; therefore, all sediment cores were collected from near-shore locations.
- *Scanlon Reservoir Technical Memorandum, Scanlon Reservoir, Cloquet, Minnesota, May 2017 (2017 Tech Memo)*

  In 2016, data was collected to support previous investigations by addressing data gaps identified by the MPCA to investigate the extent and volume of contaminated sediment within Scanlon Reservoir, and to evaluate risks to human health and the environment due to potential impacts by the benthic community (2016 Data Gap Investigation [DGI]). Sediment samples were collected and analyzed for Site COCs to gather additional chemical data for delineation of the extent and depth of contamination at the Site. Mercury sediment concentrations did not exceed Midpoint SQTs with the exception of one sample, indicating that mercury contamination appears to be relatively limited throughout the Site. Dioxin/furan sediment concentrations exceeded Level II SQTs in 60% of the samples, focused within the back bay of the eastern half of Scanlon Reservoir in both the 0.0 to 0.15 and 0.15 to 0.50-meter intervals.

  Benthic macroinvertebrates do not appear to bioaccumulate mercury due to exposure to Site sediments significantly more compared to reference samples. Benthic macroinvertebrates appear to bioaccumulate methylmercury and dioxins due to exposure to Site sediments significantly more compared to reference samples.

  Fish tissue collection and testing of seven fish species within trophic levels 2 through 4 was completed by the MCPA at the Site and reference the Site. Concentrations of mercury in fish tissue were not observed to vary greatly between the Site and the reference Site. Statistically, mercury concentrations appear to be comparable to the reference sample and coincide with the trophic level for both Site fish tissue samples and reference samples. Concentrations of methylmercury in fish tissue were observed to have a statistically significant difference between fish collected from the Site and from the reference site within tropic Level 3. Dioxins/furans concentrations in fish tissue were greater in tissue collected from the Site compared to the reference site and concentrations have a statistically significant difference in all trophic levels between Site samples and reference Site samples.

  Based on the sediment and tissue testing results, dioxins/furans should be retained as a COC for the Site. Methylmercury may also be bioconcentrating in tissue at the Site; however, additional evaluation is required before methylmercury is considered as a COC. The 2017 Tech Memo is included in **Appendix A**.
- A literature review was completed by the United States Army Research and Development Center. The review focused on how a remedy involving AC will impact the potential for mercury toxicity and bioaccumulation. Review of available research indicated either AC is a useful sorbent for reducing the potential for mercury bioaccumulation, or that AC is not effective at sorbing mercury; however, AC does not appear to increase the potential for

mercury toxicity and bioaccumulation in water or sediments (U.S. Army Research and Development Center, 2020).

As described in the following subsections, results of these investigations indicated the presence of sediment impacts located primarily in low-energy areas within the eastern half of the Site. As no official remedial investigation (RI) was conducted for the Site, historical data from 2011 and 2014 was evaluated as part of this FFS and COCs for the Site were determined. Screening criteria is described in **Section 1.4.3.3** and sediment contaminants are further detailed in **Section 1.4.3.4**.

1.4.3.3 Screening Criteria

Numerical sediment quality targets (SQTs), adopted for use in the SLR AOC to protect benthic invertebrates can be used throughout Minnesota as benchmark values for making comparisons to surficial sediment chemistry measurements. Level I and Level II SQTs for the protection of sediment-dwelling organisms are available for 8 trace metals, 13 individual PAHs, total PAHs (all 13 priority PAHs), total PCBs, and 10 organochlorine pesticides. In addition, Level I and Level II SQTs for dioxins were adopted for the protection of fish, as insufficient information is available for sediment-dwelling organisms. The dioxins SQT is based on the dioxin toxicity equivalent (TEQ) value, which incorporates results of individual dioxin and furan congeners and toxicity equivalence factors for the protection of fish, denoted as TEQ Fish. SQTs are highly useful when evaluating risk for a specific compound or a group of compounds (i.e., total PCBs and total PAHs).

Contaminant concentrations below the Level I SQTs are unlikely to have harmful effects on sediment-dwelling organisms (i.e., benthic invertebrates). Contaminant concentrations above the Level II SQTS are more likely to result in harmful effects to benthic invertebrates (MPCA, 2007). Based on conversations with the MPCA, a qualitative comparison value midway between the Level I SQTs and Level II SQTs (i.e., Midpoint SQT) were used as criteria to identify, rank, and prioritize sediment-associated COCs within the Site.

Sediment Screening Values (SSVs) were developed to provide a human health-based toxicity value specifically related to sediment for the U.S. Steel Superfund site in the SLR (Minnesota Department of Health [MDH], 2013). The SSVs were developed using reasonable maximal exposures (RMEs) specific to the U.S. Steel site and the Lower SLR. The Updated Human Health Screening Values for St. Louis River Sediments: U.S. Steel Site, dated April 2013, describes the updated SSVs utilized in this report. SSVs were compared to select PAHs, eight trace metals, and total dioxins (as TEQs for human health). Chemical concentrations in water-covered sediments at or below the SSVs are considered safe for the general public; however, chemical concentrations in sediments exceeding the SSVs should not be considered unsafe because the SSVs were developed using conservative measures of exposure, bioavailability, and toxicity. Based on ongoing ambient concentration studies, some SSVs likely approach, or are less than ambient concentrations in sediment, including SSVs for mercury, benzo(a)pyrene equivalents, PCBs, and dioxins. Further, the SSVs do not include RMEs specific to the Site and are not intended to be used as sediment cleanup values; therefore, SSVs will not be used to identify, rank, and prioritize sediment-associated COCs within the Site. Instead, the respective Midpoint SQT will be used to identify, evaluate, and prioritize sediment-associated COCs within the Site.

1.4.3.4 Contaminants of Concern

Sediment data collected during the 2011 Weston, 2014 EA, and 2016 DGI investigations was reviewed and used to determine COCs for the Site. During the data evaluation process, actual sediment sample intervals were manipulated and categorized into four discrete depth intervals to

allow for ease of interpretation of contaminant depth across the Site. The selected intervals allow for relatively easy assessment of sediment quality. The various selected intervals are as follows:

- 0.0 to 0.15 meters
- 0.15 to 0.50 meters
- 0.50 to 1.00 meters
- >1.00 meter

Each sediment sample was categorized into one of the two intervals of at least 25% of the sample length was within an interval. For example, if a sample was collected from 0.30 to 0.55 meters bss, the sample was categorized in the 0.15-to 0.50-meter category. Occasionally, at least 25% of a sample was collected within two intervals. For example, if a sample was collected from 0.10 to 0.30 meters, 25% of the upper portion of the sample is within the 0.0- to 0.15-meter interval, and 75% of the lower portion of the sample is within the 0.15- to 0.50-meter interval. In these cases, the sample was considered in the discussion and evaluation of both the 0.0- to 0.15-meter interval and the 0.15- to 0.50-meter interval. In order to examine all data in a single data set, data from each of the four intervals were combined into a single group called “All Intervals.”

Mapped data from the 2011, 2014, and 2016 investigations was reviewed for total PAH-13, total PCBs, select metals, and/or dioxins as TEQ KM Fish; data was reviewed as exceedances of established SQTs within the intervals defined above. Pesticides data contained within original report tables was also reviewed for those compounds with established SQT values. Data was not reviewed for those compounds that do not have established SQT values, including non-PAH-13 SVOC compounds, select pesticides, select metals, and DRO, ORO, and RRO hydrocarbon compounds.

Table 1 presents a statistical summary of historic analytical data evaluated against the SQTs and categorized by the sample intervals detailed previously. The table provides data for all compounds or compound groups that had at least one exceedance of their respective Level I SQT, and also presents the Level I, Midpoint, and Level II SQT screening levels. SSVs for the protection of human health were included within the summary table but were not used for COC determination per **Section 1.4.3.3**.

Through discussions with the MPCA and review of available sediment analytical data, it was determined that for the purposes of this FFS, any contaminant exceeding the associated Midpoint SQT will be considered a COC. A COC Summary is included in **Table 2**. A summary of the data review by contaminant group is provided below.

PAHs

The mean concentration of total PAH-13 over all intervals was 662 micrograms per kilogram (µg/kg), well below the Level I SQT of 1,600 µg/kg. One of the 23 samples had PAH concentrations exceeding the Level I SQT and occurred in the 0.0- to 0.15-meter interval; no samples exceeded the total PAH-13 Midpoint or Level II SQTs. PAHs are not considered a COC for the Site.

PCBs

The mean concentration of total PCBs over all intervals was 63.8 µg/kg, slightly above the Level I SQT of 60 µg/kg. Nine of the 27 PCB samples collected exceeded the Level I SQT and all exceedances occurred in the 0.0- to 0.15-meter interval; no samples exceeded the total PCBs Midpoint or Level II SQTs. PCBs are not considered a COC for the Site.

Metals

Cadmium, copper, nickel, and zinc each had at least one exceedance of their respective Level I SQT. No samples exceeded respective Midpoint or Level II SQTs for these compounds. The mean concentration of mercury over all intervals was 0.32 milligrams per kilogram (mg/kg), half of the Midpoint SQT of 0.25 mg/kg. Seventeen of the 59 samples collected exceeded the Level I SQT, 7 exceeded the Midpoint SQT, and 5 exceeded the Level II SQT. A total of 10 of the Midpoint or Level II SQT exceedances occurred within the 0.0- to 0.15-meter interval and 7 occurred within the 0.15- to 0.50-meter interval. Only a single sample was collected from the 0.50- to 1.0-meter interval; this sample exceeded the Level I SQT. Mercury was also assessed in benthic invertebrate and fish bioaccumulation studies in the 2016 DGI and was found to not bioaccumulate in tissue more than reference areas. Based on sediment and bioaccumulation results, mercury is not considered a COC for the Site as only 12% of samples exceeded the midpoint SQT and does not significantly bioaccumulate in benthic invertebrate or fish tissue. Methylmercury at the Site was evaluated and researched and has been determined to not be considered a COC for the Site. This is further discussed in **Section 1.4.3.2**.

Dioxins

The mean concentration of dioxins as TEQ KM Fish over all intervals was 37.74 ng/kg, substantially greater than the Midpoint SQT of 11.2 ng/kg. Forty-four of the 48 samples collected exceeded the Level I SQT, 23 exceeded the Midpoint SQT, and 20 exceeded the Level II SQT. A total of 28 of the Midpoint or Level II SQT exceedances occurred within the 0.0- to 0.15-meter interval and 23 occurred within the 0.15- to 0.50-meter interval. Only a single sample was collected from the 0.50- to 1.0-meter interval; this sample exceeded the Level I SQT. Dioxins were also assessed in benthic invertebrate and fish bioaccumulation studies in the 2016 DGI and were found to bioaccumulate in tissue significantly more than reference areas. Based on sediment and bioaccumulation results, dioxins are considered a COC for the Site due to numerous exceedances of the Midpoint SQT.

Pesticides

All compounds with established SQT values, and the majority of compounds without established SQT values, had non-detections or were detected below the limit of quantitation. All detection limits were below the Midpoint SQT for individual compounds except for lindane (gamma-BHC) and toxaphene. The Midpoint SQT for lindane is 3.7 µg/kg while the detection limit at location STL14-SR13 was 4.7 µg/kg. The Midpoint SQT for toxaphene is 16 µg/kg while the detection limit at all locations was 230 µg/kg or greater. Lindane and toxaphene are not anticipated to be present at the Site based on observed concentrations of the other pesticides analyzed.

Summary

Dioxins were retained as a COC for the Site. **Figure 5** presents the estimated areas of dioxins contaminated sediment that may exceed their respective Midpoint SQT.

1.4.3.5 Depth and Volume of Contaminated Sediment

The depth and volume calculations and assumptions discussed below are based on sediment chemical data collected during 2011, 2014, and 2016, historic bathymetric data, and water depths collected during field investigations. The most recent bathymetric survey data that could be obtained for the Site was completed in 1974 (MDNR, 2016). A large flooding event occurred in 2012 and may have impacted sediment distribution at the Site since the bathymetric survey date. It was assumed that the survey data is accurate for the purposes of this report, but a new bathymetric survey may need to be completed and assumptions updated at a future date.

The total area of the Site is approximately 40 acres. The 1974 MDNR Bathymetric Survey and satellite imagery indicate that the Site consists of the following features: a “primary” river channel extending north south along the western end of the Site and flowing out of the Site over the western dam; a “secondary” channel extending off the primary channel, which extends east west and flows out of the Site over the eastern dam; and a low energy area of the Site located along the eastern Site boundary, hereafter referred to as the “eastern arm.” The eastern arm comprises approximately 11 acres of the Site and is anticipated to contain the majority of sediments exceeding screening criteria (i.e., Midpoint SQT values for dioxins). There are also several inlets adjacent to the primary river channel that are anticipated to be low energy areas and consist of deposited sediments. These areas total approximately 3.5 acres in size and also contain sediments exceeding screening criteria.

Water depth ranges greatly within the Site boundary. Average maximum water depths within the river channel center ranges from 15 feet to 65 feet, while average maximum water depth within the eastern arm is approximately 5 feet. Based on aerial imagery and site observations, there are two locations at the northern end of the Site where water could enter the eastern arm during high water. These inlets likely allowed historical contamination to enter the eastern arm of the Site. The inlets appear to be relatively narrow and flow through these inlets is substantially less than through the primary river channel. Any suspended sediments carried through the inlets would presumably settle out as the expansive, low-energy areas of the eastern arm were encountered.

Historical sample data is presented in **Figures 4** for dioxins. Dioxins results consist of data from 17 sample locations from within or near the river channel and 15 locations from within the eastern arm. Three of the 17 sample locations within the river channel had exceedances of the Midpoint SQT for dioxins. Exceedances occurred within the 0.0- to 0.15-meter interval at one of the locations and the 0.15- to 0.50-meter interval at the other two. Water depth at both locations was less than 4 feet. With the exception of one location, all the sample locations within the eastern arm had exceedances of the Midpoint SQT for dioxins. Exceedances occurred at all sampled intervals. The maximum depth of contamination observed at the Site was 0.61 meter (2.0 feet). The vertical extent of dioxins contamination has not been defined within depositional areas of the Site.

Dioxin contamination appears to be present deeper within sediments located within the eastern arm. Dioxins contamination also appears to be isolated to depositional areas located adjacent to the primary river channel and the eastern arm. The presence of contaminants at the sediment surface indicate that the 2012 flood event either (1) did not substantially alter sediments within the eastern arm of the Site; or (2) did scour away contaminated sediments but substantial contamination still exists. Pre-flood data from 2011 indicates that a substantial amount of clean sediment had not deposited over contaminated sediments prior to the 2012 flood event.

It is assumed for the purposes of this FFS that depositional areas as shown on **Figure 5** that were sampled during the 2016 DGI contain sediments that exceed the Midpoint SQT for dioxins. These depositional areas were selected and incorporated into the estimated area of contamination based on bathymetry and aerial imagery showing emergent vegetation and/or shallow sediments. The total remedial footprint acreage of the Site for the purposes of this FFS is approximately 16.5 acres.

Depth of contamination was estimated at 0.50 meter (1.6 feet) within depositional areas adjacent to the primary river channel. This equates to a total volume of approximately 6,500 cubic yards. It is likely that not all depositional areas as outlined on **Figure 5** contain sediments exceeding Midpoint SQTs for dioxins; however, it is possible that some areas have exceedances deeper than 0.50 meter. Depth of contamination was estimated at 0.65 meter within all areas of the eastern arm. This estimate is supported by exceedances of the SQT for dioxins to depth (0.61

meter), the collection of sediment cores to refusal, and sediment poling. Refusal encountered during 2016 DGI sediment poling indicates a firm, possibly native, confining layer at an average of 0.58 meter in the eastern arm. This equates to a total volume of 55,000 cubic yards. A 0.15-meter over dredge was assumed over the entire remedial footprint to improve sediment removal efficiency and mitigate the occurrence of dredge residuals. The total volume of sediment assumed for removal, including over dredge, is 68,000 cubic yards.

1.4.4 Exposure Pathways

Exposure pathways represent the linkages among contaminant sources, release mechanisms, exposure pathways and routes, and receptors to summarize the current understanding of the risks to human health and the environment due to contamination. A “complete” exposure pathway means that evidence exists that a COC may be released from a source and may be transported into and through the environment to an exposure point where a receptor is assumed to be present.

The following sections provide greater detail on the human health and ecological exposure pathways.

1.4.4.1 Human Health Exposure Pathways

The Site is in a semi-rural area and located immediately east of the City of Scanlon. The surrounding properties consist primarily of forested, undeveloped lands owned by Minnesota Power and Sappi Cloquet, LLC. Access to the Site is potentially available via a public parking lot located approximately 0.25 mile south of the Site and a trail extending to the Site and near its western shoreline. Although swimming and/or wading are not prohibited by the State or Minnesota Power, no official public swimming beaches are located on the Site and exposure by the use is assumed to be minimal. The portion of the SLR directly downstream of the Site is a popular kayaking route. It is possible that kayakers also use the Site, although a hydroelectric dam separates the Site from the portion of the SLR commonly used by kayakers and no official portage is present between the Site and the sanctioned kayaking route. Fishing is also a popular recreational activity within the Duluth area. It is likely that fishing is actively conducted within the Site; however, due to limited access to the Site, and no public fishing docks or boat launches, significant fishing traffic at the Site is unlikely. Direct exposure to contaminated sediments by the public is possible given the shallow depth to sediments in some areas of the Site but unlikely do due to limited access to the public. All information to date indicates that the proposed future use of the Site is consistent with the current use.

Fish consumption advisories are in effect for selected fish species in the SLR AOC due to elevated concentrations of PCBs and mercury found in fish tissue (MDH, 2014). No fish consumption advisory is currently in place for dioxins and MDH does not currently provide meal advice based on dioxins in fish (MDH, 2014).

Dioxins are generally non-volatile and not emitted from the waters of the Site; therefore, the inhalation exposure pathway is considered incomplete for human receptors.

Based on the Site conditions, accessibility, and current advisories discuss above, human health exposure pathways are considered incomplete.

The SSVs for dioxins, which are the most conservative available screening criteria and are intended to be protective of human health, are likely well below background levels and cleanup levels (CULs) similar to background levels should be used. The Midpoint SQT for dioxins is expected to be near the background concentration of dioxins within the upper SLR AOC; therefore, the Midpoint SQT will serve as the CUL for dioxins. Ongoing background concentration studies are currently being undertaken in the SLR. Should the results of these studies identify

dioxins background concentrations in sediment different from the Midpoint SQTs the CULs in this FFS will be revised.

1.4.4.2 Ecological Exposure Pathways

Contaminated sediments within the Site are located within the Potentially Bioactive Zone (PBAZ). The PBAZ is the area within the sediment where significant biological activity may be present. There is no definitive scientific consensus on the maximum depth to which flora and fauna penetrate sediment, but the MPCA's selection of an appropriate PBAZ thickness is based on a weight-of-available-evidence approach and professional opinion. Due to the large uncertainty in this type of analysis, the PBAZ incorporates an element of conservatism (i.e., greater depth) to provide an additional safety factor. Three designated PBAZ thicknesses are applicable in the habitat, water depth, and substrate types, as described below from the Draft Technical Memorandum, Remedial Action Objectives, Preliminary Remedial Goals, PBAZ Thicknesses, SR#276 – U.S. Steel Duluth Works Site. October 2015 (Bay West, 2015):

Backshore/Foreshore Habitat Zone (Shoreline, Riparian and Wet Transition Areas)

(Minimum PBAZ thickness = 1.2 meters [3.9 feet])

Applicable in:

- Shoreline/beach areas;
- Sediment flats that are exposed due to periodic low water levels or seiche;
- Open water/wet transition areas;
- Areas potentially available to deep burrowing mammals; and
- Areas potentially available for deep rooted herbaceous and/or woody plants.

Emergent Aquatic Vegetation Habitat Zone (off the Shoreline)

(Minimum PBAZ thickness = 1.0 meter [3.3 feet])

Applicable in:

- Emergent aquatic vegetation areas;
- Areas with potential for transitioning to emergent aquatic vegetation habitat (i.e., areas with substrates and water depths conducive to establishment of emergent vegetation now or in the future); and
- Areas potentially susceptible to deep burrowing amphibians, reptiles or crustaceans.

Submerged Aquatic Vegetation and Deep Water Habitat Zone

(Minimum PBAZ thickness = 0.5 meter [1.6 feet])

Applicable in:

- Areas that support submerged aquatic vegetation habitat with no potential to transition to emergent aquatic vegetation or wetland habitat;
- Areas with water depths too deep to support emergent vegetation but may support benthic organisms; and
- Areas with a substrate not conducive to deeply rooted aquatic vegetation, wetland herbaceous or woody vegetation, or deep burrowing mammals, amphibians, or crustaceans (i.e., areas armored for erosion control or areas with root barriers or other engineering controls).

The various habitat zones found within the Site, which correspond to estimated PBAZ thicknesses for each habitat, are presented in **Figure 6**. It can be seen from a comparison between **Figure 5** and **Figure 6** that the remedial footprint contains each of the habitat area types. Fish and other aquatic organisms accumulate some chemicals, which, based on the 2016 DGI fish tissue results, include dioxins from food and sediment that they ingest or through direct partitioning from water to biological tissues.

Complete ecological exposure pathways for the Site include the following:

- Exposure to ecological receptors through incidental ingestion and dermal contact with sediments; and
- Ingestion of biota that have consumed contaminated sediments.

Based on a comparison of the complete ecological exposure pathways and available analytical data summarized in **Section 1.4.3**, sediments with concentrations of COCs that exceed the Midpoint SQT value are considered a risk to the benthic community and the larger ecological environment, where they are found.

In summary, the analysis of the 2011 and 2014 sediment data and available exposure pathways indicated that COCs are present at the Site and exposure pathways are complete; therefore, a potential risk to both human and ecological health from contaminated sediments exists at the Site.

1.4.5 Conceptual Site Model

The development of a conceptual site model (CSM) allows data obtained during ongoing investigations to be integrated in an iterative approach that increases the understanding of the physical and environmental setting of the Site and the fate and transport of COCs. The CSM provides a baseline for consideration of how remedy alternatives could be implemented to protect human and environmental health at the Site. The CSM is illustrated in **Figure 7**.

Industrial and municipal sources upriver of the Site likely began contributing contaminants to the SLR by the early 1900s, as previously discussed in **Section 1.3**. Dioxins are known constituents of the historic discharges. These waste streams, specifically the paper manufacturing effluent discharge water, were removed from the SLR in 1979 when they were rerouted to WLSSD. Another potential route of entry of dioxins into the SLR is atmospheric deposition via incineration of dioxin/furan-containing boiler sludge (Schubauer-Berigan and Crane, 1996a).

Based on previous SLR and Site investigations, the current CSM indicates that the Site has retained significant levels of COCs and associated sediment that deposited at the Site from upstream sources. Industrial sources of COCs were supposedly reduced, if not eliminated, beginning in the 1970s with only ambient COC concentrations now entering the river and Site waters. High concentrations of dioxins within the upper interval within the eastern arm of the Site, as indicated by 2011, 2014, and 2016 data, suggest one of the following:

1. Deposition of clean sediments at the Site has not been significant since source inputs were removed in 1979;
2. Clean sediments have been deposited at the Site at thicknesses less than 0.15 meter, but very high concentrations of dioxins at the lower portion of the 0.0- to 0.15-meter interval resulted in exceedances of Midpoint SQT screening criteria in 2011, 2014, and 2016 samples; or
3. An ongoing source of dioxins contamination exists and has resulted in contaminated surface sediments within certain areas of the Site.

Receptors that are potentially exposed to COCs include the following ecological receptors:

- Emergent and submerged vegetation;
- Benthic and aquatic invertebrates;
- Mammals and birds consuming fish, benthic and aquatic invertebrates, and vegetation; and
- Undetermined receptors if future maintenance dredging is needed or other intrusive activities take place.

Reducing surface sediment concentrations or chemical bioavailability is the primary goal of sediment remediation processes. The deposition of cleaner sediment that buries and isolates COCs below the upper bioturbation layer reduces risk of chemical exposure to benthic receptors. No models have been developed for the Site to predict sediment deposition rates. Based on assumptions made about the hydrodynamic environment at the Site, overall sedimentation is likely minimal or occurs infrequently and only during high water events.

2.0 APPLICABLE OR RELEVANT AND APPROPRIATE REQUIREMENTS AND REMEDIAL ACTION OBJECTIVES

Remedial actions for releases and threatened releases of hazardous substances, pollutants, or contaminants must be selected and carried out in accordance with State and Federal requirements. These requirements are referred to as ARARs. RAOs specify COCs, media of concern, potential exposure pathways, and remediation goals. Initially, Site remediation goals for the COCs are developed based on readily available information such as chemical-specific ARARs or other reliable information. The Site RAOs are modified, as necessary, as more information becomes available during the FFS process.

This section presents the preliminary ARARs, RAOs, and COCs to be used in the development of this FFS. The final ARARs, RAOs, and COCs will be developed by the MPCA for the Site.

2.1 Applicable or Relevant and Appropriate Requirements

This preliminary ARAR section summarizes the MPCA, MDNR, and MDH ARARs, and to be considered (TBC) criteria for aquatic sediment associated with the Site. Local and Federal ARARs have also been included; however, the list may not include all applicable local and Federal ARARs.

The NCP (40 CFR 300.5) defines “applicable” requirements as: “those cleanup standards, standards of control, and other substantive requirements, criteria, or limitations promulgated under Federal environmental or State environmental or facility citing laws that specifically address a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstance found at a CERCLA [Comprehensive Environmental Response, Compensation, and Liability Act] site.” Only those promulgated state standards identified by a state in a timely manner that are substantive and equally or more stringent than Federal requirements may be applicable.

The NCP (40 CFR 300.5) further defines “relevant and appropriate” requirements as: “those cleanup standards, standards of control, and other substantive requirements, criteria, or limitations promulgated under Federal environmental or State environmental or facility citing laws that, while not “applicable” to a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstances at a CERCLA site, address problems or situations sufficiently similar to those encountered at the CERCLA site that their use is well suited to the particular site.” Like “applicable” requirements, the NCP also provides that only those promulgated state requirements that are identified in a timely manner and are more stringent than corresponding Federal requirements may be relevant and appropriate.

ARARs generally fall into one of the following three classifications:

- **Chemical-specific:** These ARARs are usually health- or risk-based numerical values or methodologies that, when applied to site-specific conditions, result in numerical values. These values establish an acceptable amount or concentration of a chemical that may be found in, or discharged to, the ambient environment. These requirements provide the basis for protective Site remediation levels for the COCs in the designated media.
- **Location-specific:** These ARARs generally restrict certain activities or limit concentrations of hazardous substances solely because of geographical or land use concerns. Requirements addressing wetlands, historic places, floodplains, or sensitive ecosystems and habitats are potential location-specific ARARs.
- **Action-specific:** These ARARs are restrictions on the conduct of certain activities or the operation of certain technologies at a particular site. Examples of action-specific ARARs would be regulations dictating the design, construction, and/or operating procedures for

dredging, on-site landfilling, or capping. Action-specific requirements do not themselves determine the cleanup alternative but define how the chosen cleanup alternative should be achieved.

In addition, criteria, advisories, guidance, and proposed standards developed by Federal and State environmental and public health agencies that are not legally enforceable, but contain helpful information, are collectively referred to as TBCs. TBCs can be helpful in carrying out selected remedies or in determining the level of protectiveness of selected remedies. TBCs are meant to complement the use of ARARs, not compete with or replace them. TBCs are included, where appropriate, in the chemical-, location-, and action-specific discussions.

Several Federal and State laws govern or provide the framework for remedial actions. Remedial actions must comply with substantive portions of these laws or acts, which were also reviewed during the ARAR development process. The following provides a summary of laws and acts that do not readily fall into one of the chemical-, location-, or action-specific classifications, but are applicable to the Site:

ARAR/TBC	Citation	Description/Potential Application
CERCLA	42 U.S. Code (USC) §9601 et seq.	Federal Superfund Law.
NCP	40 CFR Part 300	Provides organizational structure and procedures for preparing for and responding to discharges of oil and releases of hazardous substances, pollutants, and contaminants.
MERLA	Minn. Stat. §§115B.01 to 115B.20	State Superfund Law.
Water Pollution Control Act	Minn. Stat. chapter (ch.) 115	Administration and enforcement of all laws relating to the pollution of any waters of the state.
Duty to Notify and Avoid Water Pollution	Minn. Stat. §115.061	Requires notification and recovery of discharge pollutants to minimize or abate pollution of the waters of the state.
Pollution Control Agency	Minn. Stat. ch. 116	Provides organizational structure and procedures for responding to problems relating to water, air, and land pollution.
Water Law	Minn. Stat. ch. 103A, 103B, 103C, 103D, 103E; 103F, and 103G	Provides regulations pertaining to any waters of the state, including surface water, wetlands and groundwater.
Safe Drinking Water Act	42 USC §300f et seq.	Established to protect the quality of drinking water (above or underground).
Clean Water Act	33 USC §1251 et seq.	Establishes structure for regulating discharges of pollutants and regulating quality standards for surface waters.
Resource Conservation and Recovery Act (RCRA)	42 USC §6901 et seq.	Establishes RCRA Program and Regulations.
Clean Air Act	42 USC §7401 et seq.	Regulates air remissions from stationary and mobile sources.
Federal Energy Regulatory Commission (FERC)	FERC was established by congress through various laws.	An independent agency that regulates transmission and wholesale sale of electricity and natural gas in interstate commerce. FERC authorizes and regulates non-federal hydropower projects.

2.1.1 Chemical-Specific ARARs and TBCs

The COCs associated with the sediments include dioxins. The following are the chemical-specific ARARs and TBCs associated with the sediments and shall be used to develop site-specific CULs:

ARAR/TBC	Citation/Source	Description/Application
Sediment		
SSVs	MDH, 2013. Public Health Consultation, Updated Human Health Screening Values for SLR Sediments: U.S. Steel Site, April.	To be used as benchmark values for making comparisons to surficial sediment chemistry measurements.
SQTs	Guidance for the Use and Application of SQTs for the Protection of Sediment-dwelling Organisms in Minnesota.	To be used as benchmark values for making comparisons to surficial sediment chemistry measurements.
All Media		
Contaminated Sediments Remediation	Contaminated Sediments Remediation. http://www.itrcweb.org/contseds_remedy-selection/	Guidance to assist in selecting remedial technology most appropriate for a specific site.
Contaminated Sediment Remediation	Contaminated Sediment Remediation Guidance for Hazardous Waste Sites, USEPA, December 2005.	Guidance to assist in selecting remedial technology most appropriate for a specific site.
Contaminated Sediment Remediation	Use of Amendments for In Situ Remediation at Superfund Sediment Sites, USEPA, April 2013.	Guidance to assist in situ remediation.
Site screening guidelines	Working Draft Site Screening Evaluation Guidelines. MPCA Risk-Based Site Evaluation Manual (09/98).	Guidelines and criteria for screening human health and ecological risks.

Sediment

Human Health Risk

To achieve protection to human health and minimize direct and indirect exposure to COCs, Preliminary Sediment Remediation Goals were developed for use in this FFS. The SSVs are tools for screening contaminated sediments for potential impacts to human health; however, based on ongoing ambient concentration studies, the SSVs applicable to COCs for the Site likely approach, or are less than ambient concentrations in sediment. Further, the SSVs do not include RMEs specific to the Site and are not intended to be used as sediment cleanup values; therefore, SSVs will not be used to evaluate risk to human health due to COC contaminated sediment at the Site. For dioxins, the Midpoint SQT is expected to be within the range of ambient dioxins concentrations in the upper AOC. The Midpoint SQT will be used to identify, evaluate, and prioritize sediment-associated risk to human health.

Ecological Risk

SQT values were adopted for use in the SLR AOC to minimize exposure of the benthic organisms to contaminated sediments and movement of contaminants up the food chain. The MPCA does not have sediment quality standards. Instead, SQTs can be used in the SLR AOC and throughout the state as benchmark values for making comparisons to surficial sediment chemistry measurements as described in **Section 1.4.3.2**. For this FFS, the Midpoint SQT was used to identify, evaluate, and prioritize sediment-associated risk to ecological receptors.

All Media

This guidance document assists in selecting remedial technology most appropriate for a specific site based on contaminated sediment and site specific characteristics (http://www.itrcweb.org/contseds_remedy-selection/).

The USEPA document, Contaminated Sediment Remediation Guidance for Hazardous Waste Sites, presents remedial options available for contaminated sediments discussing advantages and limitations associated with the options.

The USEPA document, Use of Amendments for In Situ Remediation at Superfund Sediment Sites, presents remedial options using amendments available for contaminated sediments discussing advantages and limitations associated with the options.

The MPCA Site Screening and Evaluation Document presents an overall process for conducting a Tier 1 evaluation of the various exposure pathways at a site. The screening criteria worksheet can be found at MPCA website (https://www.pca.state.mn.us/waste/risk-based-site-evaluation-guidance).

2.1.2 Location-Specific ARARs and TBCs

The Location-Specific ARARs and TBCs for the Site are as follows:

ARAR/TBC	Citation/Source	Description/Application
Waters of the State and Groundwater Protection	Minn. Stat. 103G and 103H	Groundwater protection, non-degradation, and best management practices.
Floodplain Management and Wetlands Protection	40 CFR Part 6, Appendix A, §6.a.(1)	Requires agencies to evaluate potential effects of actions in a floodplain to avoid adverse impacts.
Shoreland and Floodplain Management	Minn. Rules ch. 6120	Conserves economic and natural environmental values (MDNR).
Carlton County Land Use Ordinances	Carlton County Zoning Ordinances #27	Floodplain management, Manages on-site waste disposal and other site activities.
Shoreland Management	Carlton County Zoning Ordinance #27	Carlton County requires a permit for any excavation or grading within 1000 feet of lake or 300 feet of a stream/river.
Endangered Species Act	16 USC §1531 et seq. 50 CFR §17.11-12	Conservation of threatened and endangered plants and animals and their habitats.
Endangered, Threatened, Special Concern Species	Minn. Rules ch. 6134 Minn. Statute, §84.0895	Protection of endangered, threatened, special concern species (MDNR).
Migratory Bird Treaty Act	16 USC Chapter 7, Subchapter II §§703 and 712.2	Protects migratory birds and their ecosystems.
MDH Advisory for SLR	MDH	Provides fish consumption advisories.

The Site is located within the Lake Superior Drainage Basin. Surface water quality standards and provisions for Class 2B and 3B waters apply. In addition, USEPA and the Great Lakes states

agreed in 1995 to a comprehensive plan to restore the health of the Great Lakes. The Final Water Quality Guidance for the Great Lakes System, also known as the Great Lakes Initiative (GLI), includes criteria for states to use when setting water quality standards for 29 pollutants, including bioaccumulative COCs, and prohibits the use of mixing zones for these toxic chemicals. Because the surface water at the Site is within the drainage basin of Lake Superior, the ARARs specified in the GLI, Minn. Rules ch. 7052 are applicable to the Site. Requirements of the Great Lakes Water Quality Agreement of 2012 apply to the Site. In addition, the surface waters adjacent to the Site are identified as an Outstanding International Resource Water (OIRW). The objective for OIRW is to maintain water quality at existing conditions when the quality is better than the water quality standards. Generally, OIRWs are considered surface water quality standards applicable to the SLR for Class 2B and OIRWs, as set forth in Minn. Rules, ch. 7050 and 7052, and to the additional surface water quality standards for the SLR, as set forth in Minn. Rules ch. 7065. The OIRW was established after the Record of Decision was issued.

As stated in Minn. Rules ch. 7050.0210 Subp. 2:

> *Nuisance conditions prohibited. No sewage, industrial waste, or other wastes shall be discharged from either point or nonpoint sources into any waters of the state so as to cause any nuisance conditions, such as the presence of significant amounts of floating solids, scum, visible oil film, excessive suspended solids, material discoloration, obnoxious odors, gas ebullition, deleterious sludge deposits, undesirable slimes or fungus growths, aquatic habitat degradation, excessive growths of aquatic plants, or other offensive or harmful effects.*

Title 40 CFR Part 6, Appendix A, Section 6 Requirements: Requires Federal agencies to evaluate the potential effects of actions taken within a floodplain to avoid adversely impacting floodplains wherever possible.

Title 40 CFR Part 6, Appendix A, Section 6.a.(1) Floodplain/Wetlands Determination: Before undertaking an Agency action, each program office must determine whether or not the action will be located in or affect a floodplain or wetlands. The Agency shall utilize maps prepared by the Federal Insurance Administration of the Federal Emergency Management Agency (Flood Insurance Rate Maps or Flood Hazard Boundary Maps), Fish and Wildlife Service (National Wetlands Inventory Maps), and other appropriate agencies to determine whether a proposed action is located in or will likely affect a floodplain or wetlands. If there is no floodplain/wetlands impact identified, the action may proceed without further consideration of the remaining procedures set in this section. If floodplain/wetlands impact is identified, this section presents procedures that must be taken.

Shoreland and Floodplain Management (Minn. Rules ch. 6120): Provides standards and criteria intended to preserve and enhance the quality of surface waters, conserve the economic and natural environmental values of shorelands, and provide for the wise use of water and related land resources of the state. Carlton County Zoning Ordinances, ch. 1003, establish additional floodplain management and manage site activities such as on-site waste disposal.

Shoreland Management Permit (Carlton County Zoning Ordinance #27), as defined by Carlton County: Requires a permit for any excavation or grading above the Ordinary High Water Mark within 300 feet of a river. Each alternative will involve some of these activities. The substantive requirements of this permit are found in the ordinance and may govern removal of natural vegetation, grading and filling, placement of roads, sewage and waste disposal, and setbacks.

The Endangered Species Act (16 U.S.C.A. §1531 et seq.) and the Minnesota Endangered, Threatened, Special Concern Species Act (Minn. Rules ch. 6134): Protect threatened and endangered plants and animals and their habitats.

Title 16 USC Chapter 7, Subchapter II §§703 and 712.2., The Migratory Bird Treaty Act: Protects migratory birds and their ecosystems by specifying the taking, killing, or possessing migratory birds unlawful. Public Law 95-616, an amendment to this act, provides measures to protect identified ecosystems of special importance to migratory birds such as bald eagles against pollution, detrimental alterations, and other environmental degradations.

The MDH has established various fish consumption advisories for the SLR due to the presence of perfluorochemicals, PCBs, and mercury in water and sediments; however, there is no fish consumption advisory for dioxins for the Site.

2.1.3 Action-Specific ARARs and TBCs

The following summarizes the Action-Specific ARARs for the Site. In addition, Occupational Safety and Health Standards (Minn. Rules ch. 5205) for worker health, safety, and training are applicable to remedial actions performed at the Site.

ARAR/TBC	Citation/Source	Description/Application
Waters of the State (both surface and underground)	Minn. Rules ch. 7050 and 7052	Surface water quality during remedy construction.
Wetland Conservation Act (WCA)	Minn. Stat. §§103G.221-.2373	Protection of wetlands.
Wetlands Conservation	Minn. Rules 8420	Protection of wetlands, wetland functions for determining public values.
Floodplain Management Order	Executive Order 11988 and 40 CFR Part 6, Appendix A,	Regulates remedial action implementation in floodplains.
Section 404 Permit and Section 401 Certification (Clean Water Act)	33 CFR pts 320 and 323; 33 USC §1341	Applies to discharge of dredged or fill material into waters of the U.S.
National Pollutant Discharge Elimination System (NPDES)/ State Disposal System (SDS) permits	Clean Water Act 33 USC §1342	Surface water quality requirements for discharges of pollutants to waters of the state.
Section 10 (Rivers and Harbors Act of 1899)	33 USC 403	Applies to activities that will obstruct or alter any navigable water of the U.S.
Work in Public Waters	Minn. Stat. §103G.245	Permit requirements applicable to work in public waters that will change or diminish its course, current, or cross-section.
Public Water Resources	Minn. Rules ch. 6115	Water appropriation permitting, standards and criteria for alterations to structure of public water (MDNR).
Minnesota SQTs	Guidance for the Use and Application of SQTs for the Protection of Sediment-dwelling Organisms in Minnesota, MPCA Document Number: tdr-gl-04	Establishes procedures for PBAZ caps and covers.
WLSSD	WLSSD Industrial Pre-Treatment Ordinance	Requirements for any dredge water discharged into public sanitary sewers.

ARAR/TBC	Citation/Source	Description/Application
Construction and Use of Public Sewers	Minn. Rules ch. 4715	Governs the use of sewers and public water systems if any dredge water is disposed of in public sewers.
MDNR Invasive Species Management	Minn. Statutes 84D.02	Requirements for sediment transportation if invasive species are present.
Solid Waste	Minn. Rules ch. 7035	Requirements and standards for solid waste facilities.
Hazardous Waste	Minn. Rules ch. 7045	Hazardous waste listing, and generator, transport, and facility standards.
Air Pollution Emissions and Abatement	Minn. Stat. §116.061	Duty to notify and abate excessive or abnormal unpermitted air emissions.
Ambient Air Quality Standards	Minn. Rules ch. 7009	Provides air quality standards.
Preventing Particulate Matter From Becoming Airborne and Emission Standards	Minn. Rule pts. 7011.0150 and 7011.8010	Provides measures to control dust and emission standards for hazardous air pollutants.
Noise Pollution Control	Minn. Rules ch. 7030	Noise standards applicable to remedy construction.

Water Quality

If any activity associated with the remedial actions results in an unregulated release, in accordance with the Water Pollution Control Act and Minn. Stat. 115.061, Duty to Notify, a notification and recovery of any pollutants discharged to minimize or abate pollution of the waters of the state is required.

In accordance with Minn. Rules ch. 7050, surface water quality standards for the maintenance and preservation of surface water quality during remedy construction, including discharges from treatment/work and stormwater runoff zones, shall be based on surface water quality standards that currently apply to Class 2B and OIRWs, as set forth in Minn. Rules, ch. 7050 and 7052, and to the additional surface water quality standards for the SLR set forth in Minn. Rules ch. 7065. Therefore, if water is discharged directly to the waters on or adjacent to the Site, it shall be treated to a level that meets applicable surface water discharge standards. Groundwater non-degradation and standards for the protection of groundwater during remedy construction are presented in Minn. Rules 7060.

During remediation, the MPCA would consider the areas in which work is performed as “treatment/work zones,” to which the surface water quality standards normally applicable to the SLR would temporarily not apply. These treatment/work zones would be physically separated from adjacent waters through the use of engineering controls such as single or multiple silt curtains, inflatable dams, sheet piling, or other measures. During construction of the remedy, any discharges occurring within those controlled treatment/work zones, such as the discharge of capping material during capping operations, the release of contaminants during dredging operations, or runoff from activities on shore, would not be subject to water quality standards. Rather, water quality standards would apply outside of the treatment/work zone, beyond the outermost engineering control structure where the water from the treatment/work zone is discharged. Other discharges occurring during remedy construction that are not included in a treatment/work zone, including discharges of treated dredge water, and discharges of stormwater runoff from shoreland modifications outside of the treatment/work zones, would also be subject to regulation.

If water is discharged, it would be treated to a level that meets applicable surface water discharge standards. The MPCA water quality standards may apply to these discharges. Final standards would be determined by the MPCA prior to implementation of the remedial actions. In the event that a standard is exceeded, further management practices would likely be required during remedy construction to reduce the amount of suspended contaminants escaping the treatment/work zone.

Wetlands and Shoreland and Floodplain Management

In accordance with Minn. Rules ch. 7050, wetlands at the Site are classified as freshwater emergent wetlands, classification code PEM1F. Wetland areas are located at the far northern portion of the Site and appear to have a combined size of approximately 1 acre. The remainder of the Site is classified as lake, classification code L1UBHh. The size of the lake area is approximately 43 acres (data retrieved from http://www.fws.gov/wetlands/Data/Mapper.html).

In accordance with Minn. Rules ch. 8420, compliance with wetland ARARs will involve consultation with the MDNR to determine the category of wetlands present at the Site and any avoidance, mitigation, and replacement that may be necessary. Water quality standards for the maintenance and preservation of surface water quality during remedy construction including discharges from treatment/work and stormwater runoff zones shall be based on surface water quality standards that currently apply to Class 2B and 3B waters and shall comply with Minn. Stat. §§103G.221-.2373. Standards and specifications applicable to shoreland and floodplain management can be found in Executive Order 11988 and 40 CFR Part 6, Appendix A, Minn. Rules ch. 6120.

Minn. Stat. §103G.222 provides that a wetland replacement plan must be approved by the Local Governmental Unit before any WCA wetlands may be drained or filled, unless draining or filling falls within the “De Minimis” exemption or another exemption of Minn. Stat. §103G.2241. WCA wetlands are those wetlands that are not public water wetlands regulated by the MDNR and U.S. Army Corps of Engineers (USACE). WCA wetlands would be located above the Ordinary High-Water Mark.

Carlton County provides additional guidance regarding WCA requirements for the Site at the following website:

http://www.co.carlton.mn.us/vertical/Sites/%7B315ADE76-21A3-4241-B977-F94AEE8A7F04%7D/uploads/Carlton_County_and_the_Wetland_Conservation_Act.pdf

Permits and Certifications

Possible permits for cleanup activities include the following:

Section 404 Permit (Clean Water Act): Required for discharge of dredged or fill material into waters of the U.S. The substantive requirements of this permit shall be met for alternatives that dredge or fill waters of the state. USACE evaluates applications for Section 404 permits. Substantive requirements that may be incorporated within a Section 404 permit for off-site activities can be found in 33 CFR Parts 320 and 323.

Section 401 Certification: The Clean Water Act, 33 USC §1341, requires that any application for a Federal permit that may result in a discharge to a navigable water must be accompanied by a certification from the affected state indicating that the discharge will comply with all applicable water quality standards and effluent limitations of the Act. Thus, a Section 401 certification or a 401 certification waiver for remedial action at the Site would be necessary before the USACE may issue a Section 404 permit, and a certification may be necessary before the USACE may issue a Section 10 permit if that permit authorizes a “discharge.”

National Pollutant Discharge Elimination System (Clean Water Act 33 USC §1342): Discharges of pollutants to waters of the state associated with construction of the selected remedy would be subject to the requirements applicable to a NPDES permit. Discharges could include the discharge of amendment and/or capping material, the discharge of contaminants released and suspended by dredging operations, the discharge of treated dredge water during dredging operations, and the discharge of stormwater runoff from shoreland modifications. These types of discharges would be subject to the same regulatory standards and controls that would apply under an MPCA permit. In addition, NPDES General Permit number MNG990000 has been required for managing dredged materials; however, this permit has expired and has not been renewed. According to Managing Dredged Materials in the State of Minnesota (MPCA, 2009), an individual NPDES/State Disposal System (SDS) Dredge Materials Management permit may be required. A NPDES Construction Permit and a Stormwater Pollution Prevention Plan are required by the MPCA if more than one acre of land is disturbed by excavation activities.

Section 10 of the Rivers and Harbors Act of 1899 (33 USC 403): A Section 10 permit is required from the USACE for any construction in or over any navigable water, or the excavation or discharge of material into such water, or the accomplishment of any other work affecting the course, location, condition, or capacity of such waters. The substantive requirements that may be incorporated within a Section 10 permit can be found in 33 CFR Parts 320 and 322.

Work in Public Waters (Minn. Stat. §103G.245): A permit from the MDNR is necessary for any work in public waters that will change or diminish its course, current, or cross-section. If an alternative under consideration involves dredging or capping, a public waters permit from the MDNR may be required. The substantive requirements that the MDNR may incorporate within its public waters permit are codified in statute and at Minn. Rules, ch. 6115. These requirements include compensation or mitigation for the detrimental aspects of any major change in the resource. The MDNR permits may require restoration of bathymetry (water depth) and habitat substrate (bottom) as part of the public waters permit. The MDNR would set the specific cover depth and composition requirements.

Additionally, if capping of contaminated sediments is conducted, requirements would include specifications for cap construction. In situ caps constructed for the containment of contaminated sediment must contain an isolation zone (IZ) and a PBAZ. The IZ is the portion of the cap that is applied directly over the contaminated sediments and is designed to isolate and attenuate the Site contaminants that could potentially be transported upward into the PBAZ at concentrations above the CULs by diffusion or advection transport mechanisms. The PBAZ is the area within the cap above the IZ where significant biological activity may potentially be present. The thickness and material specifications for the IZ and PBAZ should be determined based on pore water transport and attenuation modeling.

Air Emissions and Waste Management Permits: In accordance with Minn. Stat. §116.081, a permit is required for the construction, installation or operation of an emission facility, air contaminant treatment facility, treatment facility, potential air contaminant storage facility, storage facility, or system or facility related to the collection, transportation, storage, processing, or disposal of waste, or any part thereof, unless otherwise exempted by any agency rule now in force or hereinafter adopted, until plans have been submitted to the agency, and a written permit granted by the agency.

On-Site Disposal: The placement of dredged sediment into an on-site confined aquatic disposal (CAD) area and any subsequent seepage from the CAD, if implemented, would be regulated by the MPCA under the requirements applicable to an SDS permit. The legal requirements for an SDS are found in Minn. Stat. §115.07, Minn. Rules, Parts 7065.0100 to 7065.0160 and in other MPCA water quality rules including Minn. Rules ch. 7050 and 7052.

Discharge into Sewers: A permit from the WLSSD will be necessary if any dredge water is discharged into the public sewers. Pretreatment standards that would likely apply can be found at: http://wlssd.com/wp-content/uploads/2014/12/WLSSDPretreatmentOrdinance.pdf.

The permit will also include requirements to ensure there will be no detrimental effects to their bio-solids program. A WLSSD permit would also represent compliance with Minn. Rule, Part 4715.1600 and the MPCA water rules governing indirect discharges.

Invasive Species: A prohibited/regulated invasive species permit will be required to transport sediment to a landfill, if invasive species are present near the proposed work area.

CERCLA provides for waiving of necessary permits for on-site work, provided the work is conducted in compliance with the substantial conditions of such permits. Although the permits themselves may not be required on CERLCA Sites, compliance with the substantial conditions of these identified permits shall be met.

Construction and Use of Public Sewers

Minn. Rules ch. 4715 governing the use of sewers and public water systems would apply if any water associated with remedial activities is disposed of in public sewers.

Waste Management

Solid and hazardous waste management requirements and standards can be found in Minn. Rules ch. 7035 and 7045, respectively. USEPA guidance has consistently stated that Superfund remedies involving movement of contaminated material within the area of a Site where such material is already located (sometimes referred to as an AOC) do not create a “waste” that is subject to Resource Conservation and Recovery Act (RCRA) (42 USC §6901 et seq.) or other waste management requirements. Remedy alternatives that require contaminated materials to be moved to an off-site land disposal site is considered to generate waste that must be managed under applicable waste management requirements.

St. Louis County Zoning Ordinances, ch. 1003, establish additional floodplain management and manage site activities such as on-site waste disposal.

Ambient Air Quality Standards

Air quality standards applicable to releases into the air from cleanup activities include Min. Stat. 116.061, Air Pollution Emissions and Abatement. During remedy construction, activities such as transportation, storage and placement of amendment and/or capping material may result in particulate matter becoming airborne. Minn. Rules ch. 7009 establishes ambient air quality standards for criteria pollutants regulated under the Clean Air Act. Compliance points shall be selected in accordance with Minn. Rules ch. 7009. The ambient air quality standards for particulate matter that apply to remedial actions are found at:

https://www.revisor.mn.gov/rules/?id=7009.0080

Control of the generation of airborne particulate matter during remedy construction is regulated in Minn. Rule Part 7011.0150, *Preventing Particulate Matter from Becoming Airborne*, which includes measures to control dust that may be generated during remedy construction activities such as transportation, storage, and placement of capping material, which shall be addressed in the remedial design plan. Minn. Rules Part 7011.8010, Site Remediation, incorporates the National Emission Standards for Hazardous Air Pollutants applicable during Site remediation activities.

Noise Pollution Control

Minn. Rules ch. 7030 establishes noise standards for various land uses. Compliance points will be selected in accordance with Minn. Rules ch. 7030. The noise standards that will apply to the selected remedial action can be found at:

https://www.revisor.leg.state.mn.us/rules/?id=7030.0040

2.1.4 Other Considerations

Other considerations under MERLA set forth the regulatory requirements, RAOs and CULs that must be met by a remedy to meet the legal standard for a remedy under MERLA and the threshold criterion for protection of public health and welfare and the environment. A remedy, as defined under MERLA, must also include any monitoring, maintenance and institutional controls (ICs) and other measures that MPCA determines are reasonably necessary to ensure the protectiveness of the selected remedy over the long term.

It is particularly important to consider the requirements for long-term assurance of protectiveness where the remedy alternatives involve the use of capping or containment to manage contaminated media within the Site. Some requirements may also be necessary to ensure long-term protectiveness of alternatives that involve excavation or dredging and off-site disposal of contaminated soil or sediment.

In addition, MERLA requires the MPCA to consider the planned use of the property where the release of contaminants is located when determining the appropriate standards to be achieved by a remedy.

Long-Term Assurance of Protectiveness

MERLA requires that a remedy include measures that are reasonably required to ensure the ongoing protectiveness of a remedy once the components of the remedy have been constructed and entered their operational phase. Such measures may include, but are not limited to, ICs and monitoring and maintenance requirements. This section discusses the measures that MPCA determines are reasonably necessary to ensure long-term protectiveness.

Institutional Controls

ICs are legally enforceable restrictions, conditions or controls on the use of property, groundwater or surface water at a property that are reasonably required to ensure the protectiveness of a remedy or other response actions taken at the Site. Areas of the Site where contaminated media remains in place after remedial construction will be subject to ICs (such as easements and restrictive covenants) that are legally binding on current and future owners of the property to ensure ongoing protection from disturbance of or exposure to the contamination. Restrictions on use may also be required for areas of the Site where contaminated media are treated and/or removed and where some residual contamination may remain.

Minn. Stat. §115B.16, subd. 2, requires an Affidavit Concerning Real Property Contaminated with Hazardous Substances to be recorded with the St. Louis County recorder by the owner of the property. The Uniform Environmental Covenants Act (UECA) and the authority for requiring environmental covenants can be found in Minn. Stat. ch. 114E. This statute requires MPCA approval of environmental covenants (which include restrictive covenants and access) when there is an environmental response project (which includes superfund cleanups) is overseen by the MPCA. Because the Site is not platted, the UECA may not apply and other ICs such as a City Ordinance may be required to prevent anchoring, fishing, dredging, and other activities that may disturb a cap or contaminated sediments left in place.

Long-Term Operation and Maintenance, Monitoring, and Contingency Action

On-site containment facilities and capping of impacted media (sediment) or any other alternative that may leave impacted media on-site will require post-construction monitoring, operation and maintenance (O&M), and contingency action plan to ensure that ARARs, RAOs, and CULs that apply to the alternative are fully achieved and maintained over time.

General details of the post-construction monitoring, O&M, and contingency action plan requirements would be set forth in the FFS, along with an estimate of the cost to carry out each activity.

Planned Use of Property

In a provision entitled "Cleanup Standards" (Minn. Stat. §115B.17, subd. 2a), MERLA provides that when MPCA determines the standards to be achieved by response actions to protect public health and welfare and the environment from a release of hazardous substances, the agency must consider the planned use of the property where the release is located. The purpose of this provision of MERLA is to allow the MPCA to select cleanup standards that provide a level of protection that is compatible with the uses of the Site property that can be reasonably foreseen.

The specific properties directly affected by the remedies are currently part of treatment or containment facilities considered to be commercial/industrial land use (the Site currently serves to provide generation of hydroelectric power and is anticipated to maintain its current use into the foreseeable future). In addition, impacted areas include wetlands/semi-aquatic and aquatic areas and associated habitat. The cleanup standards must provide protection of public health and welfare and the environment that is consistent with any planned or potential future uses of the Site, including natural resource and habitat restoration and maintenance of the current Site use of hydroelectric power generation. These cleanup standards are also compatible with the use of the adjacent lands for residential, recreational, habitat restoration, or commercial and industrial use.

2.2 Remedial Action Objectives

The RAOs developed by the MPCA for the Site are:

- Minimize or remove exposure to sediment contaminants that bioaccumulate in the food chain.
- Minimize or remove exposure of the benthic organisms to contaminated sediments above sediment cleanup goals.
- Maintain current reservoir operating capacity and functionality.

The following subsection presents preliminary sediment CULs developed to achieve these RAOs.

2.2.1 Preliminary Sediment Cleanup Levels

The remedy should meet the Preliminary Sediment CULs to minimize exposure of benthic organisms to contaminated sediments, and to stop movement of contaminants up the food chain. The Midpoint SQT for dioxins is expected to be near the background concentration of dioxins within the upper SLR AOC; therefore, the Midpoint SQT will serve as the preliminary CUL for dioxins. Ongoing background concentration studies are currently being undertaken in the SLR, should the results of these studies identify dioxins background concentrations in sediment different from the Midpoint SQTs the CULs in this FFS will be revised. The following table presents the CULs for the COCs identified in **Section 1.4.3.4**.

Contaminant	Units	Cleanup Level	Maximum Concentration Detected	Mean Concentration
Dioxins	ng TEQ/kg	11.2	392.7	32.58

ng TEQ/kg – nanograms toxic equivalency per kilogram

3.0 DEVELOPMENT AND SCREENING ALTERNATIVES

3.1 Remedial Technology Identification and Screening Process

Potential technologies for addressing conditions at the Site were identified through conversations with the MPCA as well as guidance developed for the remediation of contaminated sediment sites (USEPA, 2005; Interstate Technology and Regulatory Council [ITRC], 2014). Historical sample data and the development of the CSM were used to identify feasible technologies for the Site.

A qualitative approach was used to screen technologies using a three-part ranking system where each technology was evaluated on effectiveness, implementability, and relative cost:

- Effectiveness was evaluated by the predicted ability of the technology under consideration to ensure long-term protection of human health and the environment while minimizing short-term impacts during implementation, as well as the technology's ability to meet RAOs.
- Implementability was evaluated by considering the technical and administrative feasibility of the technology. Technical feasibility includes the ability to achieve RAOs and the avoidance of creating additional risk during implementation, including the degree of disruption in the project area. Administrative feasibility includes the consideration of permits required for technology implementation, availability of disposal facilities and equipment necessary for the technology, and coordination with applicable agencies and stakeholders.
- Relative costs used for technology screening were based on engineering judgment, rather than detailed estimates. The cost evaluation considered direct and indirect expenses such as costs for dredging and capping, transport and disposal of sediment, and monitoring and evaluation periods.

Table 3 presents a summary of the technology screening results. The following sections describe the technologies that were screened using the three-part ranking system.

3.1.1 Institutional Controls

ICs are legally enforceable restrictions, conditions, or controls on the use of property, groundwater, or surface water at a contaminated site that are reasonably required to ensure the protectiveness of a remedy or other response actions taken at the Site. If contaminated sediments remain in place after remedial actions are taken, the Site would be subject to ICs (such as easements and restrictive covenants) that are legally binding on current and future owners of the property to ensure ongoing protection from disturbance of or exposure to the contamination. Most remedial alternatives include ICs until long-term monitoring (LTM) indicates that risk reduction has been achieved and the RAOs have been met (ITRC, 2014). The following information obtained from USEPA sediment remediation guidance (USEPA, 2005) details ICs likely appropriate for use at the Site.

Fish consumption advisories are informational devices that are frequently already in place and incorporated into sediment site remedies. Commercial fishing bans are government controls that ban commercial fishing for specific species or sizes of fish or shellfish. Usually, state departments of health are the governmental entities that establish these advisories and bans. An advisory usually consists of informing the public that they should not consume fish from an area or consume no more than a specified number of fish meals over a specific period of time from a particular area. Sensitive sub-populations or subsistence fishers may be subject to more stringent advisories. Advisories can be publicized through signs at popular fishing locations, pamphlets, or

other educational outreach materials and programs. Consumption advisories are not enforceable controls and their effectiveness can be extremely variable (USEPA, 2005).

Waterway use restrictions may be necessary to ensure the integrity of the alternative for any alternative where subsurface contamination remains in place (e.g., capping, monitored natural recovery [MNR], or an in water confined disposal site). Examples include restricting boat traffic in an area to establish a no-wake zone or prohibiting anchoring of vessels. In considering boating restrictions, it is important to determine who can enforce the restrictions, and under what authority and how effective such enforcement has been in the past. In addition, a restriction on easements for installing utilities, such as fiber optic cables, can be an important mechanism to help ensure the overall protectiveness of a remedy (USEPA, 2005).

It may be necessary to work with private parties, state land management agencies, or local governments to implement use restrictions on nearshore areas and adjacent upland properties where contamination remains in place. For example, construction of boat ramps, retaining walls, or marina development can expose subsurface contamination and compromise the long-term effectiveness of a remedy. Where contaminated sediment exceeding CULs is identified in proximity to utility crossings or other infrastructure and temporary or permanent relocation of utilities in support of a dredging remedy may not be feasible or practical, capping may be desirable even though temporary cap disruption may be necessary periodically (USEPA, 2005).

3.1.2 Monitoring

Monitoring is the collection and analysis of data (chemical, physical, and/or biological) over a sufficient period of time and frequency to determine the status and/or trend in one or more environmental parameters or characteristics. Monitoring should not produce a “snapshot in time” measurement, but rather should involve repeated sampling over time in order to define the trends in the parameters of interest relative to clearly defined management objectives. Monitoring is recommended for all types of sediment remedies both during and after remedial action and can be classified as construction monitoring and performance monitoring (also referred to as LTM), respectively. Monitoring should be conducted for a variety of reasons, including: 1) to assess compliance with design and performance standards; 2) to assess short-term remedy performance and effectiveness in meeting sediment CULs; and/or 3) to evaluate long-term remedy effectiveness in achieving RAOs and in reducing human health and/or environmental risk. In addition, monitoring data are usually needed to complete the five-year review process where a review is conducted.

Monitoring activities applicable to the Site could include one or more of the following based on the selected remedy:

- Collection of sediment chemical data to ensure that CULs have been achieved (due to dredging, in situ treatments, or degradation);
- Measurements of cover/cap thicknesses to ensure continued isolation of contaminants;
- Measurement of COC concentrations in cover/cap material to ensure that contaminants are not migrating into or through the cover/cap; and
- Measurement of toxicity to and bioaccumulation of COCs within aquatic organisms such as benthics and fish in order to evaluate reduction trends.

Construction monitoring may also be performed to ensure that contamination or nuisance materials are not released during construction activities. Construction monitoring activities applicable to the Site include one or more of the following:

- Turbidity monitoring to ensure that the off-site release of suspended sediments containing COCs is mitigated during dredging and/or cover/cap placement;
- Air monitoring to ensure that the off-site release of nuisance and/or contaminated dusts is mitigated during construction activities such as the mixing of sediments and amendment materials, hauling over dirt or gravel roadways, and excavation or other intrusive Site work;
- Periodic sampling of treated dredge contact water to mitigate contaminant inputs to water bodies or local sewage systems and to ensure that treated water meets permit or municipality requirements;
- Periodic sampling of dredged materials to ensure that landfill requirements for acceptance are achieved;
- Periodic sampling of imported materials (e.g., cover/cap materials, shoreline restoration materials, etc.) to mitigate impacts to water bodies or upland areas as a result of placement; and
- Pre- and post-construction soil sampling to access impacts of construction activities on lands used during the construction phase.

Both construction and performance monitoring (referred to as LTM) are incorporated into each of the remedial alternatives developed for this FFS.

3.1.3 Monitored Natural Recovery

MNR is defined by the National Research Council as a remediation practice that relies on natural processes to protect the environment and receptors from unacceptable exposures to contaminants. This remedial approach depends on natural processes to decrease chemical contaminants in sediment to acceptable levels within a reasonable time frame. With MNR, contaminated sediments are left in place and monitored for ongoing physical, chemical, and biological processes that transform, immobilize, isolate, or remove contaminants until they no longer pose a risk to receptors. Natural processes that contribute to MNR may include sediment burial, sediment erosion or dispersion, and contaminant sequestration or degradation (for example, precipitation, adsorption, or transformation). These natural processes can reduce exposure to receptors (and thus reduce risk) and contribute to the recovery of the aquatic habitat and the ecological resources that it supports. MNR can be used alone or in combination with active remediation technologies to meet RAOs (ITRC, 2014).

3.1.4 Enhanced Monitored Natural Recovery

Enhanced monitored natural recovery (EMNR) with Thin-Layer Amended Cover relies on the same natural processes as MNR to decrease chemical contaminants in sediment but includes the application of material or amendments to enhance these natural recovery processes. EMNR can use several technologies including, but not limited to, thin-layer capping and introduction of adsorptive or reactive amendments such as activated carbon. Thin-layer caps (typically less than 1 foot) are often applied as part of an EMNR approach. For the purposes of this FFS, a thin-layer consisting of clean sand and/or sand blended with activated carbon (AC) with a thickness of 1 foot was evaluated; however, the thickness of the cover and amendment materials will be evaluated further, should it be selected as a remedial alternative. These caps enhance ongoing natural recovery processes, while minimizing effects on the aquatic environment. Thin-layer caps are not intended to completely isolate the affected sediment, as in a conventional isolation

capping remedy. This layer also accelerates the process of physical isolation, which continues over time by natural sediment deposition (ITRC, 2014).

Benchscale treatability testing was completed on sediments collected from the Site to evaluate the effectiveness of different AC amendments and doses to reduce the bioavailability of dioxins/furans in Site sediments (**Appendix C**). Two AC particle size ranges were evaluated—a silt-sized powdered activated carbon (PAC) and a fine sand-sized granular activated carbon (GAC). The two different types of AC amendments (PAC at 2% and 4% dose, and GAC at 4% dose) were mixed directly into Site sediments, and continuous agitation was applied to accelerate the uptake kinetics of dioxins by the AC. The results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments. Extrapolating the results of this study to assess the short- and long-term effectiveness of different AC amendment application methods requires additional calculations to be conducted in a follow-on engineering phase. Application methods will be retained for further engineering and cost evaluations.

3.1.5 Capping

Capping is the process of placing a clean layer of sand, sediments or other material over contaminated sediments in order to mitigate risk posed by those sediments. The cap may also include geotextiles to aid in layer separation or geotechnical stability, amendments to enhance protectiveness, or additional layers to armor and maintain its integrity or enhance its habitat characteristics.

When amendments (such as AC) are mixed directly into sediments, the resulting remedy is termed “in situ treatment.” When these amendments are added to cap material, the remedy is called an “amended cap,” and the amendments enhance the performance of the cap material. The same amendment used in the same proportions is generally more effective at isolating contaminants when used in a cap than when placed directly into sediments. The amended cap provides the benefits of capping in addition to the benefits of the treatment amendment (ITRC, 2014).

3.1.6 Dredging and Excavation

Dredging consists of the removal of contaminated sediment from water bodies in order to reduce risks to human health and the environment. Removal is particularly effective for source control (mass removal of hot spots) but potentially less effective for overall risk reduction because of resuspension and residual contamination. The three methods of contaminated sediment removal are mechanical dredging, hydraulic dredging, and excavation. As with any type of removal operation, additional technologies are required to appropriately handle the removed sediment. Dredged material handling technologies may involve transport, dewatering, treatment, and or disposal of sediment (ITRC, 2014). Mechanical dredging, hydraulic dredging, and excavation were screened independently in this evaluation.

After removal, the contaminated sediment can be treated or disposed of in a controlled setting, such as an off-site landfill or other treatment, storage, and disposal facility, an on-site aquatic or terrestrial confined disposal facility (CDF), or a facility that converts the sediment to a reusable product. Disposal methods were evaluated independently from dredging and excavation and are described further in **Section 3.1.8**.

3.1.7 Dewatering

Dewatering may be necessary to prepare dredged materials for disposal. Dewatering reduces the water content and hence the volume and weight of the disposed sediment. If the material is to be reused or further treated, dewatering also leads to reduced transportation cost and improves handling properties. The nature and extent of dewatering needed depends on the sediment characteristics and the type of dredging, transport, and disposal methods planned for the removed material (ITRC, 2014). Dewatering technologies may rely upon gravity draining and evaporation processes (e.g., spreading and geotextile bags), mechanical processes (e.g., filter presses), and chemical conditioning (e.g., polymer additions and stabilization additives). The type of dewatering technology selected for use may depend upon the amount of space available for dewatering, the distance of the dewatering space from dredging operations, discharge options for treated dredge contact water, project scope, and cost of implementing the technology.

3.1.8 Disposal

Disposal of dredged or excavated sediment is the placement of materials into a controlled site or facility to permanently contain contaminants within the sediment. Management is achieved through the placement of materials into facilities such as sanitary landfills, hazardous material landfills, CDFs, or CAD facilities. Off-site landfills are generally used for dredged material disposal when on-site disposal is not feasible or when off-site disposal is more cost effective.

Landfills have been used for sediment volumes of over a million cubic yards. Typically, some type of on-site or near-site disposal facility is used at sites where dredged material volumes greater than 200,000 cubic yards are generated. Landfilling is also favored at smaller or moderately sized sites, where transportation is feasible. The associated hazards and cost of transporting and landfilling large volumes of sediment, along with treating and subsequent discharging of large volumes of dredge contact water as a result of sediment dewatering, make this disposal method somewhat less desirable than other solutions. Other considerations, such as public and stakeholder acceptance, lack of access to suitable on-site land- or water-based disposal facilities, and proximity to an existing off-site landfill may support the landfilling option.

CDFs are constructed to isolate dredged sediment from the surrounding environment. CDFs can be located upland, near shore, or in the water (as an island). Material staging or a temporary CDF may be necessary for dewatering dredged sediment. CDFs represent a common disposal method and typically are built for larger volume sites (200,000 cubic yards or more of sediment).

The CAD method deposits dredged material within a nearby body of water. A pre-existing depression within the sediment surface is preferred, though one can be created if necessary. Dredged sediment is deposited in the depression and capped with clean material. This process carries with it the same risks associated with using capping as a remedy (see **Section 3.1.5**). The goal of moving the contaminated sediment to the aquatic disposal site is to reduce the risk of exposure to contaminated materials (ITRC, 2014).

Disposal at landfills, CDFs, and CADs were screened independently in this evaluation.

3.1.9 In Situ Treatment

In situ sediment treatment involves applying or mixing of an amendment (such as AC) into sediments. Mixing may be achieved either passively, through natural biological processes such as bioturbation, or actively through mechanical means such as augers. In situ treatment technologies can achieve risk reduction in environmentally sensitive environments such as wetlands and submerged aquatic vegetation habitats, where sediment removal or containment by capping might be harmful. Treatment amendments typically reduce concentrations of freely

dissolved chemicals that are available for exposure to organisms or that may be mobilized and transferred from sediment to the overlying water column. The following in situ treatment technologies were screened in this evaluation:

- Immobilization – Immobilization treatments add chemicals or cements to reduce the leachability of contaminants. Mechanisms include solidification (encapsulation) or stabilization (chemical or absorptive reactions that convert contaminants to less toxic or mobile forms);
- Enhanced bioremediation – Microbial degradation by bacteria or fungi is enhanced by adding materials such as oxygen, nitrate, sulfate, hydrogen, nutrients, or microorganisms to the sediment;
- Chemical reduction – The addition of chemical reductants such as zero valent iron can facilitate the decomposition of organic matter;
- Chemical oxidation – The addition of chemical oxidizers to sediment can cause the rapid and complete chemical destruction of many toxic organic chemicals;
- Phytoremediation – Phytoremediation uses plant species to remove, transfer, stabilize, and destroy contaminants in sediment. Generally limited to sediments in shallow water zones and low concentrations; and
- Adsorption – Adsorbents can be used as sediment amendments for in situ treatment of contaminants. Sorption of metals and organics can take place simultaneously with a suitable combination of sorbents.

As discussed in **Section 3.1.3**, the results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments.

3.1.10 Remedial Technology Screening Results

Table 3 documents the technology screening process and results. The following remedial technologies were determined to be the most effective, implementable, and cost-effective and were retained for assembling the alternatives described in **Section 3.2**:

- ICs;
- Monitoring;
- EMNR/In-Situ Treatment;
- Capping;
- Sediment Excavation;
- Gravity and Chemical Conditioning Dewatering; and
- Landfill Disposal.

3.2 Development of Alternatives

This section describes the alternatives evaluated for the Site. The alternatives were developed using the selected remedial technologies discussed in **Section 3.1**, historical Site data, and the CSM. Site sediment chemical data was used to estimate the depth and spatial extent of the remedial areas for dioxins (the COC) as presented in **Figure 5**. A summary of the proposed alternatives is presented in **Table 4**. Calculations used to determine volumes, rates, and time frames related to remedy construction are presented in Table 1 in **Appendix B**. Assumptions made to compile cost estimates were incorporated into a Technical Analysis and are also included in **Appendix B**.

The total present value costs for alternatives presented within this FFS should be considered to be rough order of magnitude (ROM) costs. Based on the Association for the Advancement of Cost Engineering ROM classification chart, estimates presented in this FFS are considered Class 4. Class 4 estimates are considered Schematic Designs; 15 to 20% of the level of effort required to have a complete estimate has been done. Actual cost of the project could be 50% greater or 30% less (+50/-30) than the estimates developed thus far. ROM cost estimates for the FSS were compiled using a variety of sources. These sources include construction cost data from RS Means estimating software for open shop pricing in Duluth, Minnesota; current Bay West and state contract rates for labor, equipment, and sample analysis; personal communication with vendors; historic cost data from projects similar in size and scope; other FFS documents, presentations, or technical papers that provided estimated or real construction cost data; and available online vendor pricing of materials. Present value calculations are included in Table 5 in **Appendix B**.

3.2.1 Alternative 1: No Action

The NCP at Title 40 CFR provides that a No Action Alternative should be considered at every site. A No Action Alternative should reflect the site conditions described in the baseline risk assessment and RI. The No Action Alternative included within this FFS does not include any treatment or engineering controls, ICs, or monitoring. There are no costs associated with the No Action Alternative.

3.2.2 Alternative 2: Enhanced Monitored Natural Recovery with Thin-Layer Amended Cover

This alternative would consist of constructing a 0.30-meter (1.0-foot) amended cover over sediments with dioxins concentrations (i.e., COC concentrations) exceeding their respective Midpoint SQT (i.e., the CULs). Areas of the Site with sediments exceeding the CULs or anticipated to exceed the CULs are presented in **Figure 5** and **Figure 8**. These areas comprise the “remedial footprint” and total approximately 16.5 acres in size. Areas were selected to comprise the remedial footprint if sediments were known to have COCs exceeding the CULs or if net deposition within the area was suspected based on bathymetry and/or aerial imagery, and thus assumed to contain historically deposited contamination. The areas shown on **Figure 5** that comprise the remedial footprint are approximate and are based on limited Site data. Further Site characterization would need to be completed during the design phase to better delineate concentrations of COCs at the Site.

The objective of constructing an amended thin-layer cover over contaminated sediments is to 1) reduce availability of Site COCs in sediments and sediment pore water to aquatic organisms and thereby limit transfer of chemical contaminants to higher trophic organisms; and 2) provide some immediate isolation of contaminated sediments to aquatic life. It is anticipated that some cover material and associated amendments would be mixed into the underlying sediments over time through natural bioturbation processes caused by burrowing organisms, larger animal life, and rooting plants. Natural mixing of amendments into in situ contaminated sediments would increase the rate of contaminant sequestration (versus contact via groundwater upwelling alone) and, therefore, more rapidly decrease availability of contaminants to aquatic life. Implementation of this alternative assumes that an ongoing source is not present.

Contaminated sediments would remain in place as part of this alternative; therefore, ICs would be implemented and LTM would commence following construction of the thin-layer amended cover. The major components of Alternative 2 are described in the following sections.

3.2.2.1 Cover Design

A 0.30-meter cover thickness was evaluated for this FFS rather than a 0.15-meter cover thickness as sediment deposition rates at the Site are unknown. As previously stated, the thickness of the cover will be evaluated further, should it be selected as a remedial alternative. Further characterization should take place prior to project design to determine sedimentation rates and net depositional and erosional areas of the Site. It is anticipated that the remedial areas are depositional zones (although deposition may be slow or deposition events infrequent); however, if remedial areas are found to be net erosional zones then Alternative 2 may not be a viable option for the Site. Although the inlets off the primary river channel appear to be net depositional, armoring with gravel and/or cobble may be required in these areas to prevent erosion of cover materials during high flow events. Costs for placement of cobble in these areas were incorporated into this FFS.

The 0.30-meter cover would consist of sand mixed with one or more amendment materials appropriate for sequestration of the bioavailable fraction of COCs. It is anticipated that a single layer of a sand/amendment mix would be constructed rather than separate amendment and sand layers. Amendments mixed into and applied with soil or sand may provide better dispersion, uniformity, placement controls, and contact time when the required quantity of amendment is small, versus bulk placement of amendment materials (USEPA, 2013). Selection of an amendment material would be conducted during the design phase. As discussed in **Section 3.1.3**, the results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments. AC was incorporated into the cost analysis to address COC contamination for the purposes of this FFS. Likewise, the application rate of amendment material would also be conducted at the design phase. The chosen application rate of amendment to be applied should be capable of sequestering COCs migrating upward through the cover material for an indefinite period of time and should account for mixing of cover material into underlying sediments over time through bioturbation processes. The final amendment application rate may depend upon COC sediment concentrations, depth of contamination, and the presence or absence of groundwater upwelling. An application rate of 4% PAC by weight was incorporated into the cost analysis for the purposes of this FFS.

Implementation of this alternative assumes that approximately 850 cubic yards of amendment material would be mixed with 25,500 cubic yards of sand and applied over a 17-acre area at an average thickness of 0.30 meter. Approximately 2,000 cubic yards of armoring would be placed in areas adjacent the primary river channel, which total approximately 2.5 acres.

Final cover construction details would be determined during the design phase.

3.2.2.2 Implementation

Implementation of the EMNR with Thin-Layer Amended Cover Alternative would require construction of an upland staging area adjacent to the Site in which to stage and conduct all construction activities. The upland support area would be located in an easily accessible area that is both acceptable to the landowners (Minnesota Power and Sappi Cloquet, LLC) and is not disruptive to nearby residences. A potential staging area includes the open lot adjacent to the eastern dam as shown on **Figure 8**. Features of the upland staging area would consist of a site entrance and haul road, waterside access area, office trailer and parking area, cover material stockpile area, and various equipment storage areas.

The major implementation assumptions made to compile the cost analysis for this alternative include the following:

- Placement of cover materials using a barge-mounted excavator with clamshell bucket;
- Movement of materials between the staging area and the barge-mounted excavator using two small hopper barges; and
- A cover construction rate of 525 cubic yards per day or approximately 50 cubic yards per active placement hour.

The above assumptions would result in a construction time frame of approximately 72 working days or 14 weeks, assuming 5-day work weeks. This time frame includes 3 weeks for construction of the staging area, mobilization and setup of equipment, and breakdown and demobilization of equipment. See the Technical Analysis (**Appendix B**) text and tables for further project implementation assumptions used to compile the cost analysis.

3.2.2.3 Long-Term Monitoring

Contaminated sediments would remain in-place as part of Alternative 2 and, therefore, an LTM period would be necessary. LTM would commence after remedy implementation and would include collection of Site data to ensure that cover integrity is maintained as long as COCs remain in sediments above the CUL; ensure that ICs continue to be enforced as long as COCs remain in sediments above the CUL; monitor reduction trends in sediment toxicity to benthic organisms and COC bioaccumulation in benthic and fish tissue; and ensure that sediment contaminants are not migrating into or through the cover.

LTM data collection would be conducted periodically for an indefinite period of time or until concentrations of COCs in sediments attenuate to levels below the CULs and are deemed protective of the environment. For the purposes of this FFS, it was assumed that data collection would occur once every 5 years for a period of 30 years. If attenuation of COC concentrations to levels below the CULs does not occur after 30 years, then monitoring will likely continue.

Data collection will consist of the following:

- Collection of sediment cores or sediment profile imagery to observe mixing of cover material throughout the sediment column;
- Collection of sediment and cover samples to be analyzed for Site COCs;
- Collection of sediment samples for benthic toxicity and bioaccumulation analysis;
- Collection of fish tissue samples for bioaccumulation analysis; and
- Review of IC enforcement status.

Potential monitoring locations are presented in **Figure 8**.

3.2.2.4 Institutional Controls

ICs applicable to this alternative include those that would protect future cap integrity. Postings warning of contaminated sediments would be posted near potential Site access locations, and restrictions would be placed on intrusive Site activities such as dredging, construction of docks or piers, or other invasive Site improvements or alterations. ICs would be maintained until RAOs are achieved.

3.2.2.5 Cost

Calculations used to determine unit rate costs for each of the alternatives are presented in Table 2 in **Appendix B**. Other project costs determined on a lump sum basis are presented in Table 3 in **Appendix B**. The monitoring and evaluation program and associated costs developed for each

alternative are presented in Table 4 in **Appendix B**. The costs associated with each alternative are presented as Class 4 (+50/-30) estimates and are appropriate for remedial design alternative evaluations only.

The estimated total present value cost for Alternative 2 is $8,219,000. **Table 5** presents a breakdown of the estimated costs associated with Alternative 2.

3.2.3 Alternative 3: Potential Bioactive Zone Cap

This alternative would consist of constructing a sand cap over sediments with COC concentrations exceeding the CULs (i.e., within the remedial footprint, as presented in **Section 3.2.2** and on **Figure 5** and **Figure 9**). The constructed cap would be equal in thickness to the PBAZ, which in turn is determined based on the various habitat areas present at the Site (see **Section 1.4.4.2**). Construction of a cap equal to or greater in thickness than the PBAZ should provide contaminant isolation from aquatic plant and animal life. Construction of a cap would also mitigate exposure to human receptors, although human health criteria are not being used as cleanup criteria at this time (see **Section 1.4.3.3**). Addition of capping material to the Site would result in shallower water depths within the remedial footprint; however, the Scanlon hydro station is run-of-river meaning it doesn't maintain water capacity for future use. The hydro station generates electricity based on the current river flow. A reduction in water depth in areas of the Site is assumed not to interfere with current use of the Site. However, reduction in water volume within the Site could increase water velocity in certain areas and, therefore, should be factored into armoring considerations.

ICs would be implemented and LTM would commence following construction of the PBAZ cap. The major components of Alternative 3 are described in the following sections.

3.2.3.1 Potentially Bio-Active Zone Cap Design

The cap in this alternative would be a minimum of 0.50 meter (1.6 feet) thick and constructed in areas where Site COCs exceed the preliminary CULs (**Figure 9**), also referred to as the remedial footprint. The cap material would consist of natural materials suitable for benthic and aquatic plant habitat yet resist migration due to physical forces occurring within the Site. The cap thickness would be sufficient to protect aquatic life from burrowing or rooting into contaminated sediments below and prevent against contaminated sediments mixing with cap materials due to plant or animal bioturbation. This "zone" of animal and plant activity is referred to as the PBAZ, as discussed in **Sections 1.4.4.2** and **3.2.3**. The final designed cap thicknesses would likely be determined based on the type of habitat zone where the cap is to be constructed, and the PBAZ thickness associated with the particular habitat zone. These habitat areas are presented in **Figure 6** and the associated cap thicknesses are presented in **Figure 9**. All three habitat areas exist within the Site boundary and, therefore, 0.50-meter, 1.0-meter, and 1.2-meter cap thicknesses were incorporated into the theoretical cap design for this FFS. In addition to the PBAZ zone thickness an additional 0.15 meter was incorporated into the cap design to account for mixing of sediments and capping material during construction.

Armoring with gravel and/or cobble may be necessary to maintain long-term cap integrity in areas that experience seasonal or other erosion events. As discussed for Alternative 2, areas that may require armoring include those immediately adjacent to the primary river channel. These areas total approximately 2.5 acres in size and costs for armoring these areas were included in the cost analyses. Further investigation into Site hydrology should be conducted during the design phase to determine areas susceptible to scouring.

It was assumed for the purposes of the cost analysis that sand and armoring materials would be purchased from an upland borrow source. Previously dredged materials, such as those contained within the Erie Pier CDF, could also be investigated for use at the Site as capping material.

Implementation of this alternative assumes that approximately 88,000 cubic yards of sand would be placed over 16.5 acres to construct the sand cap. Approximately 2,000 cubic yards of armoring would be placed in areas adjacent the primary river channel, which total approximately 2.5 acres.

Final cap construction details would be determined during the design phase.

3.2.3.2 Construction Implementation

Implementation of the PBAZ Cap Alternative would require construction of a staging area as detailed for the EMNR with Thin-Layer Amended Cover Alternative in **Section 3.1.4**. The major implementation assumptions made to compile the cost analysis for this alternative include the following:

- Placement of cap materials using a barge-mounted excavator with clamshell bucket;
- Movement of materials between the staging area and the barge-mounted excavator using two small hopper barges; and
- A sand cap construction rate of 525 cubic yards per day or 50 cubic yards per active placement hour.

The above assumptions would result in a construction time frame of approximately 187 working days or 31 weeks, assuming 6-day work weeks in order to implement construction within a single construction season. This time frame includes 3 weeks for construction of the staging area, mobilization and setup of equipment, and breakdown and demobilization of equipment. See the Technical Analysis (**Appendix B**) text and tables for further project implementation assumptions used to compile the cost analysis.

3.2.3.3 Long-Term Monitoring

Contaminated sediments would remain in-place as part of Alternative 3 and, therefore, an LTM period would be necessary. LTM would commence after remedy implementation and would include collection of Site data to: ensure that cap integrity is maintained as long as COCs remain in sediments above the CUL; ensure that ICs continue to be enforced as long as COCs remain in sediments above the CUL; monitor reduction trends in sediment toxicity to benthic organisms and COC bioaccumulation in benthic and fish tissue; and ensure that sediment contaminants are not migrating into or through the cap.

LTM data collection would be conducted periodically for an indefinite period of time or until concentrations of COCs in sediments beneath the cap attenuate to levels below the CULs and RAOs are achieved. For the purposes of this FFS, it was assumed that data collection would occur once every 5 years for a period of 30 years. If attenuation of COC concentrations to levels below the CULs does not occur after 30 years, then monitoring will likely continue.

Data collection will consist of the following:

- Collection of sediment samples from below the cap to be analyzed for COCs;
- Collection of cap samples to be analyzed for COCs;
- Coring to determine cap thicknesses and integrity;
- Collection of sediment samples for benthic toxicity and bioaccumulation analysis;
- Collection of fish tissue samples for bioaccumulation analysis; and
- Review of IC enforcement status.

Potential monitoring locations are presented in **Figure 9**.

3.2.3.4 Institutional Controls

Contaminated sediments would remain in-place as part of Alternative 3 and, therefore, ICs would be required to ensure remedy effectiveness. ICs applicable to this alternative include those that would protect future cap integrity. ICs applicable to Alternative 3 are identical to those detailed for Alternative 2 in **Section 3.2.2.4**.

3.2.3.5 Cost

Calculations used to determine unit rate costs for each of the alternatives are presented in Table 2 in **Appendix B**. Other project costs determined on a lump sum basis are presented in Table 3 in **Appendix B**. The monitoring and evaluation program and associated costs developed for each alternative are presented in Table 4 in **Appendix B**. The costs associated with each alternative are presented as Class 4 (+50/-30) estimates and are appropriate for remedial design alternative evaluations only.

The estimated total present value cost for Alternative 3 is $8,508,000. **Table 6** presents a breakdown of the estimated costs associated with Alternative 3.

3.2.4 Alternative 4: Sediment Dredging and Excavation

The objective of the Sediment Dredging and Excavation Alternative is complete removal of sediments with COCs exceeding the CULs (i.e., within the remedial footprint, as presented in **Section 3.2.2** and on **Figure 5** and **Figure 10**). Removal of contaminated sediments would mitigate exposure of aquatic receptors to sediment contaminants, thus allowing for achievement of RAOs. The presence of any dredge residuals exceeding CULs following completion of dredging activities may require additional actions to be taken, such as placement of a cover to mix, dilute, and cover any remaining dredge residuals, enforcement of ICs, and post-construction monitoring. The success of a dredging and excavation remedy at removing all contaminated sediments cannot be determined at this time and, therefore, IC and monitoring costs associated with addressing dredge residuals were not incorporated into the cost analysis. The placement of a 0.15-meter (0.5-foot) layer of clean sand following dredging implementation was assumed within the cost analysis to manage dredge residuals and to provide benthic habitat if dredging is conducted to bedrock in some areas of the Site.

Based on input from MPCA multiple dredging passes instituted based on exceedances of post dredge verification criteria would not be conducted. Dredging would be conducted to a defined dredge prism neat line using best management practices to control and reduce contaminated dredge residuals. A Normal Dredge Residue verification approach may be used to ensure that best management practices are being followed and that “undredged inventory” is accounted for.

The major components of Alternative 4 are described in the following sections.

3.2.4.1 Dredge and Excavation Volume Assumptions

The estimated remedial footprint for this alternative is the same as Alternatives 2 and 3 and is presented in **Figure 5** and **Figure 10**. As stated previously, the remedial footprint is estimated at 17 acres and all areas that comprise the remedial footprint are assumed to contain historically deposited sediments with COCs exceeding CULs. The depth of contamination was estimated at 0.50 meter (1.6 feet) within areas adjacent to the primary river channel and 0.65 meter (2.1 feet) within all areas of the eastern arm (see **Section 1.4.3.5**). These estimates equate to a total volume of approximately 55,000 cubic yards of contaminated sediments requiring removal. An average over dredge of 0.15 meter (0.5 feet) was assumed across the entire remedial footprint, which increases the total volume of sediments to be removed to 68,000 cubic yards. Further

sampling would be required to further delineate the vertical and horizontal extent of contamination at the Site.

3.2.4.2 Construction Implementation

Potential sediment removal methods were reviewed along with Site constraints to develop a construction scenario and cost analysis for this alternative. Assumptions regarding construction implementation are contained within the Technical Analysis (**Appendix B**) and consist of the following elements:

- Construction of a staging area, to include a lined sediment dewatering area and materials staging area;
- Installation of a water-tight cofferdam and subsequent dewatering of the area within the cofferdam;
- Dredging of sediments “in the wet” using a barge-mounted excavator in areas adjacent to the primary river channel and outside the cofferdam area (production rate of 50 cubic yards per hour or approximately 525 cubic yards per day);
- Debris removal and excavation of sediments “in the dry” using common earth equipment in areas within the cofferdam (production rate of 72 cubic yards per hour or approximately 750 cubic yards per day);
- Sediment solidification and off-site landfill disposal;
- Construction of a 0.15-meter sand cover over the entire remedial footprint;
- Habitat restoration and wetland plantings; and
- Site restoration.

Implementation of the construction scenario above would require multiple construction phases to be conducted concurrently in order to implement the remedy within a single construction season. For example, construction of the cofferdam and staging area could be conducted concurrently, as could dredging of sediments outside the cofferdam and dewatering of the cofferdam area. The estimated time frame to implement the sediment dredging and excavation construction period, given the assumptions outlined above, is approximately 141 working days or 28 weeks assuming a 5-day work week. See the Technical Analysis (**Appendix B**) text and tables for further project implementation assumptions used to compile the cost analysis.

3.2.4.3 Cost

Calculations used to determine unit rate costs for each of the alternatives are presented in Table 2 in **Appendix B**. Other project costs determined on a lump sum basis are presented in Table 3 in **Appendix B**. The monitoring and evaluation program and associated costs developed for each alternative are presented in Table 4 in **Appendix B**. The costs associated with each alternative are presented as Class 4 (+50/-30) estimates and are appropriate for remedial design alternative evaluations only.

The estimated total present value cost for Alternative 4 is $10,101,000. **Table 7** presents a breakdown of the estimated costs associated with Alternative 4.

3.2.5 Alternative 5: Enhanced Monitored Natural Recovery with Broadcast Amendment

This alternative would consist of broadcasting an amendment material over sediments with COC concentrations exceeding the Midpoint SQT (i.e., the CULs). Areas of the Site exceeding the CULs are presented in **Figure 11** and equal approximately 16.5 acres. The objective of applying

an amendment material to in situ sediments at the Site is to reduce availability of Site COCs in sediments and sediment pore water to aquatic organisms and thereby limit the exposure and affects to the organisms and transfer of chemical contaminants to higher trophic organisms. This alternative was developed to minimize intrusive remedial action construction activities within habitats already established at the Site.

ICs would be implemented and LTM would commence following application of the selected amendment to remedial areas. The major components of Alternative 5 are described in the following sections.

3.2.5.1 Amendment Selection and Application Rate

This alternative consists of applying a thin layer of amendment material directly on top of in situ contaminated sediments. It is anticipated that the amendment material would be mixed into the underlying sediments over time through natural bioturbation processes caused by burrowing organisms, larger animal life, and rooting plants; therefore, this alternative is intended to reduce contaminant availability rather than provide isolation from contaminants as in a traditional capping scenario. The chosen amendment material would reduce exposure of aquatic life to COCs through sequestration of the bioavailable fraction of COCs in sediments and sediment pore water. Selection of an amendment material would be conducted during the design phase; however, as discussed in **Section 3.1.3**, the results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments. For the purposes of this FFS, the selected amendment material will be pelletized activated carbon pellets.

The chosen application rate (i.e., thickness) of amendment to be applied should be capable of sequestering COCs in sediments and sediment pore water for an indefinite period of time, assuming that no ongoing source of contamination is present. It was assumed that a 0.01-meter layer of amendment material would be applied to in situ sediments strictly for cost analysis purposes. The final amendment application rate would be determined during the design phase and may largely depend upon COC sediment concentrations, depth of contamination, and the presence or absence of groundwater upwelling.

Implementation of this alternative assumes that approximately 850 cubic yards of amendment material would be broadcasted over a 16.5-acre area at an average thickness of 0.01 meter.

3.2.5.2 Long-Term Monitoring

Contaminated sediments would remain in-place as part of Alternative 5, and therefore, an LTM period would be necessary. LTM would commence after remedy implementation and would include collection of Site data to ensure that cover integrity is maintained as long as COCs remain in sediments above the CUL; ensure that ICs continue to be enforced as long as COCs remain in sediments above the CUL; monitor reduction trends in sediment toxicity to benthic organisms and COC bioaccumulation in benthic and fish tissue; and ensure that sediment contaminants are not migrating into or through the cover.

LTM data collection would be conducted periodically for an indefinite period of time or until concentrations of COCs in sediments attenuate to levels below the CULs and are deemed protective of the environment. For the purposes of this FFS, it was assumed that data collection would occur once every 5 years for a period of 30 years. If attenuation of COC concentrations to levels below the CULs does not occur after 30 years, then monitoring will likely continue.

Data collection will consist of the following:

- Collection of sediment cores or sediment profile imagery to observe mixing of cover material throughout the sediment column;
- Collection of sediment and cover samples to be analyzed for Site COCs;
- Collection of sediment samples for benthic toxicity and bioaccumulation analysis;
- Collection of fish tissue samples for bioaccumulation analysis; and
- Review of IC enforcement status.

Potential monitoring locations are presented in **Figure 11**.

3.2.5.3 Institutional Controls

ICs applicable to this alternative include those that would protect future cap integrity. The MDH currently communicates fish consumption guidelines for the lakes and rivers of Minnesota. Advisories for consumption of fish within the SLR and below the Fond du Lac Dam are in place for 11 species of fish due to the presence of mercury and PCBs within fish tissue. No specific advisories are in place related to dioxins. It is currently unknown whether the meal advice provided within the fish consumption guidelines for mercury is protective for dioxins as well; therefore, the applicability of meal guidelines to dioxins would require investigation. Postings warning of contaminated sediments would be posted near potential Site access locations, and restrictions would be placed on intrusive Site activities such as dredging, construction of docks or piers, or other invasive Site improvements or alterations. ICs would be maintained until RAOs are achieved.

3.2.5.4 Cost

Calculations used to determine unit rate costs for each of the alternatives are presented in Table 2 in **Appendix B**. Other project costs determined on a lump sum basis are presented in Table 3 in **Appendix B**. The monitoring and evaluation program and associated costs developed for each alternative are presented in Table 4 in **Appendix B**. The costs associated with each alternative are presented as Class 4 (+50/−30) estimates and are appropriate for remedial design alternative evaluations only.

The estimated total present value cost for Alternative 5 is $3,355,000. **Table 8** presents a breakdown of the estimated costs associated with Alternative 5.

4.0 REMEDY SELECTION CRITERIA

The alternatives were evaluated and compared using the NCP remedy selection criteria outlined below and in general accordance with USEPA guidelines for feasibility studies (USEPA, 1990). The NCP remedy selection criteria are divided into three groups based on the function of the criteria in remedy selection. The NCP definitions of each criterion are included below. Green Sustainable Remediation (GSR) criteria were also evaluated during this FS and are included as a fourth group of criteria. Additional detail may be added from MPCA and/or USEPA guidance where appropriate.

4.1 Threshold Criteria

The Threshold Criteria relate to statutory requirements that each alternative must satisfy in order to be eligible for selection and include the following:

4.1.1 Overall Protection of Human Health and Ecological Receptors

Alternatives shall be assessed to determine whether they can adequately protect human health and ecological receptors, in both the short- and long-term, from unacceptable risks posed by hazardous substances, pollutants, or contaminants present at the Site by eliminating, reducing, or controlling exposures to levels established during development of remediation goals. Overall protection of human health and the environment draws on the assessment of other evaluation criteria, especially long-term effectiveness and permanence, short-term effectiveness, and compliance with ARARs. RAOs for the Site do not include the protection of human health based on the exposure pathway evaluation presented in **Section 1.4.4**; therefore, the overall protection of human health was not assessed for remedial alternatives in this FFS.

4.1.2 Compliance with Applicable or Relevant and Appropriate Requirements

The alternatives shall be assessed to determine whether they attain ARARs under Federal environmental laws and State environmental or facility citing laws or provide grounds for invoking a waiver.

4.2 Primary Balancing Criteria

The Primary Balancing Criteria are the technical criteria upon which the detailed analysis is primarily based and include the following.

4.2.1 Long-Term Effectiveness and Permanence

Alternatives shall be assessed for the long-term effectiveness and permanence they afford, along with the degree of certainty that the alternative will prove successful. Factors that shall be considered, as appropriate, include the following:

1. Magnitude of residual risk remaining from untreated waste or treatment residuals remaining at the conclusion of the remedial activities. The characteristics of the residual should be considered to the degree that they remain hazardous, taking into account their volume, toxicity, mobility, and propensity to bioaccumulate.
2. Adequacy and reliability of controls, such as containment systems and ICs, necessary to manage treatment residuals and untreated waste. This factor addresses, in particular, the uncertainties associated with land disposal for providing long-term protection from residuals; the assessment of the potential need to replace technical components of the

alternative, such as a cap, a slurry wall, or a treatment system; and the potential exposure pathways and risks posted should the remedial action need replacement.

4.2.2 Reduction of Toxicity, Mobility, or Volume Through Treatment

The degree to which alternatives employ recycling or treatment that reduces toxicity, mobility, or volume shall be assessed, including how treatment is used to address the principal risks posed by the Site. Factors that shall be considered, as appropriate, include the following:

1. The treatment or recycling processes the alternatives employ and materials they will treat;
2. The amount of hazardous substances, pollutants, or contaminants that will be destroyed, treated or recycled;
3. The degree of expected reduction in toxicity, mobility, or volume of the waste due to treatment or recycling and the specification of which reductions(s) are occurring;
4. The degree to which the treatment is irreversible;
5. The type and quantity of residuals that will remain following treatment, considering the persistence, toxicity, mobility, and propensity to bioaccumulate of such hazardous substances and their constituents; and
6. The degree to which treatment reduces the inherent hazards posed by principal threats at the Site.

4.2.3 Short-Term Effectiveness

The short-term impacts of alternatives shall be assessed considering the following:

1. Short-term risks that might be posed to the community during implementation of an alternative;
2. Potential impacts on workers during remedial action and the effectiveness and reliability of protective measures;
3. Potential environmental impacts of the remedial action and the effectiveness and reliability of mitigating measures during implementation; and
4. Time until protection is achieved.

4.2.4 Implementability

The ease or difficulty of implementing the alternatives shall be assessed by considering the following types of factors, as appropriate:

1. Technical feasibility, including technical difficulties and unknowns associated with the construction and operation of a technology, the reliability of the technology, ease of undertaking additional remedial actions, and the ability to monitor the effectiveness of the remedy;
2. Administrative feasibility, including activities needed to coordinate with other offices and agencies and the ability and time required to obtain any necessary approvals and permits from other agencies (for off-site actions); and
3. Availability of services and materials, including the availability of adequate off-site treatment, storage capacity, and disposal capacity and services; the availability of necessary equipment and specialists, and provisions to ensure any necessary additional resources; the availability of services and materials; and the availability of prospective technologies.

4.2.5 Costs

The types of costs that shall be assessed include the following:

1. Capital costs, including both direct and indirect costs;
2. Annual O&M costs; and
3. Net present value of capital and O&M costs.

The USEPA guidance document *A Guide to Developing and Documenting Cost Estimates During the Feasibility Study* (USEPA, 2000) was used to develop cost estimates presented in this FFS. The cost estimates developed for this FFS are primarily for the purpose of comparing remedial alternatives during the remedy selection process, not for establishing project budgets. As previously described, cost estimates are considered Class 4 estimates, Schematic Design.

4.3 Modifying Criteria

The third group is made up of the Modifying Criteria specified below. These last two criteria are assessed formally after the public comment period, although to the extent that they are known will be factored into the identification of the preferred alternative.

4.3.1 State/Support Agency Acceptance

Assessment of state/agency concerns may not be completed until comments on this FFS are received, but may be discussed, to the extent possible, in the proposed plan issued for public comment. The state/agency concerns that shall be assessed include the following:

1. The state's/agency's position and key concerns related to the preferred alternative and other alternatives; and
2. State/agency comments on ARARs or the proposed use of waivers.

4.3.2 Community Acceptance

This assessment includes determining which components of the alternatives interested persons in the community support, have reservations about, or oppose. This assessment may not be completed until comments on the proposed plan are received.

4.4 Green Sustainable Remediation

The last group is made up of the GSR criteria specified below. There are six criteria included with this analysis, which are then summarized to provide each alternative with an overall qualitative GSR rating. The six GSR criteria evaluated with this FFS include the following:

- Greenhouse Gas (GHG) Emissions;
- Toxic Chemical Usage and Disposal;
- Energy Consumption;
- Use of Alternative Fuels;
- Water Consumption; and
- Waste Generation.

5.0 COMPARATIVE ANALYSIS OF ALTERNATIVES

The purpose of the comparative analysis is to identify and compare advantages and disadvantages of each evaluated alternative, relative to one another, with respect to remedy selection criteria presented in **Section 4.0** in order to determine which of the alternatives best meets those criteria. The comparative analysis is documented in this section and summarized in **Table 9** and **Table 10**. **Table 11** presents a numerical comparison of the evaluated alternatives.

5.1 Threshold Criteria

5.1.1 Overall Protection of Ecological Receptors

Only those alternatives that would meet the threshold criteria of providing overall protection of ecological receptors were carried forward with the comparative analysis. Alternative 1 would not meet the threshold criteria but was carried forward as it is required for analysis under the NCP. Alternatives 2, 3, 4, and 5 would adequately protect ecological receptors from unacceptable risks posed by hazardous substances, pollutants, or contaminants present at the Site; however, contaminated sediment would remain in place under Alternatives 2, 3, and 5 requiring monitoring to ensure long-term effectiveness. Alternative 4 would provide the highest level of protection, since contaminated sediments would be removed from the aquatic environment.

5.1.2 Compliance with Applicable or Relevant and Appropriate Requirements

Only alternatives that meet threshold criteria were carried forward, as stated previously. Alternative 1 does not meet the threshold criteria but was carried forward as it is required for analysis under the NCP. Alternatives 2, 3, 4, and 5 comply with the ARARs identified in **Section 2.0**.

5.2 Balancing Criteria

5.2.1 Long-Term Effectiveness and Permanence

Alternative 1 is not effective in the long term or permanent. Benchscale testing indicates Alternatives 2 and 5 will likely be effective in the long term if amendment materials mix into underlying sediments and sequester sediment contaminants throughout the entire PBAZ, and if clean sediments are deposited at the Site over time and thus isolate sediment contaminants. Sediment erosion and deposition data are limited for the Site and uncertainty of the long-term permanence of Alternatives 2 and 5 is relatively high. Unknowns in the hydrodynamic model, such as the erosion of contaminated sediments, as well as the effects of periodic flooding, may also reduce the long-term effectiveness and permanence of Alternative 2 and 5. Further, Alternative 5 does not include armoring, which makes the amendment material more susceptible to erosion during high-flow events. Alternative 3 would be effective in the long-term but would require long-term O&M and ICs to ensure long-term effectiveness as contaminated sediments would remain in place. Therefore, Alternative 3 is not as permanent as Alternative 4. Alternative 4 would provide the highest degree of long-term effectiveness and permanence as all contaminated sediments would be removed, even though contaminants would not be permanently destroyed in the landfill.

In summary, Alternative 4 would provide a high achievement of this criterion by removing all of the contaminated sediment in the aquatic environment above the SQTs. Alternatives 2 and 5 would provide a moderate to high achievement of this criterion, since amendment materials would eventually mix into the sediment column and sequester contaminants within the most biologically active sediment zone; however, deeper contamination within the PBAZ may remain in place. Additionally, it is unknown if clean sediments would be deposited over the remedial footprint in

the future. Alternative 3 would provide a moderate to high achievement of this criterion as it would isolate sediment contaminants and provide a full thickness PBAZ, yet would require long-term O&M and ICs.

5.2.2 Reduction of Toxicity, Mobility, or Volume Through Treatment

Alternatives 1, 3, and 4 would not provide a reduction in the toxicity, mobility, or volume through treatment. Although Alternative 3 does not incorporate treatment, the mobility of sediment contaminants would be reduced through capping, and concentrations of COCs in capped sediments would be reduced over time through natural processes. Alternative 4 also does not include treatment but would remove all contaminated sediment from the aquatic environment and place it in a maintained landfill, and thus would provide a reduction in toxicity, mobility, and volume of contaminants at the Site. Alternatives 2 and 5 would reduce the toxicity, mobility, or volume of sediment contaminants through treatment/sequestration of sediment contaminants in contact with amendment materials (i.e., near the sediment surface) rendering them unavailable to biota; however, it is unlikely that bioturbation processes would mix amendment materials to the maximum depth of contamination and, therefore, some contamination would remain in place indefinitely. In Alternative 2, amendment materials mixed into the sand cover would also reduce contaminant mobility into the water column by providing a sorptive barrier between contaminated sediments and the water column.

In summary, Alternatives 2 and 5 would provide a moderate to high achievement of this criterion by reducing the toxicity and mobility of sediment contaminants through treatment via amendment materials mixed into cover material and eventually mixed into the sediment column through bioturbation processes. Alternatives 3 and 4 would provide a low achievement as toxicity and mobility of sediment contaminants would be reduced at the Site, but not through treatment. Alternative 1 would provide no achievement of this criterion as sediment contaminants would remain in place and no remedial actions would be taken.

As summarized in **Section 1.4.3.2**, in order to evaluate how a remedial alternative involving AC might affect mercury, which is present in sediment at the Site but determined not to be a COC, a literature review was completed by the United States Army Research and Development Center. The review focused on how a remedy involving AC will impact the potential for mercury toxicity and bioaccumulation. Review of available research indicated either AC is a useful sorbent for reducing the potential for mercury bioaccumulation, or that AC is not effective at sorbing mercury; however, AC does not appear to increase the potential for mercury toxicity and bioaccumulation in water or sediments (U.S. Army Research and Development Center, 2020).

5.2.3 Short-Term Effectiveness

There are no short-term risks associated with Alternative 1 as no actions would be implemented at the Site. Alternatives 2, 3, 4, and 5 would have short-term risks associated with remedy implementation, and in general the potential short-term risks increase as the complexity of each alternative increases. Alternative 4 would require dredging/excavation of sediments that would result in removal of a portion of, or the entirety of, the PBAZ and temporary destruction of plant and animal habitat over the entire remedial footprint. Additionally, dry excavation of sediments would require dewatering of the area within the cofferdam and would result in disruption or death of fish, benthics, and other aquatic life. Dredging/excavation of sediments would remove contamination from beneath the water column and would require multiple transfers of contaminated sediments (and dredge contact water) by Site workers until eventual landfill disposal, thus creating additional opportunities for exposure to Site workers.

Short-term adverse effects to aquatic habitat and biota would be similar among Alternatives 2 and 3 and would include displacement of fish and smothering of benthic organisms. Alternative 5 would provide the least adverse effects of the alternative because a 0.01-meter layer of amendment material would be place over the remedial footprint rather than a thicker cover or cap as in Alternatives 2 and 3. Alternative 5 also has a significantly shorter construction duration than the other alternatives, reducing site workers' exposure to contaminants and construction safety issues. Site workers' exposure in Alternatives 2 and 3 would be substantially less as compared to Alternative 4 but risks due to heavy equipment operation would be similar among all three. The effects on aquatic habitat and biota from Alternatives 2, 3, 4, and 5 would occur during remedy implementation and during the recovery period thereafter. Biota would be expected to be reestablished for all alternatives within several growing seasons.

In summary, Alternative 1 would provide a high achievement of the short-term effectiveness criterion as there would be no impact to surrounding community and aquatic habitat and no risk to Site workers. Alternative 5 would also provide a high achievement of this criteria due to limited impact to the benthic community and short construction duration. Alternatives 2 and 3 would have a moderate to high achievement of the short-term effectiveness criterion due to an increase in short-term adverse effects to aquatic biota during cover/cap construction; however, impacts are anticipated to be small. Alternative 4 would have a moderate achievement of the short-term effectiveness criterion due to the adverse effects to benthic organisms and Site workers through handling of contaminated sediments.

5.2.4 Implementability

There are no implementability concerns associated with Alternative 1 as no remedial or monitoring actions would be taken at the Site. Alternatives 2, 3, 4, and 5 are all technically feasible and implementable from an engineering perspective. Each of these alternatives relies upon highly proven technologies that have been implemented at a wide range of contaminated sediment sites. Additionally, each of these alternatives incorporates the use of widely available equipment and materials. The primary implementation concern associated with Alternatives 2, 3, 4, and 5 is the need for a staging area to be constructed adjacent to the Site to support all construction activities. This would require cooperation of landowners surrounding the Site and use of private lands for Site access and upland staging area construction.

Construction windows for alternatives may post implementability concerns. Alternative 5 and Alternative 2 has a shorter construction duration than the other alternatives, requiring only one construction season to implement and reducing timing constraints and implementability concerns. Alternatives 3 and Alternative 4 may require significantly more time to construct, likely requiring two construction seasons to implement.

Implementation of Alternative 4 is more complex than Alternatives 2, 3, and 5 as it involves installation of a watertight cofferdam, dewatering and treatment of water inside the cofferdam, and additional material handling operations related to sediment removal. Installation of a cofferdam and subsequent dewatering assumes that groundwater infiltration can be controlled during sediment removal activities and that the sediment surface will be firm enough to support crane mat roadways and/or equipment. The additional handling of contaminated sediments associated with Alternative 4 would also require the construction of a lined sediment stabilization pad in which to stage dredged materials prior to loadout and transportation to a landfill.

In summary, Alternative 1 has no actions to be implemented and thus provides a high achievement of the implementability criterion. Alternatives 2, 3, and 5 are the next easiest to implement since they do not incorporate dredging and provide a moderate to high achievement of this criterion. Alternative 4 provides a moderate achievement of the implementability criterion due to increased complexities related to construction activities and the uncertainty regarding removal of sediments in the dry.

5.2.5 Cost

Cost estimates developed for each alternative are included in **Section 3.2** and summarized in **Table 4**. The cost estimates include: capital costs, including both direct and indirect costs; annual O&M costs; and net present value of capital and O&M costs.

In summary, Alternative 1 provides the most cost-effective option as no remedial or monitoring activities would take place, followed by Alternative 5 because it requires the least amount of time and materials of any active remedy. Alternative 2 is the next most cost-effective because it includes the construction of a cover thicker than Alternative 5 that includes both amendment material and sand. Alternative 3 is the next most cost effective as additional time and materials are required to place the thicker cap, but no expensive amendment material or sediment removal would be conducted. Alternative 4 is the least cost effective as it includes sediment removal and construction of a thin-layer cover following sediment removal. Sediment removal necessitates additional material handling, stabilization, transportation, and disposal costs. **Table 11** presents a numerical score that compares the costs for all alternatives.

5.3 Modifying Criteria

The modifying criteria, State/support agency acceptance and community acceptance, are assessed formally after the public comment period, and to the extent that they are known will be factored into the identification of the preferred alternative.

5.3.1 State Support/Agency Acceptance

State/agency input will be assessed to assist in determining the appropriate alternative for the Site. Key factors that will influence alternative selection include but are not limited to knowledge of future Site use, Site remediation prioritization, and funding source availability. Alternatives 1 through 4 will be formally assessed after public comment period.

5.3.2 Community Acceptance

All lands surrounding the Site are privately owned and, therefore, Site access is limited to trespassers. Dams located upstream and at the southern border of the Site prevent boaters, kayakers, and canoers from accessing the Site from downstream or upstream locations. No recreational activities should be interrupted by construction activities related to remedy implementation.

Implementation of Alternatives 2, 3, 4, and 5 would result in increased truck traffic within the immediate vicinity of the Site; however, truck traffic would primarily be limited to highway routes and would not travel through residential or commercial areas. The volume of truck traffic would be directly related to the volume of materials imported or exported from the Site and, therefore, Alternative 4 would result in the greatest increase in truck traffic, followed by Alternatives 3 and 2. Construction noise, odors, and/or dust should be minimal due to the confined location of the Site and distance from residential, commercial, and industrial properties.

It is anticipated that community acceptance of Alternatives 2, 3, 4, and 5 would be high due to limited impacts on the community cause by construction activities.

Any remediation work completed at the Site involving covering, capping, or removal of sediments would require construction of a nearby staging area in which to stage materials and/or sediments and conduct all construction-related activities. Construction of a staging area would require cooperation with private landowners. Acceptance of Alternative 4 by landowners may be lower than Alternatives 2, 3, and 5 due to handling of contaminated sediments at upland areas.

5.4 Green Sustainable Remediation Criteria

5.4.1 Greenhouse Gas Emissions

Alternative 1 would have no GHG emissions. Alternatives 2, 3, 4, and 5 would result in GHG emissions from the mobilization, operation, and demobilization of all fuel-powered construction equipment required for cover/cap construction and sediment removal. Alternative 4 would also produce emissions during transport of sediments by truck to the disposal facility. Reduction of emissions can be accomplished by using equipment that is compliant with the latest USEPA non-road engine standards and retrofitting older equipment with appropriate filters.

5.4.2 Toxic Chemical Usage and Disposal

There are no known toxic chemicals associated with these alternatives.

5.4.3 Energy Consumption

Alternative 1 would consume no additional energy. Alternatives 2, 3, 4, and 5 would result in the consumption of fossil fuels for the mobilization, operation, and demobilization of all diesel-powered construction equipment associated with the removal, hauling, and disposal of contaminated sediments and the installation of cover/cap materials. The amount of cover/cap material placed for Alternative 5 is considerably less than Alternatives 2 and 3, and therefore, Alternative 5 requires less energy to implement. Alternative 4 would require the greatest amount of energy to implement as it involves sediment removal and cover construction.

5.4.4 Use of Alternative Fuels

Alternative 1 would not require the use of alternative fuels. Biodiesel blended fuels (B10 or B20) could be used as a supplemental fuel source for all diesel-powered construction equipment associated with Alternatives 2, 3, 4, and 5.

5.4.5 Water Consumption

Alternatives 1, 2, 3, and 5 would not require the consumption of water. A minimal quantity of water would be required to decontaminate personnel and equipment during sediment dredging activities associated with Alternative 4.

5.4.6 Waste Generation

Alternatives 1, 2, 3, and 5 would not generate waste. Alternative 4 would generate waste that includes the dredged/excavated contaminated sediments, contaminated dewatering pad materials, and any non-recyclable water treatment media that would be removed from the Site and disposed of.

5.5 Comparative Analysis Summary

The comparative analysis of alternatives narrative discussion and quantitation table identified Alternative 5 as having a higher score than Alternatives 1, 2, 3, and 4 to address contamination at the Site. Alternative 1 does not achieve overall protection of ecological receptors, does not achieve ARARs, is not effective in the long-term, does not reduce toxicity, mobility, or volume of contamination, and is not effective in the short term; however, this alternative is implementable and cost effective. Alternatives 2, 3, 4, and 5 are all protective of ecological receptors and achieve ARARs. Alternative 4 is the most effective in the long-term and is the most permanent, while Alternatives 2 and 5 are the only alternatives that reduces the toxicity, mobility, or volume of contamination through treatment. Alternative 5 results in the least short-term effects resulting from remedy implementation and are also less expensive to implement than Alternative 2, 3, and 4. Besides Alternative 1, Alternative 5 is the least expensive to implement, followed by Alternative 2, Alternative 3, and Alternative 4. Based on the comparative analysis, Alternative 3 and Alternative 2 scored the highest and should be evaluated further for remedy selection.

Benchscale treatability testing (**Appendix C**) was completed on sediments collected from the Site to evaluate the effectiveness of different AC amendments and doses to reduce the bioavailability of dioxins/furans in Site sediments using two AC particle size ranges; a silt-sized powdered activated carbon (PAC) and a fine sand-sized granular activated carbon (GAC). The results of the benchscale treatability indicated that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins in Site sediments. Application methods will be retained for further engineering and cost evaluations.

Review of available research indicates either AC is a useful sorbent for reducing the potential for mercury bioaccumulation, or that AC is not effective at sorbing mercury; however, AC does not appear to increase the potential for mercury toxicity and bioaccumulation in water or sediments (U.S. Army Research and Development Center, 2020).

The modifying criteria, State/support agency acceptance, and community acceptance are assessed formally after the public comment period. Stakeholder and community input will provide valuable insight as the MPCA considers information for the selection of a preferred alternative. The MPCA will conduct outreach activities to resource managers, current Site users, the public and local units of government prior to the public comment period.

Further studies are recommended during the design phase of the selected alternative. These recommended studies, depending on the alternative selected, may include:

- Further delineation and determination of extent, thickness, and volume of contaminated sediment.
- Hydrodynamic study to understand natural processes such as depositional and scouring forces to inform design and placement of cover materials, and effectiveness of Monitored Natural Recovery (MNR).
- Updated bathymetric survey and mapping of substrate types.
- Investigation into the potential for ongoing sources related to diffuse regional source.

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Figures

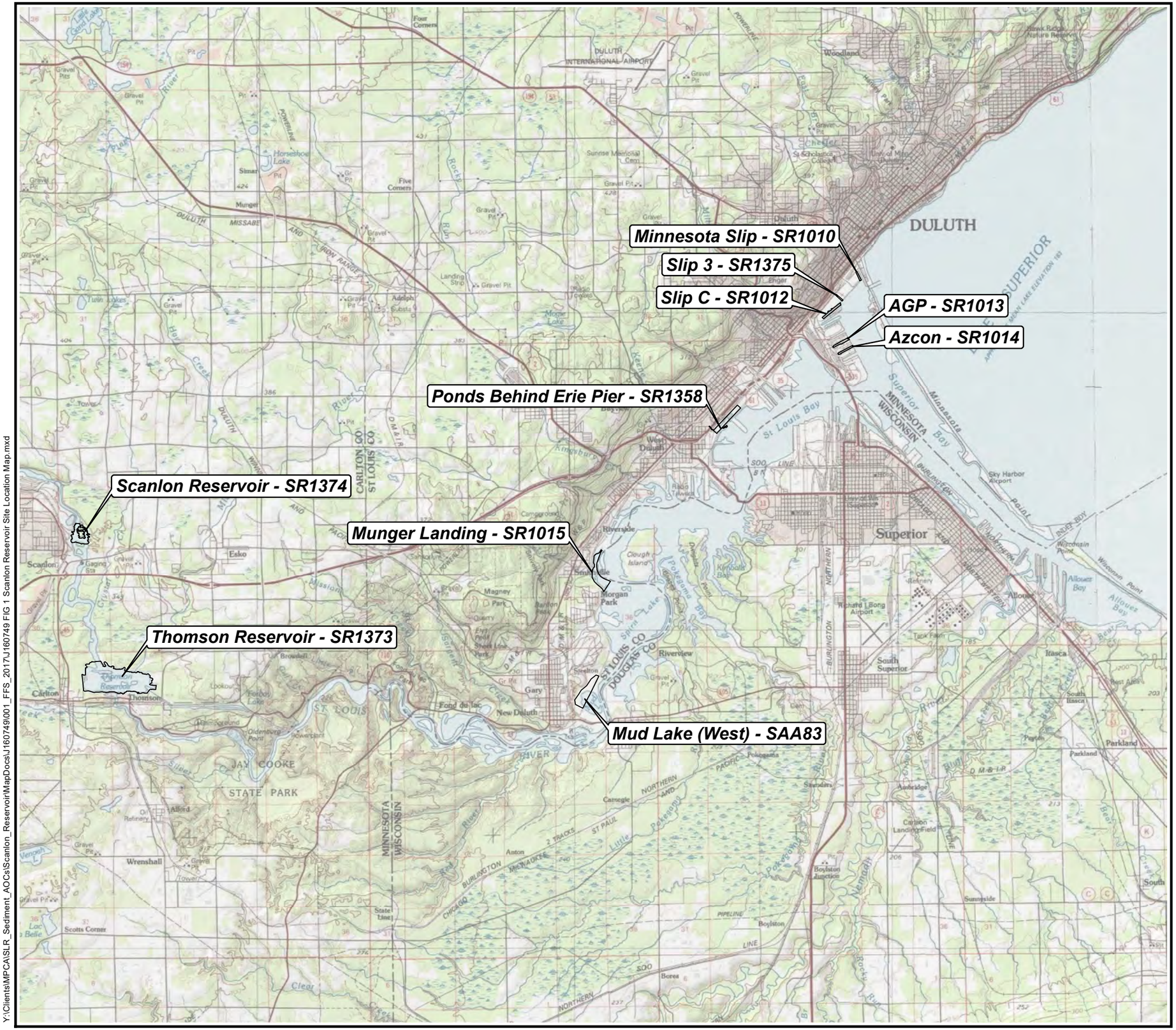
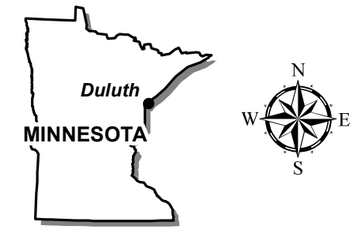


Figure 1

Site Location Map

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: National Geographic Society, i-cubed

0 2 4 Miles

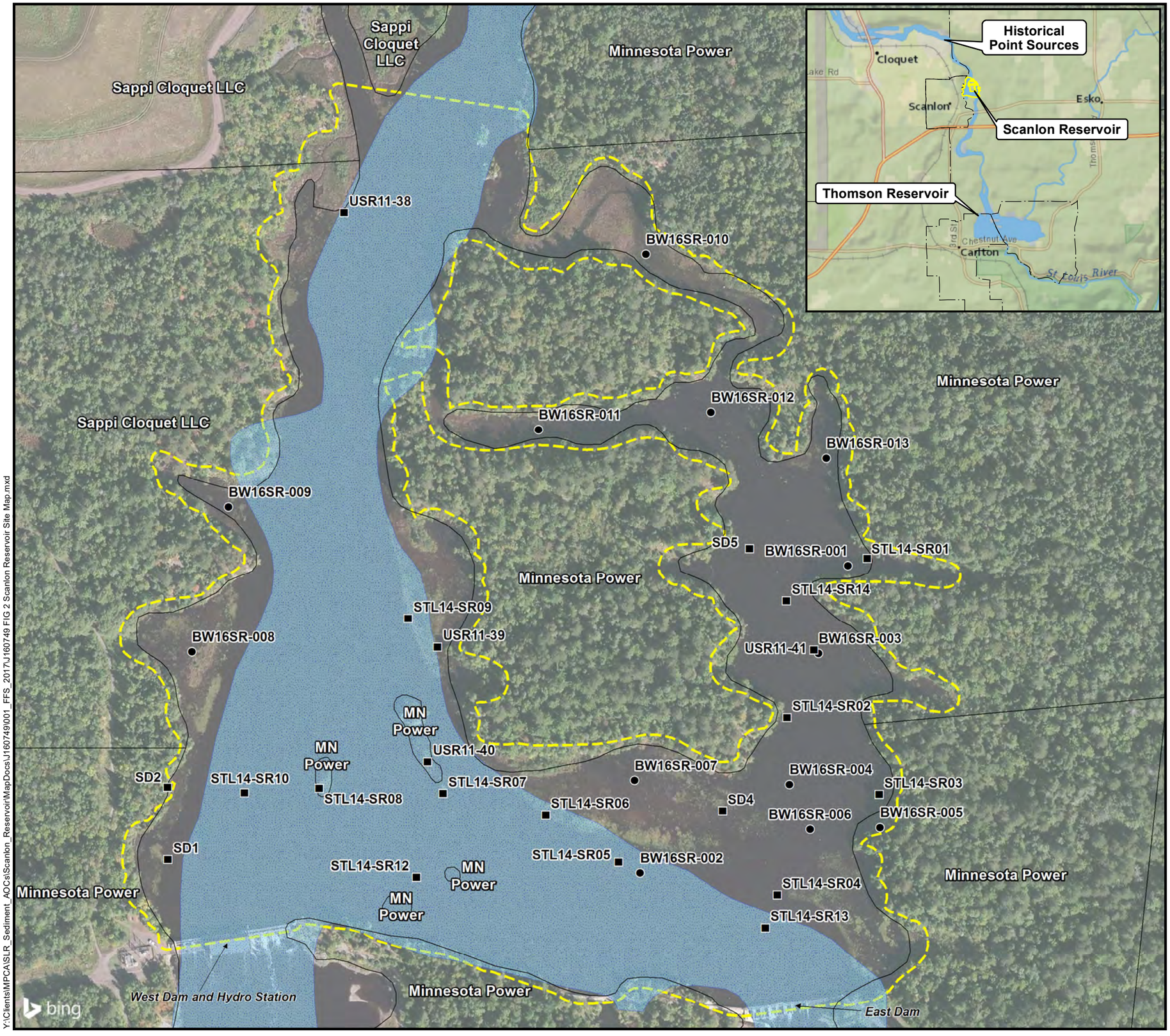
0 3 6 Kilometers

CLEAN WATER LAND & LEGACY AMENDMENT

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised:5/15/2017 Project No.J160749

Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 1 Scanlon Reservoir Site Location Map.mxd



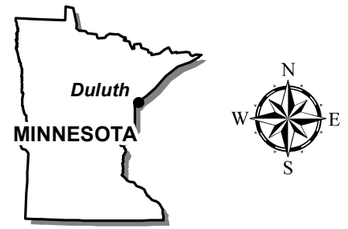
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 2 Scanlon Reservoir Site Map.mxd

Figure 2

Site Map

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

- Sediment Sample Locations (Bay West 2016)
- Historical Sediment Sample Locations (2011/2014)
- Scanlon Reservoir Site Boundary
- City Boundary
- Parcel Boundary (With Property Owners)
- Historical Stream Area (Carlton County Map, 1948)

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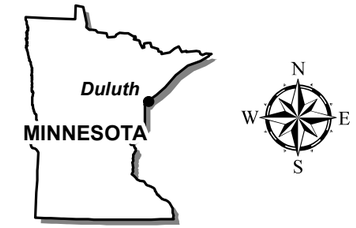
Figure 3

Bathymetry

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

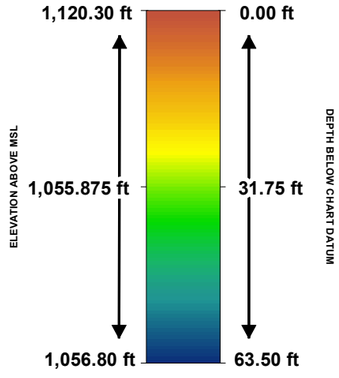
0 75 150 Meters

Bathymetry Contour Line (5ft Intervals)

Scanlon Reservoir Site Boundary

NOTE: Water elevation maintained at 1119.80 ft +/- 0.5 ft

Water Depth



(Based from MNDNR Lake Bathymetric Contours, 1996)

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Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749001_FFS_2017\J160749 FIG 3 Scanlon Reservoir Bathymetry.mxd

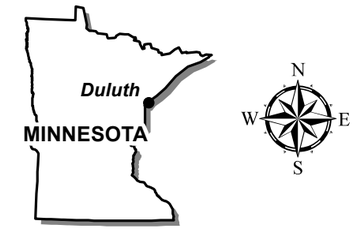
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\U160749\001_FFS_2017\U160749 FIG 4 Scanlon Reservoir TEQ KM Fish SQT Results.mxd

Figure 4

TEQ KM Fish SQT Results

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Sample Type

- △ 2016 Sediment Sample, Including In-Situ Tissue
- ⛒ 2016 Sediment Sample and Lab Bioaccumulation Testing
- ○ 2016 Sediment Sample
- □ Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Bay West
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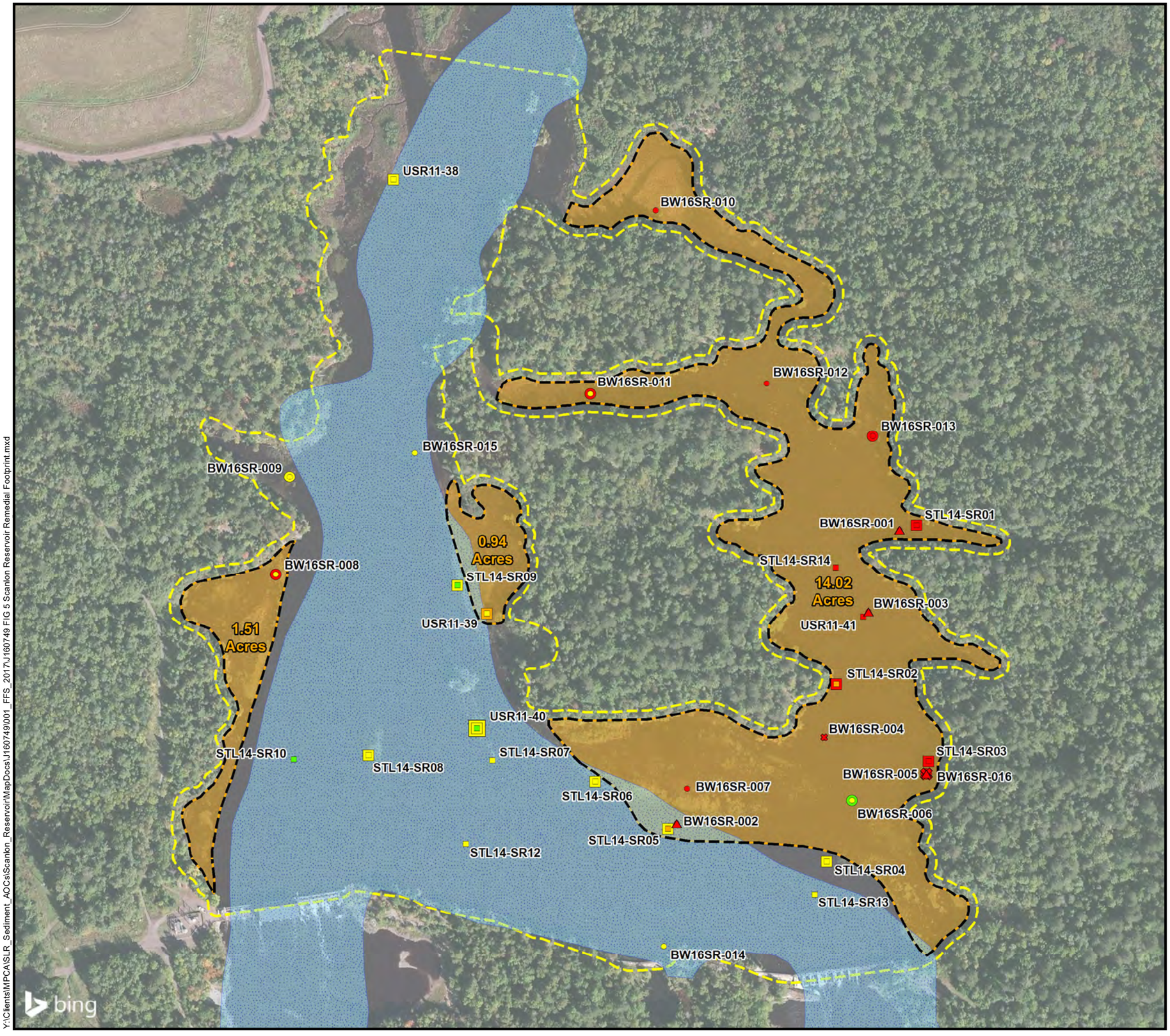
Drawn By: S.G. Date Drawn/Revised:5/31/2017 Project No.J160749



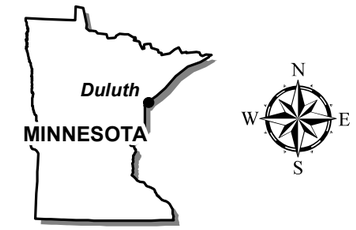
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749001_FFS_2017\J160749 FIG 5 Scanlon Reservoir Remedial Footprint.mxd

Figure 5

Remedial Footprint

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Remedial Footprint (16.47 Acres)

Historical Stream Area
(Carlton County Map, 1948)

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Dioxins/Mercury SQT Exceedance Areas

- Estimated Area Exceeding Midpoint SQT (16.47 Acres)

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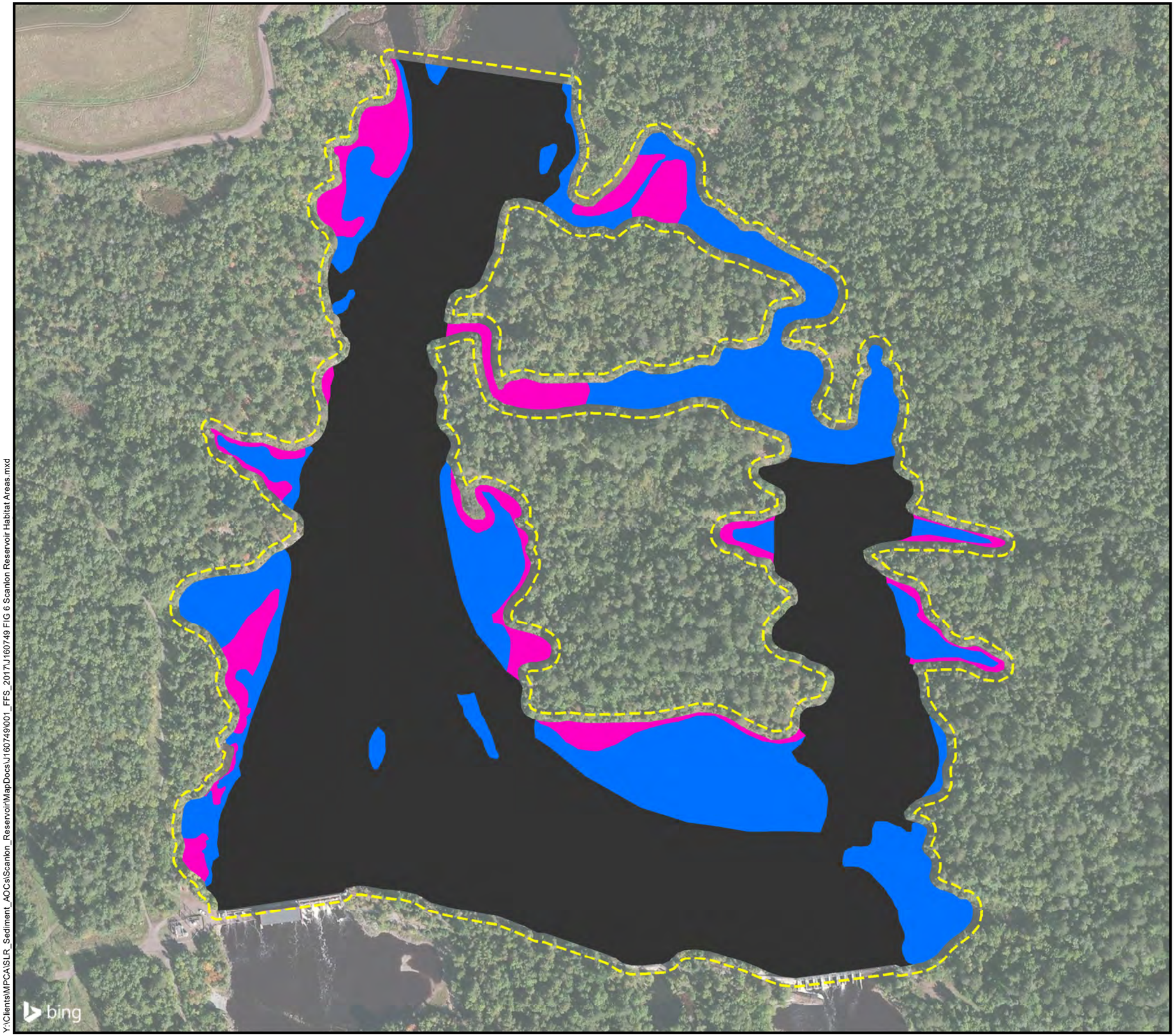
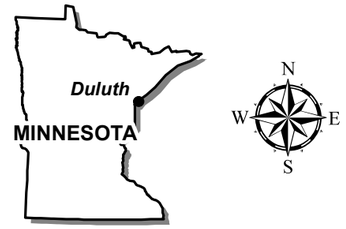


Figure 6

Habitat Areas

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

- Scanlon Reservoir Site Boundary
- Backshore/Foreshore Habitat Zone (3.18 Acres)
- Emergent Aquatic Vegetation Habitat Zone (10.45 Acres)
- Submerged Aquatic Vegetation and Deep Water Habitat Zone (30.25 Acres)

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Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749001_FFS_2017\J160749 FIG 6 Scanlon Reservoir Habitat Areas.mxd

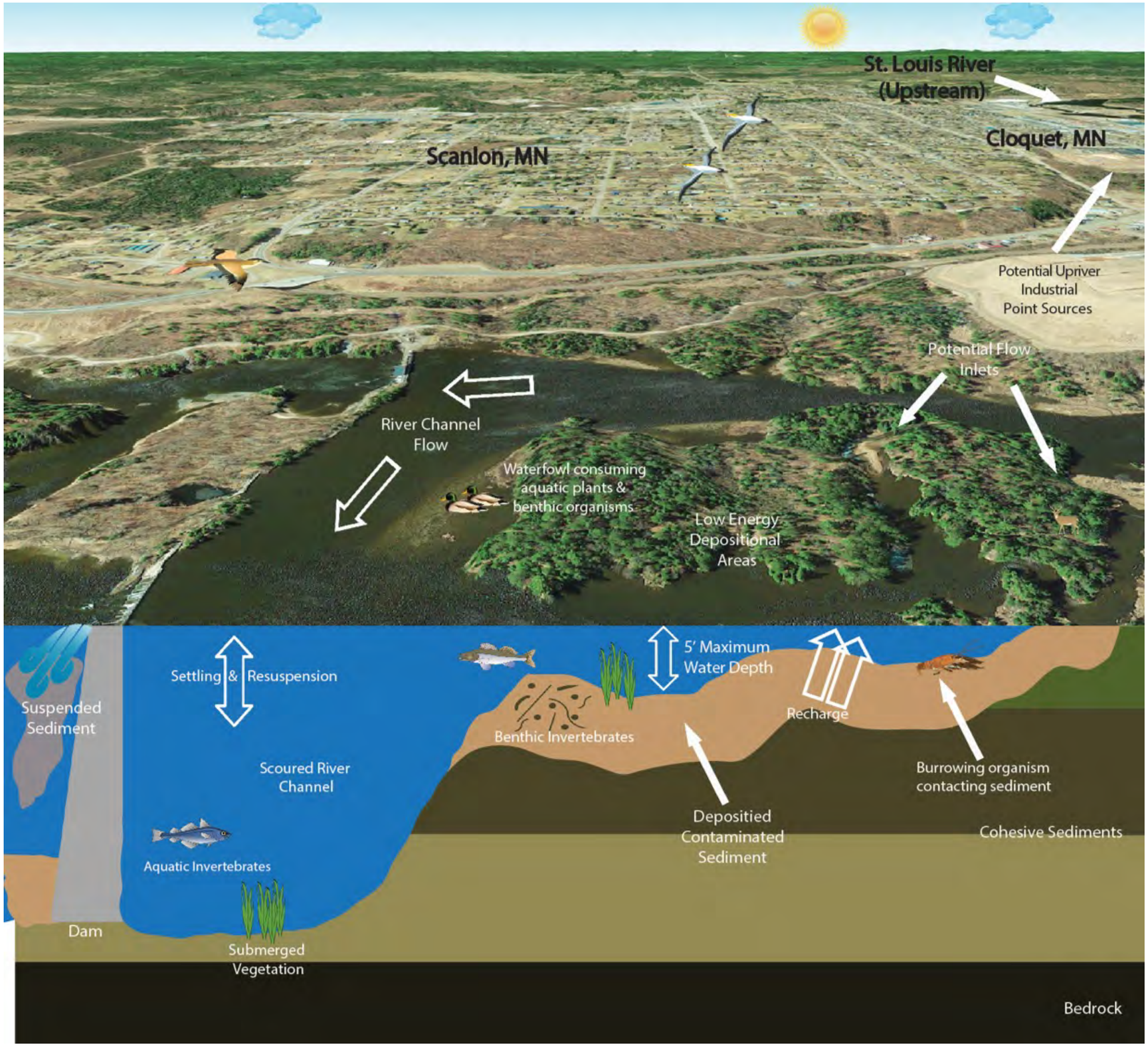
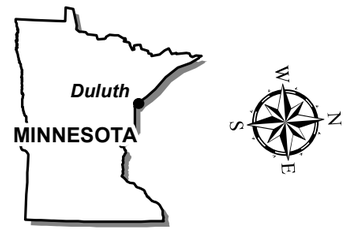


Figure 7

Conceptual Site Model

Scanlon Reservoir
SLR Sediment AOCs
Scanlon, MN



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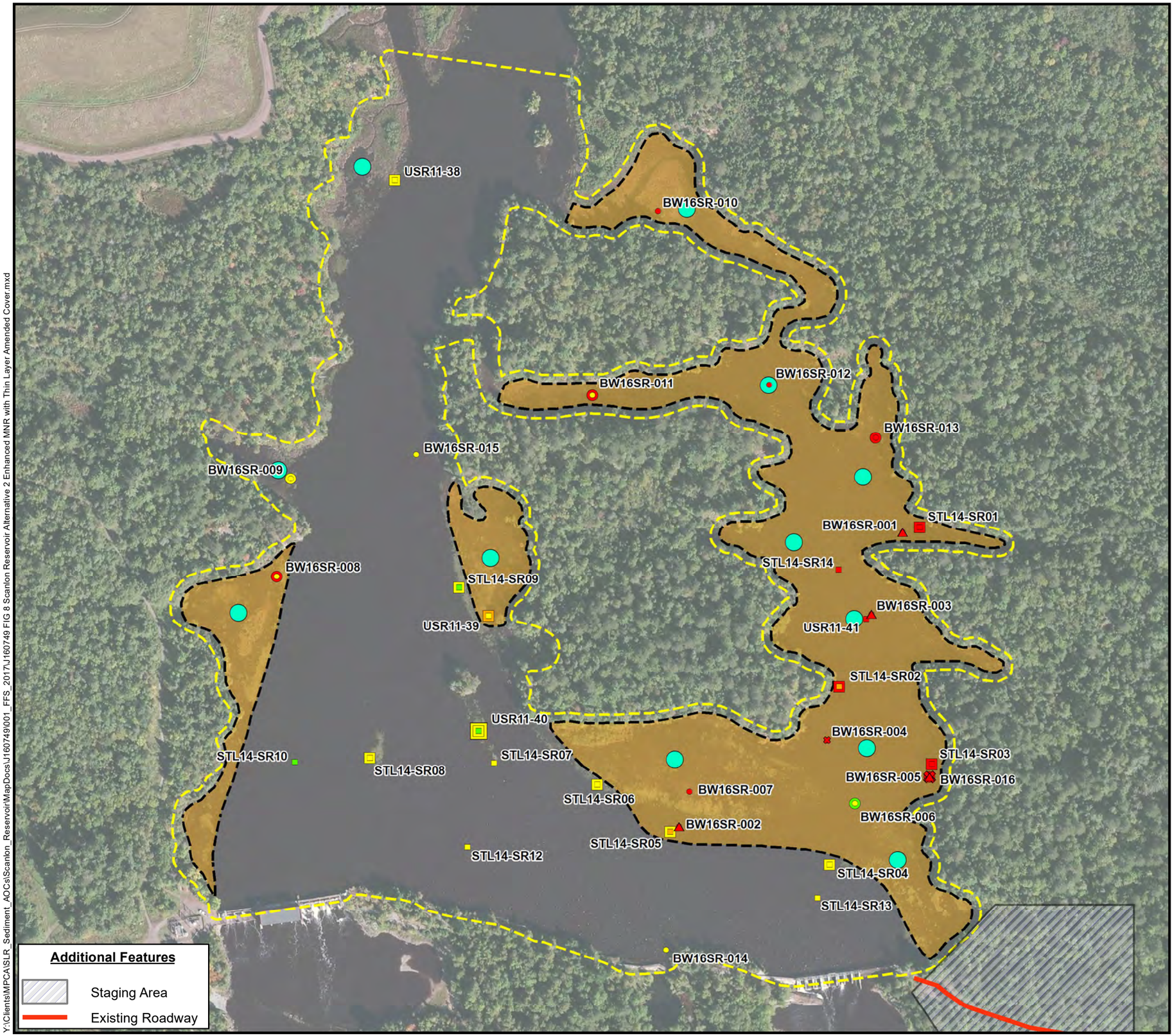
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 7 Scanlon Reservoir Conceptual Site Model.mxd

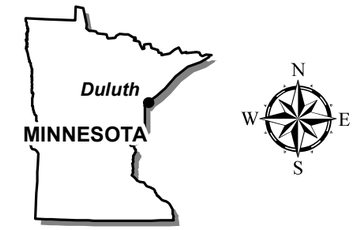


Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 8 Scanlon Reservoir Alternative 2 Enhanced MNR with Thin Layer Amended Cover.mxd

Figure 8

Alternative 2 - Enhanced MNR with Thin-Layer Amended Cover

Scanlon Reservoir
SLR Sediment AOCs
Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Proposed Sediment Monitoring Locations

Scanlon Reservoir Site Boundary

Targeted Thin-Layer Cover Areas (16.47 Acres)

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Dioxins/Mercury SQT Exceedance Areas

- Estimated Area Exceeding Midpoint SQT (16.47 Acres)

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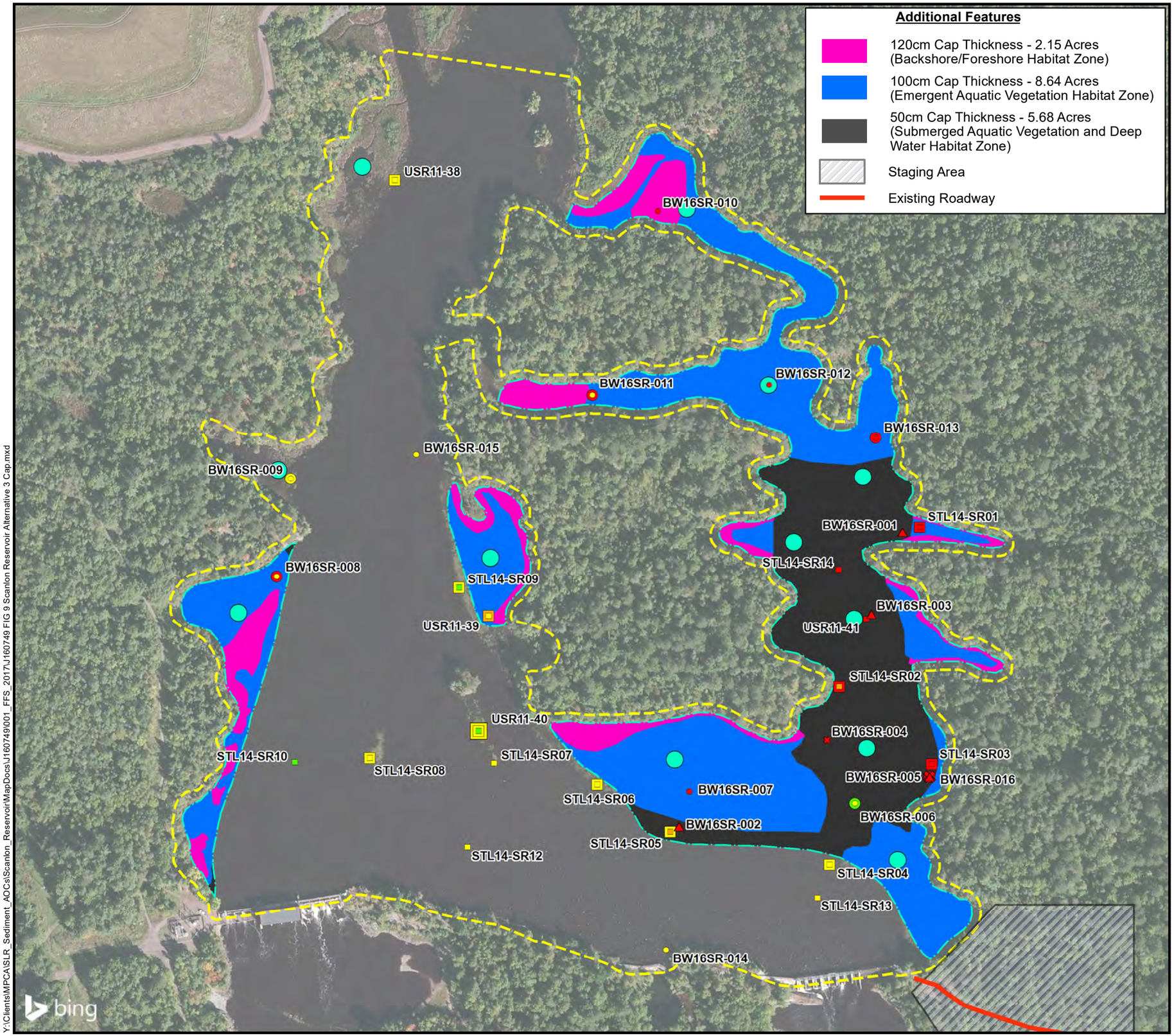


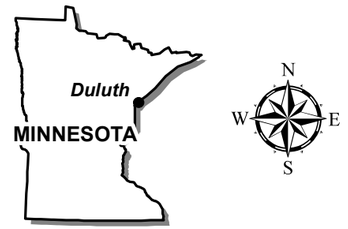
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 9 Scanlon Reservoir Alternative 3 Cap.mxd

Figure 9

Alternative 3 - Cap

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Proposed Sediment Monitoring Locations

Scanlon Reservoir Site Boundary

Targeted BAZ Cap Areas (16.47 Acres)

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

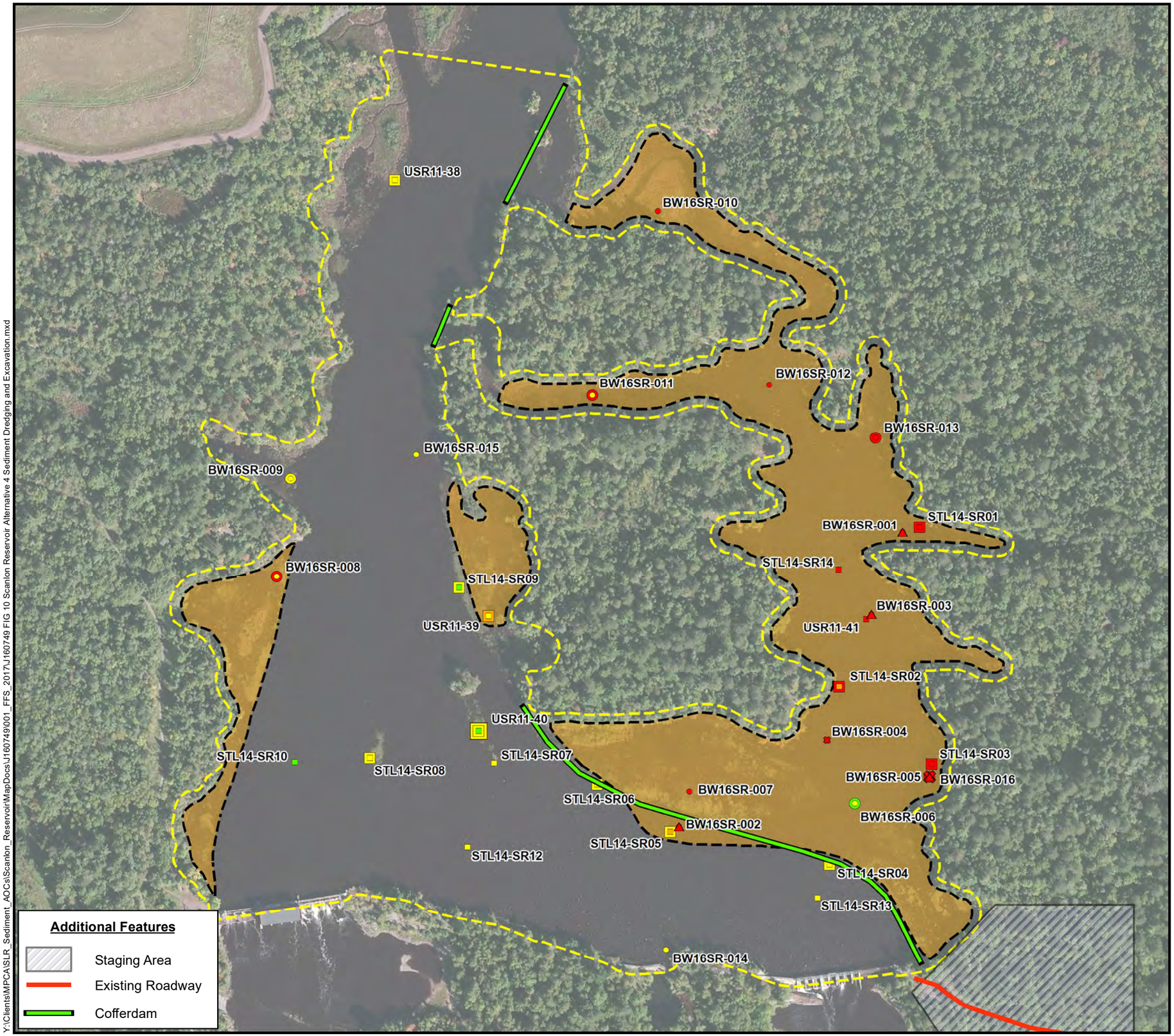
- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:5/16/2017 Project No.J160749



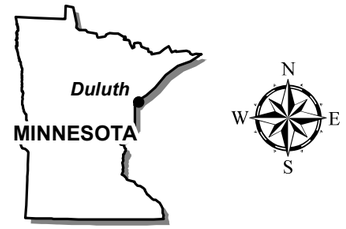
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 10 Scanlon Reservoir Alternative 4 Sediment Dredging and Excavation.mxd

Figure 10

Alternative 4 - Sediment Dredging and Excavation

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Targeted Excavation Areas ("In the Dry") (16.47 Acres)

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Dioxins/Mercury SQT Exceedance Areas

Estimated Area Exceeding Midpoint SQT (16.47 Acres)

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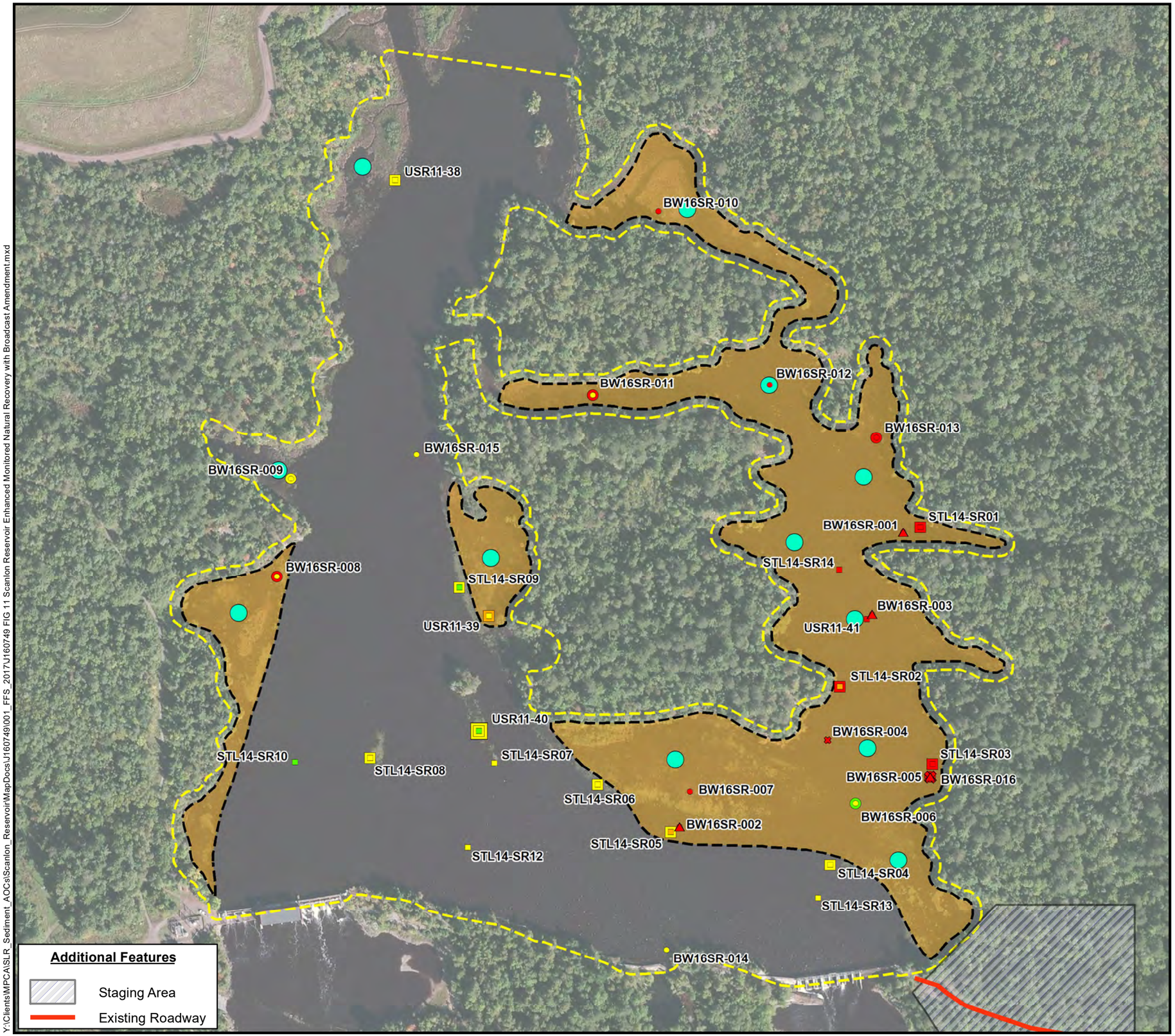
Drawn By: S.G. Date Drawn/Revised:5/16/2017 Project No.J160749



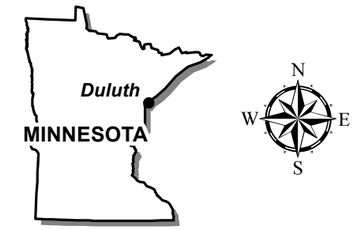
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\001_FFS_2017\J160749 FIG 11 Scanlon Reservoir Enhanced Monitored Natural Recovery with Broadcast Amendment.mxd

Figure 11

Enhanced Monitored Natural Recovery with Broadcast Amendment

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Proposed Sediment Monitoring Locations

Scanlon Reservoir Site Boundary

Targeted Thin-Layer Cover Areas (16.47 Acres)

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Dioxins/Mercury SQT Exceedance Areas

- Estimated Area Exceeding Midpoint SQT (16.47 Acres)

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:5/31/2017 Project No.J160749

Tables

Table 1
Statistics for Select Parameters of 2011 and 2014 Samples
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

All Intervals																	
Statistic	**Units**	**Midpoint SQT**	**SSV**	Number of Results	Mean	Median	Standard Deviation	Range	Minimum	Maximum	Level 1 Exceedances	Level 1 Percent	Midpoint Exceedances	Midpoint Percent	Level 2 Exceedance	Level 2 Percent	SSV Exceedances	SSV Percent
Total PAHs	**µg/kg**	**12300**	**NE**	23	662.12	414.00	819.51	4017.00	163.00	4180.00	1	4%	0	0%	0	0%	NE	NE
PCB	**µg/kg**	**370**	**5**	27	63.81	0.00	92.25	340.00	0.00	340.00	9	33%	0	0%	0	0%	11	41%
Cadmium	**mg/kg**	**3.0**	**10**	27	0.65	0.56	0.30	1.41	0.29	1.70	3	11%	0	0%	0	0%	0	0%
Copper	**mg/kg**	**91**	**NE**	27	16.19	16.00	7.95	38.60	5.10	43.70	1	4%	0	0%	0	0%	NE	NE
Mercury	**mg/kg**	**0.64**	**0.02**	59	0.25	0.10	0.39	1.65	0.01	1.66	17	29%	7	12%	5	8%	56	95%
Nickel	**mg/kg**	**36**	**NE**	27	16.33	17.00	4.98	19.90	8.10	28.00	3	11%	0	0%	0	0%	NE	NE
Zinc	**mg/kg**	**290**	**NE**	27	74.69	70.00	30.85	127.00	35.00	162.00	3	11%	0	0%	0	0%	NE	NE
D/Fs	**ng/kg**	**11.2**	**0.02**	48	37.74	9.63	66.72	339.77	0.19	339.96	44	92%	23	48%	20	42%	48	100%
0-15																		
Statistic	**Units**	**Midpoint SQT**	**SSV**	Number of Results	Mean	Median	Standard Deviation	Range	Minimum	Maximum	Level 1 Exceedances	Level 1 Percent	Midpoint Exceedances	Midpoint Percent	Level 2 Exceedance	Level 2 Percent	SSV Exceedances	SSV Percent
Total PAHs	**µg/kg**	**12300**	**NE**	15	347.0	299.0	154.6	535.0	163.0	698.0	0	0%	0	0%	0	0%	NE	NE
PCB	**µg/kg**	**370**	**5**	16	31.9	0.0	57.8	150.0	0.0	150.0	4	25%	0	0%	0	0%	4	25%
Cadmium	**mg/kg**	**3.0**	**10**	16	0.6	0.5	0.4	1.4	0.3	1.7	2	13%	0	0%	0	0%	0	0%
Copper	**mg/kg**	**91**	**NE**	16	14.2	11.5	9.0	38.6	5.1	43.7	1	6%	0	0%	0	0%	NE	NE
Mercury	**mg/kg**	**0.64**	**0.02**	42	0.3	0.1	0.4	1.6	0.0	1.7	10	24%	6	14%	4	10%	40	95%
Nickel	**mg/kg**	**36**	**NE**	16	16.0	14.5	5.3	19.9	8.1	28.0	2	13%	0	0%	0	0%	NE	NE
Zinc	**mg/kg**	**290**	**NE**	16	66.6	56.5	26.1	99.0	35.0	134.0	1	6%	0	0%	0	0%	NE	NE
D/Fs	**ng/kg**	**11.2**	**0.02**	32	31.2	9.6	55.8	31.8	0.2	292.9	29	91%	15	47%	13	41%	32	100%
15-50																		
Statistic	**Units**	**Midpoint SQT**	**SSV**	Number of Results	Mean	Median	Standard Deviation	Range	Minimum	Maximum	Level 1 Exceedances	Level 1 Percent	Midpoint Exceedances	Midpoint Percent	Level 2 Exceedance	Level 2 Percent	SSV Exceedances	SSV Percent
Total PAHs	**µg/kg**	**12300**	**NE**	9	1191.36	885.00	1178.07	3872.00	308.00	4180.00	1	11%	0	0%	0	0%	NE	NE
PCB	**µg/kg**	**370**	**5**	12	101.08	51.50	117.10	340.00	0.00	340.00	5	42%	0	0%	0	0%	7	58%
Cadmium	**mg/kg**	**3.0**	**10**	12	0.77	0.66	0.34	1.25	0.45	1.70	2	17%	0	0%	0	0%	0	0%
Copper	**mg/kg**	**91**	**NE**	12	21.13	18.90	9.04	35.60	8.10	43.70	1	8%	0	0%	0	0%	NE	NE
Mercury	**mg/kg**	**0.64**	**0.02**	24	0.33	0.17	0.38	1.38	0.02	1.40	12	50%	4	17%	3	13%	23	96%
Nickel	**mg/kg**	**36**	**NE**	12	17.11	18.00	4.83	16.30	9.50	25.80	1	8%	0	0%	0	0%	NE	NE
Zinc	**mg/kg**	**290**	**NE**	12	90.42	83.50	36.92	122.00	40.00	162.00	3	25%	0	0%	0	0%	NE	NE
D/Fs	**ng/kg**	**11.2**	**0.02**	20	67.10	32.73	95.53	339.69	0.27	339.96	19	95%	12	60%	11	55%	20	100%

Table 1
Statistics for Select Parameters of 2011 and 2014 Samples
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

50-100																	
Statistic	**Units**	**Midpoint SQT**	**SSV**	Number of Results	Mean	Median	Standard Deviation	Range	Minimum	Maximum	Level 1 Exceedances	Level 1 Percent	Midpoint Exceedances	Midpoint Percent	Level 2 Exceedance	Level 2 Percent	SSV Exceedances	SSV Percent
Total PAHs	**µg/kg**	**12300**	**NE**	1	308.00	308.00	0.00	0.00	308.00	308.00	0	0%	0	0%	0	0%	NE	NE
PCB	**µg/kg**	**370**	**5**	1	340.00	340.00	0.00	0.00	340.00	340.00	1	100%	0	0%	0	0%	1	100%
Cadmium	**mg/kg**	**3.0**	**10**	1	0.83	0.83	0.00	0.00	0.83	0.83	0	0%	0	0%	0	0%	0	0%
Copper	**mg/kg**	**91**	**NE**	1	18.80	18.80	0.00	0.00	18.80	18.80	0	0%	0	0%	0	0%	NE	NE
Mercury	**mg/kg**	**0.64**	**0.02**	1	0.45	0.45	0.00	0.00	0.45	0.45	1	100%	0	0%	0	0%	1	100%
Nickel	**mg/kg**	**36**	**NE**	1	19.40	19.40	0.00	0.00	19.40	19.40	0	0%	0	0%	0	0%	NE	NE
Zinc	**mg/kg**	**290**	**NE**	1	122.00	122.00	0.00	0.00	122.00	122.00	1	100%	0	0%	0	0%	NE	NE
D/Fs	**ng/kg**	**11.2**	**0.02**	1	1.55	1.55	0.00	0.00	1.55	1.55	1	100%	0	0%	0	0%	1	100%
100+																		
Statistic	**Units**	**Midpoint SQT**	**SSV**	Number of Results	Mean	Median	Standard Deviation	Range	Minimum	Maximum	Level 1 Exceedances	Level 1 Percent	Midpoint Exceedances	Midpoint Percent	Level 2 Exceedance	Level 2 Percent	SSV Exceedances	SSV Percent
Total PAHs	**µg/kg**	**12300**	**NE**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	NE	NE
PCB	**µg/kg**	**370**	**5**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
Cadmium	**mg/kg**	**3.0**	**10**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
Copper	**mg/kg**	**91**	**NE**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
Mercury	**mg/kg**	**0.64**	**0.02**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
Nickel	**mg/kg**	**36**	**NE**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
Zinc	**mg/kg**	**290**	**NE**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%
D/Fs	**ng/kg**	**11.2**	**0.02**	0	0.00	0.00	0.00	0.00	0.00	0.00	0	0%	0	0%	0	0%	0	0%

D/Fs = polychlorinated dibenzo-p-dioxins/polychlorinated dibenzofurans

µg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

ng/kg = nanograms per kilogram

PCB = polychlorinated biphenyl

Table 2
Contaminants of Concern Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Contaminant	Units	Cleanup Level	Maximum Concentration Detected	Mean Concentration
Dioxins	ng TEQ/kg	11.2	392.7	32.58

mg/kg - milligrams per kilogram
ng TEQ/kg – nanograms toxic equivalency per kilogram

Table 3
Technologies Screening Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Category	Technology	Description	Applicability	Ranking						Retained for Consideration	Rationale
				Effectiveness		Implementability		Relative Cost			
Institutional Controls	Institutional Controls	Institutional controls in the form of an environmental restrictive covenant or conditions of future permits may be used to prevent exposure and contact with impacted soil or sediment by restricting land uses or disturbances to the material.	May consist of fish consumption advisories, commercial fishing bans, waterway use restrictions, or deed restrictions	○	Effective in meeting RAOs when combined with other remedies.	●	Easily implemented with little disruption to the Site.	$	Minimal but there are long term costs associated with initiating and maintaining institutional controls.	Yes.	Some institutional controls already in place; however, additional controls are expected to be a required component of any remedy.
Monitoring and Evaluation	Monitoring	The collection and analysis of chemical, physical, and/or biological data over a sufficient period of time and frequency to determine the status and/or trend in one or more environmental parameters or characteristics.	Monitoring should be conducted to asses compliance with design and performance standards; to assess short-term remedy performance and effectiveness in meeting sediment cleanup levels; and/or to evaluate long-term remedy effectiveness in achieving RAOs and in reducing human health and/or environmental risk.	○	Effective in meeting RAOs when combined with other remedies.	●	Highly implementable with no disturbance to the Site.	$	The main cost is associated with laboratory analysis.	Yes.	Monitoring is expected to be a required component of any remedy.
Natural Recovery	Monitored Natural Recovery	MNR leaves impacted sediment in place and relies on ongoing, naturally occurring processes to isolate, destroy, or reduce exposure or toxicity of impacted sediment.	Burial of contaminated sediments may be occurring at the Site but deposition rates are unknown and may not be sufficient to isolate COCs in a reasonable timeframe. The rate of contaminant degradation via natural processes at the Site is unknown.	○	Burial may be occurring at certain areas of the Site; additional information is required to determine the effectiveness of MNR.	●	Highly implementable with no disturbance to the Site.	$	The main cost of NR is associated with monitoring.	No.	Effectiveness at the Site has not been demonstrated and does not appear to be effective under current conditions.
	Enhanced Monitored Natural Recovery	EMNR adds amendments to the sediment to accelerate physical isolation process and facilitates re-establishment of benthic or plant habitat. May include a granular or carbon sorbent cover (over sediments) or biological stimulants (to soil).	Use of an amendment may increase the rate at which sediment contaminant concentrations are reduced/made less available over time. Natural bioturbation processes will assist in mixing amendments into in-situ sediments.	◉	Sediment amendments have been used successfully in the past to reduce the availability of contaminants to biota.	◉	Implementable; however, requires site access, staging area, and placement equipment. Maintenance may be required depending on hydrologic conditions.	$$	Greater initial cost than MNR due to thin cover or amendment placement, but less expensive than conventional cap or sediment removal.	Yes.	Effectiveness of chemical contaminant sequestration in sediments via addition of amendments has been demonstrated. Additional information is required to determine potential of long-term burial with clean sediments.
Capping	Capping	Capping provides a physical barrier and chemical isolation from COCs. Caps may be constructed from clean sediment, sand, gravel, geotextiles, liners, reactive or absorptive material and may consist of multiple layers. Granular sediment caps can provide erosion protection and limit bioturbation.	Cap thickness depends on bioactive zone (BAZ) thickness requirements, which vary by habitat, substrate and water depth. A cap may alter hydrologic conditions and Site use.	●	Highly effective and proven technology. COCs have low solubility and mobility. Armoring potentially required in areas of scour.	◉	Implementable; however, requires site access, staging area, and placement equipment. Maintenance may be required depending on hydrologic conditions.	$$$	Capping costs are generally less than sediment removal, and depend on cap thickness, material, lateral extent and surface water engineering factors. Material costs for a synthetic cap are generally higher than a granular cap.	Yes.	Proven and effective method to control exposure to contaminants.

Table 3
Technologies Screening Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Category	Technology	Description	Applicability	Ranking						Retained for Consideration	Rationale
				Effectiveness		Implementability		Relative Cost			
Excavation and Removal	Mechanical Dredging	Sediment is lifted to the surface using a mechanical excavator or crane and placed on a barge for transport. Removed sediment has a similar moisture content as the in situ material, requiring dewatering prior to disposal. Residual cover is typically needed to manage remaining impacts.	Mechanical dredging may be inhibited if slanted slate bedrock underlying sediments is encountered. Sediment resuspension controls expected to be needed.	●	Highly effective and proven technology; however, resuspension may limit effectiveness.	◉	Requires dredging equipment and upland staging infrastructure for sediment dewatering and transportation. Less staging space required than hydraulic dredging.	$$$	Main capital costs include equipment mobilization, staging area development, equipment operation, residual cover materials, and construction and operation of a containment area for dredged material.	**Yes.**	Suitable for use within some areas of the Site.
	Hydraulic Dredging	Hydraulic dredging captures water with the sediment and removes it by pumping the sediment slurry typically through a pipeline to the dewatering location or final disposal site. High water content of slurry requires significant dewatering. Residual cover is typically needed to manage remaining impacts.	Hydraulic dredging is implementable at the Site, but would require a large area for sediment dewatering.	●	Highly effective and proven technology; however, resuspension may limit effectiveness.	○	Implementable; however, requires large staging area for dewatering equipment, requires more water treatment than mechanical dredging.	$$$$	Additional treatment and disposal costs due to greater water content of the slurried sediment.	**No**	Not suitable for use at the Site when compared to mechanical dredging or excavation removal.
	Mechanical Removal in Dry Conditions	Water is diverted or drained from the excavation area using a containment barrier such as a cofferdam to allow for excavation of dry sediment with conventional equipment (e.g. backhoe). Typically limited to shallow areas.	Well suited for shallow areas and geometry that allows for construction of containment barrier and water diversion.	●	Effective and proven technology. Allows for visual inspection during removal. Minimal resuspension/redeposition. High degree of accuracy.	●	Site well suited for cofferdam construction and dewatering due to confined, low energy and shallow remedial areas.	$$$	Costs are similar to mechanical dredging, with the added cost to construct diversion or containment structures.	**Yes.**	Anticipated to be more cost effective than mechanical and hydraulic dredging and to be compatible with Site geometry.
Disposal	Off-Site	Removed sediment is transported to an offsite disposal location that will accept the waste. Dewatering of sediments is generally required before transport.	Transportation of large volumes of sediment would create significant truck traffic through the surrounding community for a long duration.	●	Effective at meeting RAOs, low risk of spills during transportation.	◉	Disruption to neighbors during trucking, may result in limited work hours. Seasonal restrictions may also apply.	$$$$	Costs for offsite disposal include dewatering, water treatment, loading and transportation costs and landfill disposal fees. Transportation costs depend on distance to the landfill.	**Yes.**	Suitable with proper truck routing. Onsite storage facilities are not available.
	Confined Disposal Facility (CDF)	CDFs are engineered structures enclosed by dikes and specifically designed to contain sediment. CDFs may be located either upland (above the water table), near-shore (partially in the water), or completely in the water (island CDFs).	Creation of a CDF would result in destruction of wetland areas.	●	Most widely used method for disposal and has been demonstrated effective.	○	Requires high level of design, detailed knowledge of dredge plans, requires large permanent area for construction, and treatment of discharge.	$$$	Costs for a CDF include engineering and design costs, materials for dikes and suspended solids control, and construction equipment and labor.	**No**	Surrounding lands are privately owned and thus consolidation areas assumed infeasible.
	On-site Contained Aquatic Disposal (CAD)	Dredged or excavated sediment is disposed within a natural or excavated depression elsewhere in the water body.	A suitable low-energy location to accommodate entire sediment volume is not available.	○	Is effective for dioxins. Stabilization of sediments reduces erosion potential. May result in poor environment for benthic community.	⊗	A suitable location to accommodate entire sediment volume is not available.	$$$	Specialized equipment for a CAD may be required, especially if the disposal site is in deep water. Dredging to create a CAD would add cost.	**No**	Based on the Site characteristics, a suitable location is not available at the Site to accommodate the required disposal volume.

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Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Category	Technology	Description	Applicability	Ranking						Retained for Consideration	Rationale
				Effectiveness		Implementability		Relative Cost			
In Situ Treatment	Immobilization	Immobilization treatments add chemicals or cements to reduce the leachability of COCs. Mechanisms include solidification (encapsulation) or stabilization (chemical or absorptive reactions that convert COCs to less toxic or mobile forms).	Implementation at a sediment site is difficult, due to submerged work requirement and concerns with interfering with reservoir/dam operations.	○	Is effective for COCs. Stabilization of sediments reduces erosion potential. May result in poor environment for benthic community.	⊗	Sediment mixing can be difficult. May require dewatering. Requires equipment for mixing. Solidified sediment would prevent reservoir maintenance dredging.	$$$	Costs for solidification or stabilization affected by the quantity and type of reagents added to the waste and the need for specialized equipment for mixing reagents with sediment.	**No**	Not proven to be effective for sediments. Costly and more difficult to implement than other technologies.
	Enhanced Bioremediation	Microbial degradation by bacteria or fungi is enhanced by adding materials such as oxygen, nitrate, sulfate, hydrogen, nutrients, or microorganisms to the sediment.	Can be effective for dioxins.	○	Requires specific geochemical parameters to be successful (temperature, Ph, nutrient availability)	○	Easily implemented with little disruption to the Site.	$$$	Costs of enhanced bioremediation are relatively low, but several treatments and monitoring similar to MNR may be required.	**No**	Difficult to implement sub aqueously.
	Oxidation/Reduction	Chemicals are injected into sediment to act as an oxidant/electron acceptor to facilitate aerobic decomposition of organic matter.	chemical addition may create toxic conditions.	○	Chemical addition may create toxic conditions.	○	Bench-scale testing and pilot-scale testing required to determine the type, concentration, and quantity of oxidant and amendments required.	$$$	Costs include bench- or pilot-scale tests. Monitoring may be required.	**No**	Not proven safe for subaqueous conditions.
	Chemical Oxidation	The addition of chemical oxidizers to sediment can cause the rapid and complete chemical destruction of many toxic organic chemicals.	Limited effectiveness for Site COCs.	⊗	Addition of chemicals may form temporarily toxic conditions for benthic or aquatic organisms	◉	Pilot studies would be required to determine the effectiveness of specific oxidants for COCs.	$$$	Costs include bench- or pilot-scale tests to determine effectiveness, oxidants for injection, and a delivery system. Monitoring may also be required.	**No**	Limited effectiveness. Chemical addition may create toxic conditions.
	Phytoremediation	Phytoremediation uses plant species to remove, transfer, stabilize, and destroy COCs in soil and sediment. Generally limited to sediments in shallow water zones and low concentrations.	Habitat restoration not likely necessary, technology not effective in open water areas of Site.	○	Effective in shallow contaminated areas, which comprise only a portion of the Site.	●	Implementation involves planting and in some cases harvesting with little disruption to the Site.	$$	Primary costs are purchasing and planting applicable species. Monitoring may also be required.	**No**	May be implemented for habitat restoration, but not effective alone.
	Adsorption	Adsorbents can be used as sediment amendments for in situ treatment of COCs. Sorption of inorganics and organics can take place simultaneously with a suitable combination of sorbents.	May be useful as EMNR amendment.	◉	Sorption of COCs possible with amendment materials.	◉	Amendments can be delivered to the sediment in the form of pellets or mixed into other media (i.e., sand) to resist re-suspension.	$$	The main costs include the adsorbent material, and a method for depositing it on the surface sediment. Monitoring may also be required.	**Yes.**	Effectiveness of chemical contaminant sequestration in sediments via addition of amendments has been demonstrated. Can be used as part of an EMNR approach.

Table 3
Technologies Screening Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Category	Technology	Description	Applicability	Ranking						Retained for Consideration	Rationale
				Effectiveness		Implementability		Relative Cost			
Dewatering	Passive Dewatering	Passive dewatering relies on natural evaporation and drainage to remove moisture from the sediment. Drainage may be driven by gravity or assisted with a vacuum pump. Passive dewatering may occur in CDFs, lagoons, tanks, or temporary holding/rehandling facilities.	Upland areas need to be developed at the Site to serve as a staging area and passive dewatering area.	○	Passively dewatered sediments may not have low enough water content for landfill disposal, so supplemental technologies may be required.	◉	Implementable if adjacent staging area can be located. Time frames for passive dewatering likely longer than for mechanical dewatering.	$$	Costs to consider include construction of a dewatering facility or adequately sized CDF.	Yes.	Appropriate for off-site disposal when used with amendment addition and/or sufficient dewatering timeframe.
	Sediment Reworking	Reworking sediments to promote drainage, and mixing sediments with excavation equipment can enhance passive dewatering.	If a CDF is constructed, sediment reworking could be performed within the CDF.	◉	Sediment mixing and reworking would facilitate a timelier and more complete dewatering, but may not be sufficient for off-site disposal.	◉	Mixing and reworking sediments would decrease time needed to dewater with passive methods. Reworking and mixing could be done with standard excavation equipment already required for the project.	$$	Cost savings are expected over passive dewatering alone due to time saved.	Yes.	Appropriate for use with amendment addition (amendment must be worked into sediments through mixing).
	Hydroscopic Amendment Addition	Dredged sediments are mixed with amendments such as slags or cementitious materials to remove moisture and improve strength and stability.	Could be used to enhance dewatering in conjunction with passive dewatering	◉	Effectiveness of amendments depends on the moisture content of removed sediment. The majority of sediment removed from the Site will be excavated in the dry and thus have a low water content.	◉	Would require staging, mixing, and curing areas. Amendment addition creates a greater volume and mass, which needs to be considered in disposal options.	$$	Costs include amendment materials and mixing equipment. Costs increase with increased moisture content. Both the addition rate and the bulking factor of treated material should be considered when evaluating costs of amendment material.	Yes.	Is compatible with sediment reworking and passive dewatering.
	Geotextile Tube Dewatering	Sediment slurry from hydraulic dredging is pumped into the geotextile tube and filtered by the geotextile fabric. Sediment is retained within the geotextile tube, while free liquids pass through the exterior of the tube.	Not applicable to the planned methods of sediment removal at the Site (mechanical dredging with barge transport and excavation in the dry).	●	Proven technology and widely used for slurried dredge sediments.	○	Implementable if a nearby dewatering area can be located. Would require a substantially larger area for staging of sediments as compared to other dewatering methods.	$$$	Costs include flocculent and coagulant materials, cost of geotextile tubes and construction of staging area.	No	Not applicable to the planned methods of sediment removal at the Site (mechanical dredging with barge transport and excavation in the dry).
	Mechanical Dewatering	Mechanical dewatering technologies include use of plate filters, presses, centrifuges or other equipment to squeeze, press, or draw water from dredged sediment.	Requires homogeneous waste stream, which may not be achieved with mechanical removal and dry excavation.	○	Generally works best with a homogeneous waste stream produced via hydraulic dredging. Selection of specific mechanical dewatering equipment depends on treatment or disposal methods that follow.	○	Faster than passive dewatering and requires less space. Production rates depend on size and quality of the dewatering device and on the solids content of the input stream.	$$$$	Costs of mechanical dewatering are generally higher than passive dewatering due to the energy and equipment requirement.	No	Requires homogeneous waste stream, which may not be achieved with mechanical removal and dry excavation.
	Rapid Dewatering Systems	A system that continuously processes the slurry from a hydraulic dredge and separates solids into piles of debris; shells and gravel, sand, and fines. Includes polymer addition and flocculation, which may remove some COCs.	Not applicable to mechanically dredged waste streams or sediments excavated in the dry.	⊗	Highly effective and proven technology but typically utilized for large-scale and long-term dredging operations.	⊗	Faster than passive dewatering and requires less space. Production rates depend on size and quality of the dewatering device and on the solids content of the input stream.	$$$$	Costs of mechanical dewatering are generally higher than passive dewatering due to the energy and equipment requirement.	No	Not applicable to mechanically dredged waste streams or sediments excavated in the dry.

Table 3
Technologies Screening Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Category	Technology	Description	Applicability	Ranking						Retained for Consideration	Rationale
				Effectiveness		Implementability		Relative Cost			
Water Treatment	Filtration	Filters remove solids and sediments from wastewater, also removing absorbed COCs from the waste stream. Flocculants may be added to the waste stream to facilitate solids removal.	Filtration is a standard method for water treatment and would be effective at removing site COCs sorbed to suspended sediments in the waste stream.	○	Filters can be selected based on the required particulate size. Treatability study to determine if filtration is effective at reducing the COC concentration.	◉	Filtration is a widely used method for water treatment. Selection of the filtration methods and type requires engineering design and site specific knowledge of the waste stream. Would require a dewatering area.	$$$	Costs depend on change out frequency of filtration material.	Yes.	Effective for COC removal when used in combination with liquid adsorption.
	Liquid Adsorption	Involves pumping water through a vessel containing granular activated carbon (GAC), organoclay, or another adsorbent material; dissolved compounds to adsorb to its surface.	Conventional absorptive materials would remove dioxins.	◉	Activated carbon vessels are appropriate for treating dioxins. The presence of multiple constituents can impact the performance of activated carbon systems.	◉	Liquid adsorption systems are widely available, have a relatively small footprint, and require a relatively short timeframe for treatment.	$$$	Costs include media, vessels, and disposal/recycling costs for media. The adsorbent must be recharged or replaced periodically. Power is required for pumping.	Yes.	Effective for COC removal.
	Advanced Oxidation	Advanced oxidation uses UV light and the addition of strong oxidizers to primarily destroy organic constituents in water.	Advanced oxidation is applicable for treating most organics, including PAHs; however, it is not applicable to treatment of COCs.	○	Advanced oxidation is applicable for treating most organics, including dioxins.	◉	Advanced oxidation systems are widely available, have a relatively small footprint, and require a relatively short timeframe for treatment. Handling and storage of oxidizers would require special safety precautions.	$$$$	Costs may be higher because of energy requirements to power UV lights.	No	Effective for COC removal but cost too high.

	Effectiveness	Implementability	Relative Cost
⊗	Not effective at reaching RAOs	Not implementable at the Site	$$$$ - High
○	Partially effective for some COCs or Site areas	Difficult to implement	$$$ - Medium-high
◉	Effective under certain conditions	Implementable, requires technical knowledge	$$ - Moderate
●	Demonstrated effective technology	Readily implemented	$ - Low

Table 4
Alternatives Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Alternative	Alternative 1: No Action	Alternative 2: Enhanced MNR with Thin-Layer Amended Cover	Alternative 3: Potential Bioactive Zone Cap	Alternative 4: Sediment Dredging and Excavation	Alternative 5: Enhanced MNR with Broadcast Amendment
Total Present Worth Cost	$0	$8,219,000	$8,508,000	$10,101,000	$3,355,000
Cover/Cap Area	0 acres	16.5 acres; 0.30-meter (1-foot) amended sand cover	16.5 acres; 0.65-meter (2.1-feet) to 1.35-meter (4.4-feet) sand cap; includes 0.15-meter (0.5-foot) mixing layer	16.5 acres; 0.15-meter (0.5-foot) sand cover	16.5 acres; 0.01 meter broadcast amendment cover
Dredge Area	0 acres	0 acres	0 acres	2.5 acres dredged to 0.65 meter (2.1 feet); 14 acres dredged to 0.80 meter (2.6 feet); includes 0.15-meter (0.5-foot) over dredge	0 acres
Cover Volume - Sand/Amendment	0 CY/ 0 CY	25,700 CY/ 850 CY	92,000 CY/ 0 CY	13,000 CY/ 0 CY	0 CY/ 851 CY
Dredge Volume	0 CY	0 CY	0 CY	68,000 CY	0 CY
Construction Timeframe	0 weeks	14 weeks	33 weeks	29 weeks	3 weeks
Monitoring Program	None	Chemical and physical sediment and cover; benthic toxicity and bioaccumulation; fish tissue	Chemical and physical sediment and cap; benthic toxicity and bioaccumulation; fish tissue	None	Chemical and physical sediment and cover; benthic toxicity and bioaccumulation; fish tissue

Table 5
Cost Estimate - Alternative 2: Enhanced Monitored Natural Recovery with Thin-Layer Amended Cover
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Description	Unit	Estimated Unit Cost	Estimated Quantity	Extended Value	Present Value	Comments
Construction Costs						
Mobilization/Demobilization	Lump Sum	$ 133,000	1	$ 133,000	$ 124,299	All construction occurs on Year 1
Construct Staging Area	Lump Sum	$ 198,000	1	$ 198,000	$ 185,047	
Purchase Amendment Materials and Stockpile at Staging Area	Ton	$ 3,000	1465	$ 4,393,729.13	$ 4,106,289	Activated carbon
Purchase Sand and Stockpile at Staging Area	CY	$ 20.80	25721	$ 534,991	$ 499,992	
Purchase Armor and Stockpile at Staging Area	CY	$ 28.25	1976	$ 55,831	$ 52,179	
Construct Cover	CY	$ 35.17	26572	$ 934,580	$ 873,439	0.30-meter cover; 1 lift
Place Armor	CY	$ 50.25	1976	$ 99,303	$ 92,806	
Construction Monitoring/CQA and Oversight	Week	$ 12,802	14	$ 179,228	$ 167,503	
Monthly Operating Expenses and Site Security	Month	$ 18,000	4.0	$ 72,000	$ 67,290	
Implement Institutional Controls	Lump Sum	$ 20,000.00	1	$ 20,000	$ 18,692	Site postings; restrictions
			SUBTOTAL	$ 6,620,662	$ 6,187,535	
Long-Term Monitoring						
Monitoring and Evaluation Report	Each	$ 4,000	6	$ 24,000	$ 8,631	Every 5 years for 30 years
Field Sampling	Event	$ 34,000	6	$ 204,000	$ 73,366	Every 5 years for 30 years
Sample Analysis	Event	$ 49,427	6	$ 296,562	$ 106,654	Every 5 years for 30 years
			SUBTOTAL	$ 524,562	$ 188,651	
			TOTAL	$ 7,145,224	$ 6,376,186	
			25% Contingency	$ 687,874	$ 567,474	Contingency excludes purchasing amendment materials.
			CONSTRUCTION GRAND TOTAL	$ 7,833,098	$ 6,943,660	
Professional and Technical Services						
Remedial Design (6%)	Lump Sum	$ 470,000	1	$ 470,000	$ 470,000	Year 0
Project Management and Permitting (5%)	Lump Sum	$ 392,000	1	$ 392,000	$ 366,355	Year 1
Construction Management (6%)	Lump Sum	$ 470,000	1	$ 470,000	$ 439,252	Year 1
			SUBTOTAL	$ 1,332,000	$ 1,275,607	
			TOTAL	**$ 9,165,000**	**$ 8,219,000**	

Notes:
All values are based on 2016 dollars with an assumed discount rate of 7 percent per year. See Appendix A for present value calculations.
Assumptions are based on professional judgment and experience of specialists at Bay West. Actual project costs will be highly dependent upon final design.

Table 6
Cost Estimate - Alternative 3: Potential Bioactive Zone Cap
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Description	Unit	Estimated Unit Cost	Estimated Quantity	Extended Value	Present Value	Comments
Construction Costs						
Mobilization/Demobilization	Lump Sum	$ 133,000	1	$ 133,000	$ 124,299	All construction occurs on Year 1
Construct Staging Area	Lump Sum	$ 198,000.00	1	$ 198,000	$ 185,047	
Purchase Sand and Stockpile at Staging Area	CY	$ 20.80	87688	$ 1,823,901	$ 1,704,581	
Purchase Armor and Stockpile at Staging Area	CY	$ 28.25	1945	$ 54,938	$ 51,344	
Construct Cover	CY	$ 35.17	87688	$ 3,084,159	$ 2,882,392	
Place Armor	CY	$ 50.25	1945	$ 97,714	$ 91,321	
Construction Monitoring/CQA and Oversight	Week	$ 12,802	31	$ 396,862	$ 370,899	
Monthly Operating Expenses and Site Security	Month	$ 18,000	8	$ 144,000	$ 134,579	
Implement Institutional Controls	Lump Sum	$ 20,000	1	$ 20,000	$ 18,692	Site postings; restrictions
			SUBTOTAL	$ 5,952,574	$ 5,563,154	
Long-Term Monitoring						
Monitoring and Evaluation Report	Each	$ 4,000	6	$ 24,000	$ 8,631	Every 5 years for 30 years
Field Sampling	Event	$ 34,000	6	$ 204,000	$ 73,366	Every 5 years for 30 years
Sample Analysis	Event	$ 49,427	6	$ 296,562	$ 106,654	Every 5 years for 30 years
			SUBTOTAL	$ 524,562	$ 188,651	
			TOTAL	$ 6,477,136	$ 5,751,805	
			25% Contingency	$ 1,619,284	$ 1,437,951	
			CONSTRUCTION GRAND TOTAL	$ 8,096,420	$ 7,189,756	
Professional and Technical Services						
Remedial Design (6%)	Lump Sum	$ 486,000	1	$ 486,000	$ 486,000	Year 0
Project Management and Permitting (5%)	Lump Sum	$ 405,000	1	$ 405,000	$ 378,505	Year 1
Construction Management (6%)	Lump Sum	$ 486,000	1	$ 486,000	$ 454,206	Year 1
			SUBTOTAL	$ 1,377,000	$ 1,318,710	
			TOTAL	**$ 9,473,000**	**$ 8,508,000**	

Notes:

All values are based on 2016 dollars with an assumed discount rate of 7 percent per year. See Appendix A for present value calculations.

Assumptions are based on professional judgment and experience of specialists at Bay West. Actual project costs will be highly dependent upon final design.

Table 7
Cost Estimate - Alternative 4: Sediment Dredging and Excavation
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Description	Unit	Estimated Unit Cost	Estimated Quantity	Extended Value	Present Value	Comments
Construction Costs						
Mobilization/Demobilization	Lump Sum	$ 191,068	1	$ 191,068	$ 178,568	All construction occurs on Year 1
Site Work	Lump Sum	$ 350,000	1	$ 350,000.00	$ 327,103	Clear and grub, haul roads/staging areas, dewatering pad, site fencing
Install and Remove Cofferdam	Lump Sum	$ 572,032	1	$ 572,031.76	$ 534,609	
Dewater Excavation Area	Lump Sum	$ 63,124	1	$ 63,124.11	$ 58,994	Assume no treatment of first 80% of water removed from within cofferdam
Treat Excavation Area Water and Infiltration/Precipitation	Lump Sum	$ 700,795	1	$ 700,795.00	$ 654,949	Assume treatment of last 20% of water removed from within cofferdam
Debris Removal	Lump Sum	$ 63,078	1	$ 63,078.00	$ 58,951	Assume 3 days for debris removal
Dredge Sediments "In the Wet"	CY	$ 44.75	8427	$ 377,103.88	$ 352,434	
Excavate Sediments "In the Dry"	CY	$ 27.81	59352	$ 1,650,732.90	$ 1,542,741	
Sediment Hauling and Landfill Disposal	Ton	$ 17.66	118614	$ 2,094,150.31	$ 1,957,150	
Purchase Sand and Stockpile at Staging Area	CY	$ 20.80	13073	$ 271,923.13	$ 254,134	
Construct Sand Cover "in the Wet"	CY	$ 35.17	1945	$ 68,400	$ 63,925	
Construct Sand Cover "in the Dry"	CY	$ 14.01	11129	$ 155,888	$ 145,689	
Turbidity Controls	Lump Sum	$ 78,128	1	$ 78,128	$ 73,017	
Restore Wetland Areas	Acre	$ 16,880	1	$ 16,880	$ 15,776	Assume up to 1 acre of restoration
Construction Monitoring/CQA and Oversight	Week	$ 12,802	28	$ 358,456	$ 335,006	
Monthly Operating Expenses and Site Security	Month	$ 18,000.00	8	$ 144,000	$ 134,579	
Construction Monitoring and Sample Analysis	Lump Sum	$ 86,000	1	$ 86,000	$ 80,374	
Monthly Operating Expenses and Site Security	Month	$ 18,000	7	$ 126,000	$ 117,757	
			SUBTOTAL	$ 7,367,758	$ 6,885,755	
			25% Contingency	$ 1,841,940	$ 1,721,439	
			CONSTRUCTION GRAND TOTAL	$ 9,209,698	$ 8,607,194	
Professional and Technical Services						
Remedial Design (6%)	Lump Sum	$ 550,000	1	$ 550,000	$ 550,000	Year 0
Project Management and Permitting (5%)	Lump Sum	$ 460,000	1	$ 460,000	$ 429,907	Year 1
Construction Management (6%)	Lump Sum	$ 550,000	1	$ 550,000	$ 514,019	Year 1
			SUBTOTAL	$ 1,560,000	$ 1,493,925	
			TOTAL	**$ 10,770,000**	**$ 10,101,000**	

Notes:
All values are based on 2016 dollars with an assumed discount rate of 7 percent per year. See Appendix A for present value calculations.
Assumptions are based on professional judgment and experience of specialists at Bay West. Actual project costs will be highly dependent upon final design.

Table 8
Cost Estimate - Alternative 5: Enhanced Monitored Natural Recovery with Broadcast Amendment
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Description	Unit	Estimated Unit Cost	Estimated Quantity	Extended Value	Present Value	Comments
Construction Costs						
Mobilization/Demobilization	Lump Sum	$ 133,000	1	$ 133,000	$ 124,299	All construction occurs on Year 1
Construct Staging Area	Lump Sum	$ 198,000	1	$ 198,000	$ 185,047	
Install and Remove Dolphin Pilings	Lump Sum	$ 95,000.00	1	$ 95,000	$ 88,785	
Purchase Amendment Materials and Stockpile at Staging Area	Ton	$ 4,000	511	$ 2,042,280.00	$ 1,908,673	Activated carbon pellets
Broadcast Amendment	CY	$ 79.04	851	$ 67,264	$ 62,864	0.01 meter cover
Construction Monitoring/CQA and Oversight	Week	$ 12,802	3	$ 38,406	$ 35,893	
Monthly Operating Expenses and Site Security	Month	$ 18,000	1	$ 18,000	$ 16,822	
Implement Institutional Controls	Lump Sum	$ 20,000.00	1	$ 20,000	$ 18,692	Site postings; restrictions
			SUBTOTAL	$ 2,611,950	$ 2,441,075	
Long-Term Monitoring						
Monitoring and Evaluation Report	Each	$ 4,000	6	$ 24,000	$ 8,631	Every 5 years for 30 years
Field Sampling	Event	$ 34,000	6	$ 204,000	$ 73,366	Every 5 years for 30 years
Sample Analysis	Event	$ 46,935	6	$ 281,610	$ 101,277	Every 5 years for 30 years
			SUBTOTAL	$ 509,610	$ 183,274	
			TOTAL	$ 3,121,560	$ 2,624,349	
			25% Contingency	$ 269,820	$ 178,919	Contingency excludes purchasing amendment materials.
			CONSTRUCTION GRAND TOTAL	$ 3,391,381	$ 2,803,268	
Professional and Technical Services						
Remedial Design (6%)	Lump Sum	$ 203,000	1	$ 203,000	$ 203,000	Year 0
Project Management and Permitting (5%)	Lump Sum	$ 170,000	1	$ 170,000	$ 158,879	Year 1
Construction Management (6%)	Lump Sum	$ 203,000	1	$ 203,000	$ 189,720	Year 1
			SUBTOTAL	$ 576,000	$ 551,598	
			TOTAL	**$ 3,967,000**	**$ 3,355,000**	

Notes:

All values are based on 2016 dollars with an assumed discount rate of 7 percent per year. See Appendix A for present value calculations.

Assumptions are based on professional judgment and experience of specialists at Bay West. Actual project costs will be highly dependent upon final design.

Table 9
Comparative Analysis Summary - Threshold, Balancing, and Modifying Criteria
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Evaluation Criteria	Alternative 1: No Action	Alternative 2: Enhanced MNR with Thin-Layer Amended Cover	Alternative 3: PBAZ Cap	Alternative 4: Sediment Dredging and Excavation	Alternative 5: Enhanced MNR with Broadcast Amendment
Threshold Criteria					
Overall Protection of Human Health & Environment	Provides **no achievement** of protection of Human Health and the Environment as contaminant concentrations remain with minimal controls to prevent exposure.	Provides a **moderate achievement** of protection of Human Health and the Environment. Sediment contaminants would be reduced through addition of an amendment material and controlled by providing a clean sand layer between contaminated sediments and the water column. May require monitoring to ensure effectiveness and future additions of cover material.	Provides a **moderate to high** achievement of protection of Human Health and the Environment. Contaminated sediment would remain in place but contaminants would be completely isolated and would be reduced over time.	Provides a **high achievement** of protection of Human Health and the Environment. Only residual contaminated sediment would remain in place; however, it is anticipated that the residual contamination will not exceed the RAOs.	Provides a **moderate achievement** of protection of Human Health and the Environment. Sediment contaminants would be reduced through addition of an amendment material May require monitoring to ensure effectiveness and future additions of cover material.
ARARs	Provides **no achievement** of ARARs since chemical-specific TBCs are not met for sediment. Location and action-specific ARARs do not apply to this alternative.	Provides a **moderate achievement** of ARARs if implemented properly; however, COCs may not be reduced to concentrations less than RAOs in a reasonable time frame.	Provides a **moderate achievement** of ARARs if implemented properly; however, COCs may not be reduced to concentrations less than RAOs in a reasonable time frame.	Provides a **high achievement** of ARARs if implemented properly. Contaminants above the RAOs would be removed.	Provides a **moderate achievement** of ARARs if implemented properly; however, COCs may not be reduced to concentrations less than RAOs in a reasonable time frame.
Primary Balancing Criteria					
Long-term Effectiveness and Permanence	Provides **no achievement** of long-term effectiveness or permanence.	Provides a **moderate to high** achievement of long-term effectiveness and permanence because sediment contaminants would eventually be sequestered by amendment materials and rendered unavailable to biota within the most biologically active zone; however, sequestration of contaminants at deeper intervals may not occur and monitoring and possible reapplication of cover material may be necessary as contaminants would remain in place.	Provides a **moderate to high** achievement of long-term effectiveness and permanence because it isolates contaminated sediments from receptors and reduces contaminant concentrations in sediments to less than RAOs over time; however, monitoring, and possible reapplication of the cap material may be necessary as all contaminants would remain in place .	Provides a **high achievement** of long-term effectiveness and permanence. Contaminated sediments would be permanently removed from the Site; however, contaminated sediments would be placed in a disposal facility requiring long-term O&M.	Provides a **moderate** achievement of long-term effectiveness and permanence because sediment contaminants may eventually be sequestered by amendment materials and naturally deposited sediment and rendered unavailable to biota within the most biologically active zone; however, sequestration of contaminants at deeper intervals may not occur and monitoring and possible reapplication of amendment material may be necessary as contaminants would remain in place.
Reduction of Toxicity, Mobility or Volume through Treatment	Provides **no achievement** of this criterion as no reduction in toxicity, mobility, or volume is provided.	Provides a **moderate to high achievement** of this criterion as the toxicity and mobility of sediment contaminants would be reduced through addition of an amendment material near the sediment surface; however, it is possible that deeper sediment contamination could remain in place indefinitely.	Provides a **low achievement** of this criterion as the toxicity and mobility of sediment contaminants at the Site would be reduced, but not through treatment.	Provides a **low achievement** of this criterion as the toxicity and mobility of sediment contaminants at the Site would be reduced, but not through treatment.	Provides a **moderate to high achievement** of this criterion as the toxicity and mobility of sediment contaminants would be reduced through addition of an amendment material at the sediment surface; however, it is possible that deeper sediment contamination could remain in place indefinitely.
Short-term effectiveness	Provides a **high achievement** of this criterion as no actions are implemented; however, receptors would continue to be exposed to contaminated sediment.	Provides a **moderate to high achievement** of this criterion since cover placement would temporarily displace the benthic community. Risks to workers is low.	Provides a **moderate to high achievement** of this criterion since cap placement would temporarily displace the benthic community. Risks to workers is low.	Provides a **moderate achievement** of this criterion since dredging and removal of the PBAZ would take place across the entire remedial footprint. Risks to Site workers is moderate due to potential exposure to contaminated sediments.	Provides a **moderate to high achievement** of this criterion since placement of amendment may temporarily displace the benthic community. Risks to workers is low.
Implementability	Provides a **high achievement** of this criterion as no actions would be implemented.	Provides a **moderate to high achievement** of implementability since it only requires placement of cover material using proven methods with a low to moderate level of complexity.	Provides a **moderate to high achievement** of implementability since it only requires placement of cap material using proven methods with a low to moderate level of complexity.	Provides a **moderate achievement** of implementability since it requires cofferdam installation and dewatering and additional material handling operations.	Provides a **moderate to high achievement** of implementability since it only requires placement of amendment material using proven methods with a low to moderate level of complexity.
Cost (1)	$0	$8,200,000	$8,500,000	$10,100,000	$3,400,000
Modifying Criteria					
State Support / Agency Acceptance	TBD	TBD	TBD	TBD	TBD
Community Acceptance	TBD	TBD	TBD	TBD	TBD

Notes
(1) Cost are presented as Present Value.
M = Million
** Not included in numerical comparison on (Table 5-2).*
TBD = To Be Determined

Table 10
Comparative Analysis Summary - Green Sustainable Remediation Criteria
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Evaluation Criteria	Alternative 1: No Action	Alternative 2: Enhanced MNR with Thin-Layer Amended Cover	Alternative 3: PBAZ Cap	Alternative 4: Sediment Dredging and Excavation	Alternative 5: Enhanced MNR with Broadcast Amendment
Green Sustainable Remediation (GSR) Criteria*					
Green House Gas (GHG) Emissions	None.	Total GHG emissions produced during mob/demob activities, material import, cover construction, and mobilization related to O&M sampling activities.	Total GHG emissions produced during mob/demob activities, material import, cap construction, and mobilization related to O&M sampling activities.	Total GHG emissions produced during mob/demob activities, sediment removal, material import, cover construction, and mobilization related to O&M sampling activities.	Total GHG emissions produced during mob/demob activities, material import, broadcasting amendment, and mobilization related to O&M sampling activities.
Toxic Chemical Usage and Disposal	None.	No toxic chemicals are used or disposed.	No toxic chemicals are used or disposed.	No toxic chemicals are used or disposed.	No toxic chemicals are used or disposed.
Energy Consumption	None.	Fossil fuels used during mob/demob activities, material import, cover construction, and mobilization related to O&M sampling activities.	Fossil fuels used during mob/demob activities, material import, cap construction, and mobilization related to O&M sampling activities.	Fossil fuels used during mob/demob activities, sediment removal, material import, cover construction, and mobilization related to O&M sampling activities.	Fossil fuels used during mob/demob activities, material import, cover construction, and mobilization related to O&M sampling activities.
Use of Alternative Fuels	None.	Alternative fuels could be used to run heavy construction equipment.	Alternative fuels could be used to run heavy construction equipment.	Alternative fuels could be used to run heavy construction equipment.	Alternative fuels could be used to run heavy construction equipment.
Water Consumption	No water consumption is necessary.	No water consumption is necessary.	No water consumption is necessary.	Little water consumption is necessary.	No water consumption is necessary.
Waste Generation	None.	No waste generation.	No waste generation.	Contaminated sediments, dewatering pad materials, media	No waste generation.
GSR Criteria Summary	Provides a **high achievement** of the GSR criterion.	Provides a **moderate achievement** of the GSR criterion.	Provides a **moderate achievement** of the GSR criterion.	Provides a **low achievement** of the GSR criterion.	Provides a **moderate achievement** of the GSR criterion.

Notes
(1) Cost are presented as Present Value.
M = Million
** Not included in numerical comparison on (Table 5-2).*
TBD = To Be Determined

Table 11
Numerical Comparative Analysis Summary
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Evaluation Criteria	Alternative 1: No Action	Alternative 2: Enhanced MNR with Thin-Layer Amended Cover	Alternative 3: PBAZ Cap	Alternative 4: Sediment Dredging and Excavation	Alternative 5: Enhanced MNR with Broadcast Amendment
Overall Protection of Human Health & Environment	0	2	2.5	3	2
ARARs	0	2	2	3	2
Long-term Effectiveness and Permanence	0	2.5	2.5	3	2.5
Reduction of Toxicity, Mobility or Volume through Treatment	0	2.5	1	1	2.5
Short-term effectiveness	3	2.5	2.5	2	3
Implementability	3	2.5	2.5	2	2.5
Cost (1)	3	2	2	1	3
State Support / Agency Acceptance	TBD	TBD	TBD	TBD	TBD
Community Acceptance	TBD	TBD	TBD	TBD	TBD
Total Numerical Value	**9**	**16**	**15**	**15**	**17.5**

Notes

(1) Cost are presented as Present Value.

Ratings are based on achievement of criterion: no achievement, low achievement; moderate achievement; and high achievement.

Scores are based on 0 = no achievement; 1 = low achievement; 2 = moderate achievement; and 3 = high achievement.

Scoring for cost are based on the following cost breakpoints: > $9 million = low achievement; $4-9 Million = moderate achievement; and < $4 million = high achievement.

GSR criteria not included in this numerical comparison.

See Table 6 for a discussion of each criterion.

Appendix A

Scanlon Reservoir Technical Memorandum, June 2017

Scanlon Reservoir Technical Memorandum

Scanlon Reservoir
Cloquet, Minnesota

June 2017

Scanlon Reservoir Technical Memorandum

Scanlon Reservoir
Cloquet, Minnesota

June 2017

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Table of Contents

List of Tables

List of Figures

List of Appendices

Acronyms and Abbreviations

%	percent
µg/kg	micrograms per kilogram
28-d	28-day
ANOVA	analysis of variance
AOC	Area of Concern
ASTM	American Society for Testing and Materials
Bay West	Bay West LLC
bss	below sediment surface
BUI	beneficial use impairment
CoC	chain of custody
COC	chemicals of concern
COI	Constituent of Interest
DQO	data quality objective
ELAP	Environmental Laboratory Accreditation Program
FFS	focused feasibility study
FSP	Field Sampling Plan
GLEC	Great Lakes Environmental Center, Inc.
GPS	Global Positioning System
HD	Hester Dendy
HH	human health
ID	identification
IDW	investigation-derived waste
J	estimated
KM	Kaplan-Meier
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDL	method detection limit
mg/kg	milligrams per kilogram
MPCA	Minnesota Pollution Control Agency
MS/MSD	matrix spike/matrix spike duplicate
ng	nanograms
NR	Not Recorded
Pace	Pace Analytical Services, Inc.
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
SOP	standard operating procedure
SQT	sediment quality target
SR	Scanlon Reservoir
TEF	toxicity equivalency factor
TEQ	toxicity equivalent
TEQ/kg	toxicity equivalent per kilogram
TestAmerica	TestAmerica Laboratories, Inc.
TOC	total organic carbon
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources
WHO	World Health Organization

1.0 INTRODUCTION

Bay West LLC (Bay West) has completed a Technical Memorandum to support the Focused Feasibility Study (FFS) completed June 2016 by Bay West under contract with the Minnesota Pollution Control Agency (MPCA) at Scanlon Reservoir (SR), also designated as SR #1374 (the Site). Limited field activities were conducted as part of ongoing work to investigate the extent and volume of contaminated sediment within SR, and to evaluate risks to human health and the environment due to potential impacts by the benthic community. A site location map is included as **Figure 1**, and a site map is included as **Figure 2**.

This Technical Memorandum describes investigation field activities conducted in September and October of 2016, presents chemical, physical, and bioaccumulation site data collected during this event, discusses data results, conclusions, and recommendations. This Technical Memorandum is intended to be a supplement to the FFS; therefore, only data from the September and October 2016 event will be presented in this document. Historical data collected at the Site is summarized in the FFS.

1.1 Purpose and Objectives

Historical industrial activities in the St. Louis River (SLR) Area of Concern (AOC) has resulted in beneficial use impairments (BUIs). BUIs are a change in the chemical, physical or biological integrity of the Great Lakes system sufficient to cause any one of the 14 established BUIs, or other related uses, such as the microbial objective for waters used for body contact recreational activities (2013 Joint Commission). Sediment contamination in the SLR AOC causes BUIs, including degradation of bottom-feeding invertebrate communities, increased incidence of fish tumors and other abnormalities, fish consumption advisories, and restrictions on dredging (MPCA, 2008). The MPCA and Wisconsin Department of Natural Resources (WDNR) are currently working together to implement a comprehensive long-term plan to restore beneficial use and delist BUIs in the SLR AOC. Many of the BUIs in the AOC are linked to the presence of sediment contaminants. Some sediment-derived contaminants also appear suspended in the water column and are carried by the river to Lake Superior.

The purpose of this Technical Memorandum was to collect information to supplement existing information discussed in the FFS regarding sediment quality at the Site, including chemical and physical site data. Data collected will ultimately be used to develop a course for remedial action, if needed, to restore and delist the Site BUIs.

Specific objectives for the September and October 2016 investigation were to:

- Provide site-specific information regarding benthic organisms and the chemicals of concern (COCs; i.e., mercury and dioxins/furans as defined within the FFS to benthic organisms);
- Conduct limited benthic macroinvertebrate community assessments to assess the “health” of the benthic community at locations with elevated COC concentrations and to provide an additional line of evidence regarding contaminant impacts at the Site using the sediment quality triad approach;
- Collect and analyze sediment samples for Site COCs to corroborate findings of bioaccumulation testing and to further define the vertical extent of contamination at the Site;
- Collect and analyze fish tissue samples for Site COCs to assess potential COC bioaccumulation; and

- Refine the conceptual site model that evaluates contaminant fate and transport, and provides a comparison between SLR AOC-specific risk-based screening values and existing conditions to identify unacceptable risks to human health and/or the environment.

1.2 Report Organization

Section 1.0 – Introduction: This section provides a brief overview of the SLR AOC, SR, and summarizes previous investigations and constituents of interest (COIs) relative to the Site.

Section 2.0 – Field Activities and Methods: This section describes the field activities and methods utilized.

Section 3.0 – Summary of Results: This section summarizes the results of the data collection, including chemical and physical site data.

Section 4.0 – Data Quality Review: This section describes the data quality review process and the results of quality assurance (QA)/quality control (QC) review of chemical data.

Section 5.0 – Discussion and Conclusion: This section discusses the results and conclusions.

Section 6.0 – References: This presents references for the report.

1.3 Site Setting

This document serves as a supplement to the existing FFS completed in 2016, which provides a full description of the site settings and history.

1.4 Investigation History and COIs

Numerous investigations of sediment quality have occurred at the Site, resulting in various report documents, which have been summarized in the FFS. Prior to reading this document, a review of Section 1.4.3 of the FFS should be completed to provide a better understanding of the Site history. Those investigations and reports not summarized in the FFS are summarized as follows.

***Data Gap Investigation Field Sampling Plan (FSP), Scanlon Reservoir,* prepared by Bay West, September 2016.**

The FSP was developed by Bay West and approved by the MPCA in September 2016.

2.0 FIELD ACTIVITIES AND METHODS

Sampling activities and procedures were conducted in accordance with the September 2016 Scanlon Reservoir Site-Specific Data Gap Investigation FSP, the September 2016 Quality Assurance Project Plan Addendum (2016 Quality Assurance Project Plan [QAPP] Addendum) for the Baseline and Data Gap Investigation at the SLR Areas of Concern, and applicable Bay West standard operating procedures (SOPs). The following section describes applicable physical site data, sediment sampling and procedure, and analytical results evaluation procedure.

All sample locations were pre-determined and aerial background maps were loaded onto a Trimble Global Positioning System (GPS) unit with sub-meter accuracy prior to site mobilization. The GPS was used to navigate as close to the pre-determined sample locations as possible, and GPS locational data was also collected at each of the sampled locations.

2.1 Sediment Sampling Overview

Site sediment sampling took place September 19 through October 5, 2016, and was conducted from a boat. The objectives of the September and October event were to collect bulk surface sediments for laboratory bioaccumulation testing, sediment community assessments, and to gather additional vertical and horizontal sediment quality data, as further defined in the FSP.

Four locations, BW16SR-001 through BW16SR-005, excluding BW16SR-004, were sampled for in situ tissue, community assessments, and physical and chemical analysis. BW16SR-004 and BW16SR-016 were sampled for physical and chemical analysis, bioaccumulation testing, and laboratory exposure testing.

Sediment samples were collected only for physical and chemical analysis at the following locations: BW16SR-006 through BW16SR-015.

The following sections contain additional information on the sampling event, and the methods, procedures, and equipment used during sediment sample collection, if not already covered in the FFS or FSP. Sample locations are shown on **Figure 3**.

2.1.1 Ponar Equipment Description and Procedure

All surface sediment samples were collected using a Wildco Petite Ponar grab sampler (ponar). The ponar was used to collect sediments from the sediment/water interface for submission as a bioaccumulation testing media, for benthic community assessments, and for physical and chemical analysis.

The ponar has a maximum sediment penetration depth of 2.75 inches (0.07 meter) and a total jaw volume of 2.4 liters. Due to the small size of the sampler, multiple “grabs” of sediment were performed at each location to collect a sufficient volume of sediment for testing/analysis (up to 5 gallons of sediment per location). After each grab of sediment, the team repositioned the sampler so that the next grab was collected approximately 0.25–0.50 meter away from the previous grab. This method of sediment collection was repeated to ensure that the final composite samples were representative of a single in situ sediment elevation (i.e., 0–0.07 meter).

Collected sediment was transferred directly from the ponar into clean, laboratory supplied, 5-gallon buckets. Once a sufficient volume of sediment had been collected, overlying water was decanted and the sediment was thoroughly homogenized within the buckets. A sub-sample was then collected and placed within Ziploc-type bags (double bagged) for grain size analysis.

2.1.2 Check Valve Push Core Collection Equipment and Procedure

Samples to be analyzed for physical and chemical parameters were collected using a 3-inch (inner diameter) check valve push core sampler. The check valve push core sampler used disposable acetate liners that minimized equipment decontamination and facilitated easy transport and storage of samples.

To begin sampling at a location, the water column depth was measured using a weighted 100-foot measuring tape. The water depth was then added to the desired depth of sampler advancement (i.e., desired core length), and this value was marked on the sampler's extension rods using marking tape. To collect the core, the sampler was lowered through the water column and advanced into the sediment until the mark met the water surface (indicating that a full push had been achieved), or until refusal was encountered. If refusal was encountered, the push was recorded by subtracting the distance between the mark and the water surface from the desired depth of sampler advancement.

Once the push was complete, the sampler was retracted while remaining in a vertical orientation. The recovery goal of the sampling event was 80 percent (%). If less than 80% recovery was achieved after three push attempts, or if refusal was encountered, the team attempted to obtain a core with the best feasible length and percent recovery based on Site conditions. Once the Engineer determined that the sample recovery was acceptable, the sample core was prepared for transport by draining excess overlying water, removing any excess core tubing to limit head space, and sealing both ends using disposable plastic caps. The core was then measured and identifying information was recorded on the core using an indelible ink marker.

In addition to core collection, poling was conducted at each check valve push core sampler location, based on Site conditions, using an approximate 2-inch diameter aluminum rod with graduated depth markings. Data recorded included: depth to resistance, depth to refusal, refusal type (i.e., soft [stiff sediments] or hard [rock or wood]), and observations of sediment type encountered. All field data related to sample collection and poling was recorded within a field notebook and/or on field sampling data sheets. The recorded field data included sample location, sample date/time, push, recovery, and any other observations that occurred during sampling, such as refusal. Core collection information is presented in **Table 3**, and field notes are included in **Appendix A**. Poling locations and sediment thicknesses are shown on **Figure 4**.

2.1.3 Equipment Decontamination

After each coring attempt, all materials in contact with sediments were washed with lake water to remove visible sediments (i.e., Wildco Petite Ponar and check valve push core sampler). After each sample location, sampling equipment was decontaminated using Alconox, water, and a stiff bristled brush.

2.2 Benthic Macroinvertebrate Tissue Sampling Overview

2.2.1 In Situ Benthic Macroinvertebrate Tissue Sampling

Benthic invertebrate tissue sampling consisted of the attempted collection of benthic invertebrates using sediment sampling and sifting techniques, as well as Hester Dendy (HD) traps; however, tissue collection using sediment sampling and sifting techniques resulted in no appreciable mass of tissue. Only HD traps resulted in sufficient tissue mass for laboratory analysis. Benthic macroinvertebrate samples collected from HD traps will be referred to as "in situ" tissue samples. In situ benthic macroinvertebrate tissue sampling was collected using an HD trap placed in the sediment at the Site for approximately one month. The United States Environmental Protection Agency (USEPA) installed the HD traps at the Site in September 2016 and collected the traps in October 2016. The traps were placed into the sediment, allowing the benthic macroinvertebrates

to burrow into the spaces between the disks within the trap. The USEPA removed the HD traps and sorted the macroinvertebrates by species. The in situ tissue samples consisted of composited macrobenthos and crawfish from the Site, and were used for chemical analysis.

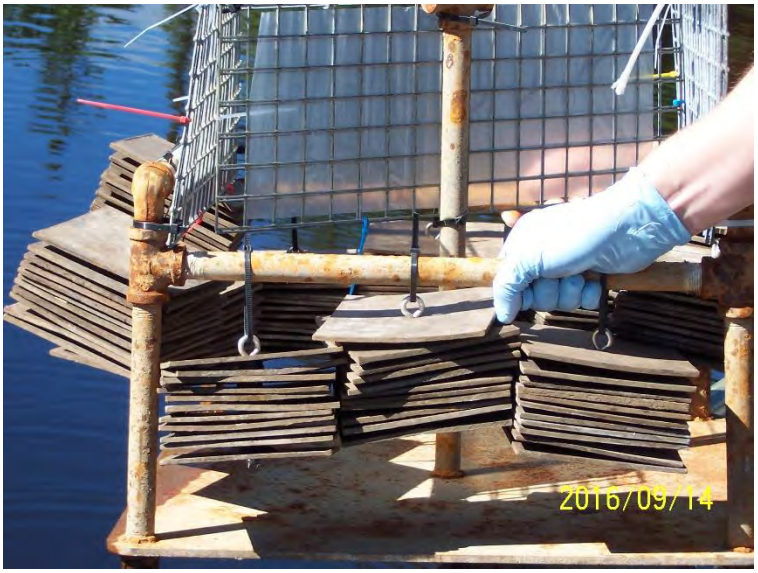

Photo showing discs within the HD trap

The in situ tissue samples consisted of composited macrobenthos, dragonflies, mayflies, and crawfish from the Site, and were used for chemical analysis. For the purpose of this report, macrobenthos are a composition of macroinvertebrates, such as mayflies, dragonflies, and alderflies, encountered at the Site. The compositing of macroinvertebrates was done at the Site due to insufficient mass of tissue for one specific macroinvertebrate species. Macrobenthos were composited from BW16SR-001 through BW16SR-005, with the exception of BW16SR-004. Mayflies were collected from BW16SR-001 through BW16SR-003 and BW16SR-005. Dragonflies were collected from BW16SR-002, BW16SR-003, and BW16SR-005. Crawfish were collected from BW16SR-005. One tissue sample was submitted for analysis using HD traps (BW16SR-HD-001-MRCS).

2.2.2 Ex Situ Benthic Macroinvertebrate Tissue Sampling

Sediment was also collected for the purpose of performing laboratory-controlled 28-day (28-d) Lumbriculus variegatus bioaccumulation testing. These samples will be referred to as “ex situ” tissue samples. Ex situ benthic invertebrate tissue collection was done at locations where HD traps were not able to produce sufficient in situ tissue volume for analysis. Sediment was collected using the Ponar grab sampler and stored in laboratory-supplied buckets. The sediment was submitted to the laboratory for bioaccumulation analysis and chemical and physical analysis. Sediment for ex situ analysis was collected at BW16SR-004 and BW16SR-016.

Details regarding the in situ and ex situ tissue analysis was documented in the *Tissue Analysis Project Plan for Duluth Reservoirs*, Draft Report, AEM (United States Army Corps of Engineers [USACE], 2016), which was prepared for USACE in **Appendix B**.

2.3 Fish Sampling Overview

Fish sampling was completed by the MPCA. Details regarding fish sampling was documented in the field forms and COCs provided to Advanced Environmental Management Group (AEM Group) as described in The Tissue Analysis Project Plan for Duluth Reservoirs, Draft Report, AEM Group,

which was prepared for USACE (2016) in **Appendix B**, Section 3.1, which provides a detailed explanation of fish sampling procedures completed at the Site.

2.4 Community Assessment Equipment Description and Procedure

Community assessments were completed by collecting approximately three ponar grabs of sediment from each sample location. The sediment was sieved through a 425-micron (35 mesh) screen. All material captured on the screen was placed into white plastic trays with fresh, cool water. Benthic organisms were removed from the tray, separated by organism type, and placed into small ice cube trays.

Photo showing a community assessment in progress

Search and removal of organisms from each plastic tray took place for 15 minutes to retain consistency across all sample locations. A count of each species identified was recorded on community assessment worksheets, a field notebook, or an electronic log. Benthic organisms were released back into the water once assessments were complete. Additional information regarding benthic community assessments is included in the Bay West Site Specific Benthic Macroinvertebrate Community Assessment SOP found in the FSP and as an appendix to the QAPP addendum. Sediment for community assessments were collected at BW16SR-001 through BW16SR-005.

2.5 Sample Processing

Collected sediment was brought back to shore for processing for submittal to a laboratory as a media during bioaccumulation testing, for physical and chemical analysis, and for community assessment. Sediment to be used as media and for physical and chemical analysis from each location either remained in the 5-gallon bucket or was transferred into the appropriate laboratory supplied containers, dependent on sampling parameters for that particular sample location. Once a sample was collected and the container sealed, the container (not the lid) was labeled with the sample location identification (ID), sample date, and time of collection using an indelible ink marker.

Sediment samples were processed and submitted for chemical analysis in accordance with the approved site-specific FSPs.

All sample processing was conducted following the sampling event. The following activities were conducted during sample processing:

- Sample collection information (e.g., location ID, sample time, push, recovery, interval depth, etc.) was transferred from each sample core or Ziploc bag to Bay West's Sediment Sampling Log Sheet;
- Each sample was photographed during field sampling or during processing;
- Visual and physical observations of the sample were recorded on the log sheet in accordance with the site specific FSPs following the American Society for Testing and Materials (ASTM) D 2488 and the United States Department of Agriculture descriptor classification, including sample color, material composition, grain size, firmness, cohesiveness, odor, and any other notable observations such as sheen.
- Analytical sample intervals were determined for core samples in accordance with the site-specific FSPs;
- Sample material was placed in appropriate laboratory-supplied containers, labeled, and placed on ice for delivery to either Pace Analytical Services, Inc. (Pace), TestAmerica Laboratories, Inc. (TestAmerica), Axys, or Great Lakes Environmental Center, Inc. (GLEC); and
- All reusable sampling tools used for homogenization or other purposes were decontaminated after processing in a solution of Alconox and distilled water using the procedures described in **Section 2.1.3**.

To complete the community assessment approximately three ponar grabs of sediment from each sample location were sieved through a 425-micron (35 mesh) screen. All material captured on the screen was placed into white plastic trays with fresh, cool water. Benthic organisms were removed from the tray, separated by organism type, and placed into smaller ice cube trays. Search and removal of organisms from each plastic tray took place for 15 minutes to retain consistency across all sample locations. A count of each species identified was recorded on community assessment worksheets, a field notebook, or an electronic log. Benthic organisms were released back into the water once assessments were complete. Additional information regarding benthic community assessments is included in the Bay West Site Specific Benthic Macroinvertebrate Community Assessment SOP found in the FSP and as an appendix to the QAPP addendum.

2.5.1 Sample Collection and Analysis

2.5.1.1 *Sediment Physical/Chemical Analysis*

Sediment samples from BW16SR-006 through BW16SR-015 were collected to gather additional chemical data for delineation of extent and depth using a check valve push core sampler as detailed in **Section 2.1.2**.

Samples collected for additional delineation of contamination were submitted for the following:

- Dioxins/furans as congeners (Pace, USEPA 8290A);
- Mercury (Pace, USEPA 7471B); and
- Total organic carbon (TOC; Pace, USEPA 9060A).
- Grain size (Pace, ASTM D422 with hydrometer).

All samples were collected, prepared, and handled in accordance with the FSP, project QAPP and addendum, and Bay West SOPs.

The specific analysis for each sample is detailed in **Table 1**. Each sample was accounted for on chain of custody (CoC) completed during sample processing. All samples were stored on ice and delivered to the appropriate laboratory.

QC samples collected by the processing team consisted of duplicates and matrix spike/matrix spike duplicates (MS/MSDs). Field duplicates and matrix MS/MSD samples were collected for sediments at a frequency of 10% and 5%, respectively, for dioxins/furans and mercury. No duplicate or MS/MSD sample was collected for TOC or grain size analysis. Field equipment rinsate blanks were collected at a frequency of 1 per day for each day the ponar sampler was used and analyzed for mercury. No duplicate or MS/MSD samples were collected in relation to benthic tissue analysis due to constraints in available tissue mass and project budget.

2.5.1.2 *Community Assessments*

Site benthic macroinvertebrates were collected from locations BW16SR-001 through BW16SR-005 for community assessments. Community assessments were completed as described in **Section 2.4** and the community assessment findings are discussed in **Section 3.5**.

2.5.1.3 *Bioaccumulation Testing*

As outlined in the FSP, site benthic macroinvertebrate tissue collected from locations BW16SR-001 through BW16SR-005, excluding BW16SR-004, were sampled for in situ and ex situ tissue. Ex situ bioaccumulation testing, which was not included as part of the FSP, was completed at locations BW16SR-004 and BW16SR-016 because a sufficient tissue volume for analysis could not be collected during the field event. Sediment samples collected from BW16SR-004 and BW16SR-016 were submitted for ex situ laboratory exposure testing and physical and chemical analysis of sediment and tissue.

In situ site benthic macroinvertebrate tissue was sampled only at BW16TR-001. Ex situ tissue samples were exposed to the sediment collected from the following locations, BW16TR-008, BW16TR-013, BW16TR-017, and BW16TR-018, because insufficient tissue volume could not be collected from the Site. Additionally, USACE conducted fish sampling at the Site. The bioaccumulation fish tissue sampling results were provided to Bay West by the USACE and are summarized in **Section 3.4.1.2**. The specific analysis for each sample is detailed in **Table 1**.

Sediment collected from BW16SR-004 and BW16SR-016 were containerized and delivered to the GLEC laboratory in laboratory-supplied containers. The GLEC laboratory conducted the following test:

- 28-d *Lumbriculus variegatus* bioaccumulation testing (USEPA Method 100.3 and laboratory SOP)

Following the 28-d *Lumbriculus variegatus* bioaccumulation testing, *Lumbriculus variegatus* tissue was extracted from the sediment substrate by GLEC. Subsamples from the sediment samples and *Lumbriculus variegatus* tissue samples were submitted to multiple laboratories for analysis summarized as follows. Additionally, Bay West submitted in situ collected tissue and sediment from locations BW16SR-001 through BW16SR-005 (excluding BW16SR-004) to the following laboratories using the following methods:

- Tissue Analysis–Dioxins/furans as congeners and lipids content (TestAmerica USEPA 1613B or 8290A);
- Sediment Analysis–Dioxins/furans as congeners (Pace, USEPA 1613B or 8290A);
- Tissue Analysis–Mercury (TestAmerica; ASTM D2216);
- Sediment Analysis–Mercury (Pace; ASTM D2216);

- Tissue Analysis–Methyl Mercury (TestAmerica, EPA 1630 USEPA);
- Sediment Analysis–TOC (Pace; USEPA method such as 9060A); and
- Sediment Analysis–Grain size (Pace, ASTM D422 with hydrometer).

Bioaccumulation testing samples were collected, prepared, and handled in accordance with the laboratory's SOPs on collection and handling of environmental samples. For a detailed description of bioaccumulation testing, procedures, and results see the 2016 Tissue Analysis Report in **Appendix B**.

2.5.1.4 *Fish Tissue Testing*

Both the MPCA and USEPA collected six different fish species from the Site, homogenized by fish species and delivered to the TestAmerica in laboratory-supplied containers. Fish homogenization and laboratory testing is discussed in the 2016 Tissue Analysis Report fish sampling procedures completed at the Site (**Appendix B**, Section 3.1). Samples were submitted to the following laboratories using the following methods:

- Dioxins/furans as congeners and lipids content (TestAmerica, USEPA 1613B or 8290A);
- Mercury (TestAmerica; ASTM D2216); and
- Methyl Mercury (TestAmerica, EPA 1630 USEPA);

Fish tissue samples were collected, prepared, and handled in accordance with the laboratory's SOPs on collection and handling of environmental samples. For a detailed description of fish tissue testing, procedures, and results, see the 2016 Tissue Analysis Report in **Appendix B**.

2.5.2 Rinsate Blanks

Rinsate blank samples were collected by pouring distilled water over non-disposable sampling equipment and into bottles provided by the analytical laboratory to verify proper decontamination of sampling equipment. Rinsate blanks were only taken for the ponar to verify proper decontamination of sampling equipment; it was not necessary to take rinsate samples from the check valve push core sampler because the majority of the sample only comes in contact with the disposable liner. The rinsate blanks were labeled BW16-RB01-092816, BW16-RB01-092216, and BW16-RB01-100516 and were analyzed for mercury. Mercury was not detected at concentrations exceeding the laboratory reporting limit for rinsate blanks.

2.5.3 Waste Characterization and Disposal

Investigation-derived waste (IDW) consisting of excess sediment and disposable sampling supplies was placed in two 55-gallon steel drums along with the IDW generated during the sampling event and two additional sampling events completed at Thomson Reservoir and Mud Lake West. A total of two drums of waste were generated during the three sampling events. An IDW sample was collected from the drums at the completion of sampling and submitted for analysis of landfill disposal parameters. The drums were transported to Bay West, under MPCA approval, and stored until IDW sample results were obtained. All IDW was characterized as non-hazardous waste and disposed of by Veolia ES Technical Solutions. Disposal documentation is included in **Appendix C**.

2.6 Data Interpretation

2.6.1 Treatment of Non-Detect Data

Scaling censored (non-detected) data was performed for dioxin/furan toxicity equivalent (TEQ) calculations for sediment and tissue with the goal to provide an accurate and consistent way to estimate TEQ values by eliminating false positives and false negatives from the final data set.

Unless otherwise noted, the sediment and tissue dioxin/furan data was input into a USEPA TEQ Kaplan-Meier (KM) calculator, which includes calculations that support a simple, quasi-sensitivity analysis that examines the effect of various ways of handling non-detect or rejected (R-flagged) analytical data results within a sample's congener profile. The TEQ KM calculator utilized 1998 World Health Organization (WHO) toxicity equivalency factors (TEFs) for fish (TEQ KM fish value) and WHO 2005 TEFs for human health (TEQ KM Human Health [HH] value).

The fish tissue mercury, methylmercury, TEQ KM HH, and TEQ KM Fish data was input into the USEPA ProUCL Oneway Analysis of Variance (ANOVA) statistics calculator. The ANOVA module has both classical and nonparametric Kruskal-Wallis tests and is used to compare means (or medians) of multiple groups such as comparing mean concentrations of areas of concern and to perform inter-sample comparisons. Classical Oneway ANOVA tests were used to determine statistical differences in each trophic level between the Site and Boulder Lake Reservoir (reference Site).

2.6.2 Sediment Quality Targets

Numerical sediment quality targets (SQTs), adopted for use in the SLR AOC to protect benthic invertebrates, can be used throughout Minnesota as benchmark values for making comparisons to sediment chemistry measurements. Level 1 and Level 2 SQTs for the protection of sediment-dwelling organisms are available for 8 trace metals, 13 individual polycyclic aromatic hydrocarbons (PAHs), total PAHs (all 13 priority PAHs), total polychlorinated biphenyls (PCBs), and 10 organochlorine pesticides. In addition, Level 1 and Level 2 SQTs for COCs were adopted for the protection of fish, as insufficient information is available for sediment-dwelling organisms. SQTs are highly useful when evaluating risk for a specific compound or a group of compounds (i.e., total PCBs and total PAHs).

Contaminant concentrations below the Level 1 SQTs are unlikely to have harmful effects on sediment-dwelling organisms (i.e. benthic invertebrates). Contaminant concentrations above the Level 2 SQTS are more likely to result in harmful effects to benthic invertebrates (MPCA, 2007). Based on conversations with the MPCA, a qualitative comparison value midway between the Level 1 SQTs and Level 2 SQTs (i.e., midpoint SQT) will be used as conservative criteria to identify, rank, and prioritize sediment-associated contaminants within the Site.

2.6.3 Sample Interval Categorization

Sediment samples were collected from horizons (A, B, and C) within the sediment core, in accordance with the FSP. Horizons were determined by core length, recovery, and the observation of anthropogenic materials, such as sheens, staining, or non-native debris. Because of varying core lengths and recovery, sediment sample collection depth was not consistent between sample locations. In order to spatially evaluate analytical results and sediment screening criteria comparisons between sample locations, sediment samples were categorized into depth intervals. Sediment intervals and the methods for categorizing sediment samples into intervals were determined through discussions with the MPCA. Sediment samples were categorized into four intervals based on the depth of collection. The intervals focus on the stratigraphy of contamination within the bioactive zone, which is assumed to be the upper meter of sediment. The intervals are as follows:

- 0.0 to 0.15 meter;
- 0.15 to 0.50 meter;
- 0.50 to 1.00 meter; and
- >1.0 meter.

Each sediment sample was categorized into one of the three intervals if at least 25% of the sample length was within an interval. For example, if a sample was collected from 0.30 to 0.55 meter below the sediment surface, the sample would be categorized in the 0.15 to 0.50-meter category. Occasionally, 25% of a sample was collected within two intervals. For example, if a sample was collected from 0.64 to 1.15 meters, 71% of the upper portion of the sample is within the 0.50 to 1.00-meter interval, and 29% of the lower portion of the sample is within the >1.00-meter interval. In these cases, the sample was considered in the discussion and evaluation of both the 0.5 to 1.00-meter interval and the >1.00-meter interval.

3.0 SUMMARY OF RESULTS

This section summarizes the results obtained from field activities.

3.1 Sample Depth and Sediment Recovery

The sampling objective at the Site, as outlined within the FSP, was to collect surface sediment samples and sediment samples for vertical and horizontal extent of Site contaminants.

As stated in **Section 2.1.1**, surface sediment samples were collected using a Wildco Petite Ponar sampler. Grab sample recovery was a 100%.

As stated in **Section 2.1.2**, additional sediment samples were collected using a check valve push core sampler. The sampler was advanced from the sediment surface to an average depth of at 1.8 meter below sediment surface (bss) at the Site. Refusal was encountered at five of the eight locations sampled. Refusal appeared to be due to clay and gravel layers encountered below an average 2.07 meters bss, creating increased resistance as the sampler was advanced. The average sediment recovery was approximately 70%, not achieving sediment recovery goals for the Site due to refusal at multiple locations.

Completed sediment collection logs and photographs of sediment prior to processing are included in **Appendix A**. **Table 1** and **Table 2** provide a summary of sample locations, water depths, sediment elevations, type of sample collected, and number of samples from each location.

3.2 Sediment Chemistry Data

The following discussion presents the summarized sediment analytical results from samples obtained from 16 locations collected during the September and October 2016 sampling event at the Site. Analytical results are presented with respect to the following depth intervals: 0.0 to 0.15 meter, 0.15 to 0.5 meter, 0.5 to 1.0 meter, and >1.0 meter. An explanation of sample interval calculations can be found in the **Section 2.5**.

Laboratory analyses and sampling frequency selected for sediment samples collected at the Site include the following:

- Mercury by SW-7471B (100%); and
- Dioxins and furans by SW-846 8290A (100%).

Table 5 provides a summary of sediment samples and laboratory analyses selected for each sample. Analytical results are presented in **Table 8** and **Table 9**, and laboratory analytical reports are included in **Appendix D**. **Figures 5** through **6** present analytical results for mercury and dioxin/furans at distinct intervals compared to their respective SQTs. The following sections present a summary of analytical results and calculations.

3.2.1 Mercury (Sediment)

All sediment samples were analyzed for mercury, and results were screened in comparison to the respective SQT values. The following table summarizes the mercury results. Mercury SQT results are illustrated in **Figure 5**.

Level 1 SQT exceedances occurred in samples BW16SR-002, BW16SR-008, BW16SR-012, and BW16SR-016 in the 0.0 to 0.15 and 0.15 to 0.50-meter intervals. No sample exceeded the Midpoint or Level II SQT. The maximum concentration of mercury (0.54 milligrams per kilogram [mg/kg]) was identified at location BW16SR-008.

Sample Name	Sample Interval (meter)	Result (mg/kg)	Results Qualifier
BW16SR-001-0.0-0.15	0.0-0.15	0.16	
BW16SR-002-0.0-0.15	0.0-0.15	0.42	
BW16SR-003-0.0-0.15	0.0-0.15	0.10	
BW16SR-004-0.0-0.15	0.0-0.15	0.17	
BW16SR-005-0.0-0.15	0.0-0.15	0.11	
BW16SR-006-0.0-0.15	0.0-0.15	0.086	
BW16SR-006-0.27-0.52	0.15-0.50	0.019	J
BW16SR-007-0.0-0.15	0.0-0.15	0.052	
BW16SR-007-0.06-0.31[1]	0.0-0.15 and 0.15-0.50	0.11	
BW16SR-008-0.0-0.15	0.0-0.15	0.072	
BW16SR-008-0.23-0.48	0.15-0.50	0.54	
BW16SR-009-0.0-0.15	0.0-0.15	0.033	
BW16SR-009-0.17-0.42	0.15-0.50	0.072	
BW16SR-010-0.0-0.24	0.0-0.15 and 0.15-0.50	0.099	
BW16SR-011-0.0-0.15	0.0-0.15	0.082	
BW16SR-011-0.16-0.41	0.15-0.50	0.12	
BW16SR-012-0.0-0.25	0.0-0.15 and 0.15-0.50	0.35	J
BW16SR-013-0.0-0.15	0.0-0.15	0.16	
BW16SR-013-0.11-0.36	0.15-0.50	0.34	
BW16SR-014-0.0-0.15	0.0-0.15	0.045	
BW16SR-015-0.0-0.15	0.0-0.15	0.047	
BW16SR-016-0.15-0.60	0.15-0.50	0.28	

Notes:
[1]25% of the sample was collected within two intervals, the sample was evaluated for both intervals, as described in ***Section 2.6.3****.*
J = estimated value
Values highlighted in yellow indicate concentration exceeding SQT Level I (0.18 mg/kg).
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II (0.64 mg/kg).
Values highlighted in red indicate concentration exceeding SQT Level II (1.1 mg/kg).

3.2.2 Dioxins/Furans (Sediment)

The following table summarizes the TEQ results for Site sediment samples with respect to the dioxin TEQ KM for aquatic health (fish), calculated as described in **Section 2.6.1** and compared to the respective SQTs.

Sample Name	Sample Interval (meters)	TEQ KM Fish Results	Result Qualifier
BW16SR-001-0.0-0.15	0.0-0.15	52.1	
BW16SR-002-0.0-0.15	0.0-0.15	44.46	
BW16SR-003-0.0-0.15	0.0-0.15	36.15	
BW16SR-004-0.0-0.15	0.0-0.15	45.03	
BW16SR-005-0.0-0.15	0.0-0.15	37.57	
BW16SR-006-0.0-0.15	0.0-0.15	9.74	J
BW16SR-006-0.27-0.52	0.0-0.15 and 0.15-0.50	0.27	J
BW16SR-007-10.0-0.15	0.0-0.15	4.10	J
BW16SR-007-0.06-0.31[1]	0.0-0.15 and 0.15-0.50	292.87	J
BW16SR-008-0.0-0.15	0.0-0.15	6.59	J
BW16SR-008-0.23-0.48	0.0-0.15 and 0.15-0.50	96.04	
BW16SR-009-0.0-0.15	0.0-0.15	1.28	J
BW16SR-009-0.17-0.42	0.0-0.15 and 0.15-0.50	5.55	J
BW16SR-010-0.0-0.24	0.0-0.15 and 0.15-0.50	52.00	
BW16SR-011-0.0-0.15	0.0-0.15	9.53	J
BW16SR-011-0.16-0.41	0.0-0.15 and 0.15-0.50	25.98	
BW16SR-012-0.0-0.25	0.0-0.15 and 0.15-0.50	49.51	J
BW16SR-013-0.0-0.15	0.0-0.15	73.95	
BW16SR-013-0.11-0.36	0.0-0.15 and 0.15-0.50	39.48	
BW16SR-014-0.0-0.15	0.0-0.15	1.26	J
BW16SR-015-0.0-0.15	0.0-0.15	1.74	J
BW16SR-016-0.15-0.60	0.0-0.15 and 0.15-0.50	139.49	

Notes:
[1]25% of the sample was collected within two intervals, the sample was evaluated for both intervals, as described in ***Section 2.6.3****.*
ng TEQ/kg = nanograms of dioxin toxicity equivalent per kilogram
Values highlighted in yellow indicate concentration exceeding SQT Level I (0.85 ng TEQ/kg).
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II (11.2 ng TEQ/kg).
Values highlighted in red indicate concentration exceeding SQT Level II (21.5 ng TEQ/kg).
TEQ values calculated using the USEPA Advanced KM TEQ Calculator with 1998 WHO TEFs for fish.
Dioxins analyzed by EPA Method SW8290.

For TEQ KM Fish, Level 1 SQT exceedances occurred in multiple samples. Level II exceedances occurred in BW16SR-001 through BW16SR-005, and BW16SR-013 in surface sediments. Level II exceedances in the 0.0 to 0.15 and 0.15 to 0.50-meter depth occurred at BW16SR-007, BW16SR-008, BW16SR-010 through BW16SR-013, and BW16SR-016. The maximum concentration of TEQ KM Fish (292.87 ng TEQ/kg) was identified in the 0.0 to 0.15 and 0.15 to 0.50-meter intervals at location BW16SR-007.

3.3 Physical Sediment Characterization

Surface sediment samples collected at the Site generally contained dark brown to very dark brown silt loam, consisting of up to 10% fibrous woody debris.

Deeper sediment samples collected at the Site, up to a maximum depth of 0.52 meters, generally contained brown to dark brown sandy loam and dark brown to very dark brown silty loam, consisting of up to 50% fibrous woody debris. In sample locations BW16SR-006, BW16SR-007, BW16SR-011, and BW16SR-012, the silty loam was underlain by a peat layer up to a depth of 0.41 meters. A firm dark grey brown clay to silty clay was observed within the bottommost sediments in core samples collected from locations BW16SR-006, BW16SR-009, and BW16SR-011. Based on the depth of sampler advancement at these locations, the dark grey brown silty clay layer depth varied between 0.18 meters bss at location BW16SR-011 and 0.24 meters at location BW16SR-006.

3.3.1 Grain Size

Grain size distribution was analyzed in 100% of sample locations to meet site investigation objectives presented in the FSP. The following table summarizes this grain size analysis. Grain size distribution charts are presented in laboratory analytical reports included in **Appendix D**.

Sample ID Depth Interval (meters)	Soil Classification	Percent +3 inches	Percent Gravel		Percent Sand			Percent Fines		d10
			Coarse	Fine	Coarse	Medium	Fine	Silt	Clay	Percent Finer
BW16SR-001 (0.0–0.15)	Silt	0	0	0	0	1	41	70	15	100.0
BW16SR-002 (0.0–0.15)	Silty Sand	0	0	3	3	7	41	53	18	100.0
BW16SR-003 (0.0–0.15)	Silt with Sand	0	0	0	0	1	17	65	17	100.0
BW16SR-005 (0.0–0.15)	Silty with Sand	0	0	0	0	1	17	64	18	100.0
BW16SR-006 (0.0–0.15)	Silt	0	0	0	0	1	12	65	22	100.0
BW16SR-006 (0.27–0.52)	Silt with Sand	0	0	0	0	1	28	53	18	100.0
BW16SR-007 (0.0–0.15)	Silty Sand	0	0	0	0	8	65	24	3	100.0
BW16SR-007 (0.06–0.31)	Silty Sand	0	0	1	1	13	53	26	6	98.0
BW16SR-008 (0.0–0.15)	Silt	0	0	0	0	1	11	79	9	100.0
BW16SR-008 (0.23–0.48)	Silt with Sand	0	0	0	0	2	24	60	14	100.0
BW16SR-009 (0.0–0.15)	Silty Sand	0	0	0	0	4	56	34	6	100.0
BW16SR-009 (0.17–0.42)	Silty with Sand	0	0	0	0	2	46	44	8	100.0
BW16SR-010 (0.0–0.24)	Sandy Silt	0	0	1	2	5	40	41	11	97.0
BW16SR-011 (0.0–0.15)	Sandy Silt	0	0	0	1	2	38	49	10	99.0

Sample ID Depth Interval (meters)	Soil Classification	Percent +3 inches	Percent Gravel		Percent Sand			Percent Fines		d10
			Coarse	Fine	Coarse	Medium	Fine	Silt	Clay	Percent Finer
BW16SR-011 (0.16–0.41)	Sandy Silt	0	0	0	0	2	29	59	10	100.0
BW16SR-012 (0.0–0.25)	Silt	0	0	0	0	2	11	62	25	100.0
BW16SR-013 (0.0–0.15)	Sandy Silt	0	0	4	3	7	35	42	9	93.0
BW16SR-013 (0.11–0.36)	Sandy Silt	0	0	0	1	2	45	42	10	100.0
BW16SR-014 (0.0–0.15)	Silt with Sand	0	0	0	0	1	27	60	12	100.0
BW16SR-015 (0.0–0.15)	Sandy Silt	0	0	0	0	3	47	39	11	100.0

3.3.2 Total Organic Carbon

TOC analyses were performed on all sediment samples collected. A summarized results table is presented as follows, a full table with TOC results summarized can be found in **Table 6**.

TOC results ranged from 7,090 to 60,100 mg/kg; the average TOC value was 68,7190 mg/kg.

Sample Name	Sample Depth Start (meters)	Sample Depth End (meters)	Result (mg/kg)
BW16SR-001-0.0-0.15	0	0.15	39500
BW16SR-002-0.0-0.15	0	0.15	47100
BW16SR-003-0.0-0.15	0	0.15	31400
BW16SR-004-0.0-0.15	0	0.15	39500
BW16SR-005-0.0-0.15	0	0.15	33900
BW16SR-006-0.0-0.15	0	0.15	27000
BW16SR-006-0.27-0.52	0.27	0.52	7090
BW16SR-007-0.0-0.15	0	0.15	19700
BW16SR-007-0.06-0.31	0.06	0.31	33300
BW16SR-008-0.0-0.15	0	0.15	25700
BW16SR-008-0.23-0.48	0.23	0.48	48300
BW16SR-009-0.0-0.15	0	0.15	11500
BW16SR-009-0.17-0.42	0.17	0.42	20200
BW16SR-010-0.0-0.24	0	0.24	51700
BW16SR-011-0.0-0.15	0	0.15	22900
BW16SR-011-0.16-0.41	0.16	0.41	30900

Sample Name	Sample Depth Start (meters)	Sample Depth End (meters)	Result (mg/kg)
BW16SR-012-0.0-0.25	0	0.25	60100
BW16SR-013-0.0-0.15	0	0.15	54500
BW16SR-013-0.11-0.36	0.11	0.36	18200
BW16SR-014-0.0-0.15	0	0.15	14300
BW16SR-015-0.0-0.15	0	0.15	13900
BW16SR-016-0.15-0.60	0.15	0.6	36500

Notes:
mg/kg = milligrams/kilogram

3.4 Tissue Chemistry Data

The following discussion presents the summarized analytical results from samples obtained from six locations (BW16SR-HD-001, BW16SR-001 through BW16SR-005, and BW16SR-016) collected during the September and October 2016 sampling event at the Site.

Laboratory analyses and sampling frequency selected for tissue samples collected at the Site include the following:

- Mercury by SW-846 7471B (100%);
- Methyl Mercury by EPA 1630 (100%); and
- Dioxins and furans by SW-846 8290A (100%).

Tissue samples were either collected from in situ benthic macroinvertebrates from pre-selected sample locations or were grown ex situ in the lab from sediment collected from the sample location. The following tables specify whether tissue was in situ or ex situ and which species of benthic macroinvertebrate was sampled. **Table 1** provides a summary of tissue samples and laboratory analyses selected for each sample. Analytical results are presented in **Table 10** through **Table 12**, and laboratory analytical reports are included in **Appendix D**. **Figure 7** through **Figure 8** present bioaccumulation data. The following sections present a summary of analytical results and calculations.

3.4.1 Mercury and Methylmercury (Tissue)

3.4.1.1 *Benthic Macroinvertebrate Tissue*

The following table summarizes the sample results for the total observed range of mercury and methylmercury for Site in situ benthic macroinvertebrate tissue samples.

In Situ Benthic Macroinvertebrate Tissue				
Species	**Mercury (mg/kg)**		**Methylmercury (µg/kg)**	
	Range	**Average**	**Range**	**Average**
Mayfly	0.031–0.036	0.034	3.1–4.5	3.63
Dragonfly	0.029–0.030	0.0295	23.0–25.0	24.0
Crayfish	0.030	0.030	18.0	18.0

In Situ Benthic Macroinvertebrate Tissue				
Species	**Mercury (mg/kg)**		**Methylmercury (µg/kg)**	
	Range	**Average**	**Range**	**Average**
HD[b] Sample[a]	0.037	0.037	4.5	4.5
Site Average[a]	0.0326–0.034	0.0333	9.768–10.464	10.081
Reference Sample–Boulder Lake				
HD[a] Sampler*[a]	0.032	0.032	4.3	4.3

Notes:
[a]Only one sample analyzed, data range and average were not applicable
[b]Hester Dendy Sampler
**Sample weight was subsidized with additional macroinvertebrates sampled from Boulder Lake by Bay West*
µg/kg = micrograms per kilogram

Concentrations of mercury in in situ tissue from various organism appear to be similar. Average mercury concentrations for in situ tissue observed for all species (Site Average) as compared to the reference sample also appear to be similar. These results indicate that organisms do not appear to be affected differently by mercury in Site sediments when compared to reference site sediments.

Concentrations of methylmercury in in situ tissue was observed to have greater variation between species. Methylmercury concentrations in the Site HD sample and the reference HD sample appear to be similar. The remaining samples from varying benthic macroinvertebrate species as compared to the reference HD sampler have much greater variation, indicating that bioaccumulation results are likely impacted by sampling methods, contaminate distribution, and benthic macroinvertebrate species type and possibly life cycle stage. Due to the variety in species and sampling methods between the Site and reference site, a one-way ANOVA test was not performed.

The following table summarizes the sample results for the total observed range of mercury and methylmercury for Site ex situ Lumbriculus variegatus tissue samples.

Ex Situ Benthic Macroinvertebrate Tissue						
Species	**Number of Sample Locations**	**Duration of Bioaccumulation Test (days)**	**Mercury (mg/kg)**		**Methylmercury (µg/kg)**	
			Range	**Average**	**Range**	**Average**
Lumbriculus variegatus[1]	4	28	0.036–0.037	0.0365	0.24–0.32	0.28
Reference Sample–Boulder Lake						
Lumbriculus variegatus[2]*	1	28	0.038	0.038	0.15	0.15
Reference Sample–Background (Day 0)						
Lumbriculus variegatus[3]*	1	0	0.038	0.038	0.088	0.088

Notes:
**Only one sample analyzed, data range and average were not applicable*
[1]Lab grown in Site sediment samples
[2]Lab grown in Boulder Lake sediment samples
[3]Lab grown in lab supplied sediment samples

For ex situ, benthic macroinvertebrate organism life cycle stage and species type are controlled under laboratory conditions. As observed in in situ samples average mercury concentrations in

tissue appears to be comparable between the Site and the reference samples, indicating that benthic organisms do not appear to be bioaccumulating mercury due to exposure to Site sediments.

Methylmercury in tissue from organisms exposed to Site sediments appears to be an order of magnitude greater than the background day 0 sample and double the reference sample, indicating that benthic organisms may be bioaccumulating methylmercury due to exposure to Site sediments. Due to the variety in species and sampling methods between the Site and reference site, a one-way ANOVA test was not performed.

3.4.1.2 *Fish Tissue*

The following table summarizes mercury and methylmercury results for Site fish tissue samples by fish species and trophic level.

Mercury and methylmercury concentrations in fish tissue appear to coincide with trophic level for both Site fish tissue samples and reference samples. Trophic Level 4, carnivorous fish, appear to have the greatest levels of mercury and methylmercury with the exception of the smallmouth bass. The smallmouth bass has the highest concentration of mercury and methylmercury in comparison to the other fish sampled for Site data. The trophic level associated with the smallmouth bass is 3.6, the upper half of trophic Level 3.

Fish Tissue							
Fish Species	**Number of Fish**	**Total Weight of Fish (g)**	**Trophic Level**	**Mercury (mg/kg)**		**Methylmercury (µg/kg)**	
				Range	**Average**	**Range**	**Average**
Walleye[1]	NR	61	4.5	0.12	0.12	110	110
Northern Pike	3	2383	4.1	0.12–0.13	0.125	100–110	105
Yellow Perch	8	1247	3.7	0.079–0.092	0.086	74–98	87
Smallmouth Bass	9	3917	3.6	0.071–0.22	0.164	110–230	170
White Sucker	9	5283	2.8	0.067–0.083	0.075	58–96	83
Shiner Mix'	2	827	2.1	0.054	0.054	41	41
Reference Sample–Boulder Lake							
Walleye	10	420	4.5	0.098–0.13	0.120	130–140	136.7
Black Clappie'	6	116	3.8	0.68	0.68	53	53
Yellow Perch	26	841	3.7	0.068–0.077	0.073	54–65	58
Rock Bass'	9	208	3.4	0.077	0.077	76	76
White Sucker	9	9289	2.8	0.051–0.071	0.059	57–110	82.67
Shiner Mix	12	467	2.1	0.064–0.071	0.068	62–65	63

Notes:
[1]Only one sample analyzed, data range and average were not applicable
NR = Not Recorded

Bioconcentration of mercury and methyl mercury appear to increase as trophic level increases, consistent with common understanding of bioconcentration in fish trophic levels. Bottom feeding fish, lower trophic levels, accumulate less contaminant. As trophic level increases, fish are more predatory, eating lower trophic level fish and accumulating higher concentrations of contaminant.

A Classical Oneway ANOVA test was completed using fish tissue concentrations to determine if there is a statistically significant difference between tissue concentrations from Site fish and

reference area Fish, for any given trophic level. The following table summarizes the mercury and methylmercury Classical Oneway ANOVA test results for Site and reference fish tissue samples by fish species and trophic level, calculated as described in **Section 2.6.1**.

Fish Tissue			
Fish Species	**Trophic Level**	**Mercury (p-value)**	**Methylmercury (p-value)**
Walleye	4.5	NC	NC
Northern Pike[a]	4.1		
Black Clappie	3.8	0.119	**0.0347**
Yellow Perch	3.7		
Rock Bass	3.4		
White Sucker	2.8	0.362	0.983
Shiner Mix	2.1		

Notes:
[a]Fish species was only collected from SR.
***Bold** values indicate statistically significant difference between site trophic level species and reference area trophic level species.*
NC = Not Calculated. Insufficient data to complete the Classical Oneway ANOVA statistics tests.
A p-value ≤ 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance.
A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

Mercury concentrations appear to be statistically similar to the reference sample and coincide trophic level for both Site fish tissue samples and reference samples.

Methylmercury concentrations were observed to have greater statistical variation between trophic levels. Trophic Level 2, bottom feeders, appear to be comparable between Site fish tissue samples and reference samples. Tropic Level 3 fish showed statistically significant differences between fish collected from the Site and reference Site. Although, the fish tissue mercury and methylmercury concentrations within trophic Level 4 were not able to be statistically calculated, due to insufficient data, the significant differences between fish collected from the Site and reference indicate that methylmercury appears to be bioaccumulating more in fish at the Site compared to the reference area. The Classical Oneway ANOVA statistics tests are included in **Appendix E**.

3.4.2 Dioxins/Furans (Tissue)

3.4.2.1 *Benthic Macroinvertebrate Tissue*

The following tables summarize the total observed range of dioxins/furans (as TEQ Fish) for Site in situ benthic macroinvertebrate tissue samples (**Section 2.6.1**).

In Situ Benthic Macroinvertebrate Tissue			
Benthic Macroinvertebrate Species	**Number of Samples Locations**	**TEQ Fish (ng TEQ/kg)**	
		Range	**Average**
Mayfly	3	0.33–1.48	0.98
Dragonfly	3	–0.28-0.55	0.38
Crayfish[1]	1	0.43	0.43
HD[2] Sampler	1	NA	NA
Site Average[a]	-	-	0.61
Reference Sample – Boulder Lake			
HD[a] Sampler[1]*	1	-	0.11

Notes:
[1]Only one sample analyzed, data range and average were not applicable.
[2]Hester Dendy Sampler
**Sample weight was subsidized with additional macroinvertebrates sampled from Boulder Lake by Bay West.*
** TEQ values calculated using the USEPA Advanced KM TEQ Calculator with 1998 WHO TEFs for fish.*

The Site TEQ Fish average is over double the reference sample although, as previously discussed, data comparisons between the reference HD sampler and specific benthic macroinvertebrate species rather than a direct comparison between HD samplers may be skewed. These results indicate that dioxins/furans may be bioaccumulating more in benthic organisms at the Site than the references site. No data is available for comparisons between Site HD sample and the reference HD Sample due to a lack of available tissue for analysis in the Site HD sample. Due to the variety in species and sampling methods between the Site and reference site, a one-way ANOVA test was not performed.

The following table summarizes the total observed range of range of dioxins/furans (as TEQ Fish) for Site ex situ Lumbriculus variegatus tissue samples.

Ex Situ Benthic Macroinvertebrate Tissue				
Benthic Macroinvertebrate Species	**Number of Samples Locations**	**Duration of Bioaccumulation Test (days)**	**TEQ Fish (ng TEQ/kg)**	
			Range	**Average**
Lumbriculus variegatus[1]	2	28	0.98–3.98	2.48
Reference Sample – Boulder Lake				
Lumbriculus variegatus[2]	1	28	0.09	009
Reference Sample – Background				
Lumbriculus variegatus[3]	1	0	0.06	0.06

Notes:
[1]Lab grown in Site sediment samples.
[2]Lab grown in Boulder Lake sediment samples.
[3]Lab grown in lab supplied sediment samples.
** TEQ values calculated using the USEPA Advanced KM TEQ Calculator with 1998 WHO TEFs for fish.*

Ex situ average TEQ Fish for the Site is an order of magnitude greater than both the reference sample and background day 0 sample, indicating that benthic organisms exposed to site sediments likely bioaccumulate dioxins/furans.

3.4.2.2 *Fish Tissue*

The following table summarizes dioxin/furans concentrations [as TEQ calculation results for Fish TEFs (TEQ Fish) and Human Health TEFs (TEQ HH), calculated as described in **Section 2.6.1** detected in Site fish tissue samples and reference site samples.

Fish Tissue							
Fish Species	**Total Number of Fish**	**Total Weight of Fish (g)**	**Trophic Level**	**TEQ Fish (ng TEQ/kg)[2]**		**TEQ HH (ng TEQ/kg)[3]**	
				Range	**Average**	**Range**	**Average**
Walleye[1]	NR	61	4.5	0.32	0.32	0.33	0.33
Northern Pike[1]	3	2383	4.1	0.34	0.34	0.30	0.30
Yellow Perch	8	1247	3.7	0.39 – 0.67	0.54	0.26 – 0.51	0.41
Smallmouth Bass	9	3917	3.6	0.63-0.93	0.80	0.56-0.81	0.70
White Sucker	9	5283	2.8	0.53-1.25	091	0.47-1.02	0.73
Shiner Mix[1]	2	827	2.1	0.10	0.10	0.11	0.11
Reference Sample–Boulder Lake							
Walleye	10	420	4.5	0.05-0.16	0.09	0.05-0.18	0.09
Black Clappie[1]	6	116	3.8	0.05	0.05	0.05	0.05
Yellow Perch	26	841	3.7	0.05-0.07	0.06	0.04-0.07	0.06
Rock Bass[1]	9	208	3.4	0.06	0.06	0.06	0.06
White Sucker	9	9289	2.8	0.06-0.11	0.11	0.06-0.11	0.11
Shiner Mix	12	467	2.1	0.04-0.49	0.20	0.04-0.50	0.20

Notes:
[1]Only one sample analyzed; data range and average were not applicable.
[2] TEQ values calculated using the USEPA Advanced KM TEQ Calculator with 1998 WHO TEFs for fish.
[3] TEQ values calculated using the USEPA Advanced KM TEQ Calculator with 2005 WHO TEFs for human health.

TEQ fish values for fish tissue samples from the Site are typically greater than TEQ Fish values for fish tissue samples from the reference site. Dioxin/furan concentrations in trophic Level 3 species exceeds concentrations in Level 4 and Level 2 species for both the Site and the reference site. This may be attributed to a preferential uptake of dioxin/furans in fish species at trophic Level 3.

TEQ HH values for the Site are greater than those at the reference site by an order of magnitude; however, this difference is due to the method in which non-detect values were treated. TEQ. For the Site, trophic Level 3 exceeds Level 4 and Level 2 values. For the reference site, trophic Level 2 exceeds Level 3 and Level 4 values.

Overall, dioxin/furan concentrations in fish collected from the Site are greater than reference site, indicating conditions at the Site are resulting in the bioaccumulation of dioxins/furans in fish at the Site.

A Classical Oneway ANOVA test was completed using fish tissue concentrations to determine if there is a statistically significant difference between tissue concentrations from Site fish and reference area Fish, for any given trophic level. The following table summarizes the TEQ Fish and TEQ HH Classical Oneway ANOVA test results for Site and reference fish tissue samples by fish species and trophic level, calculated as described in **Section 2.4**.

Fish Tissue			
Fish Species	**Trophic Level**	**TEQ Fish (p-value)**	**TEQ HH (p-value)**
Walleye	4.5	NC	NC
Northern Pike[1]	4.1		
Black Crappie	3.8	**0.00006743**	**0.0003300**
Yellow Perch	3.7		
Rock Bass	3.4		
White Sucker	2.8	**0.0341**	**0.0453**
Shiner Mix	2.1		

Notes:
[1]Only one sample analyzed; data range and average were not applicable.
***Bold** values indicate statistically significant difference between site trophic level species and reference area trophic level species.*
NC = Not Calculated. Insufficient data to complete the Classical Oneway ANOVA statistics tests.
A p-value ≤ 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance.
A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

Concentrations of dioxins/furans in fish tissue have a statistically significant difference between fish collected from the Site and reference Site in tropic levels 2 and 3. Although, fish tissue dioxin/furans concentrations within trophic Level 4 were not able to be statistically calculated, due to insufficient data, the significant differences between fish collected from the Site and reference Site indicate that conditions at the Site are resulting in the bioaccumulation of dioxins/furans in fish at the Site. The Classical Oneway ANOVA statistics tests are included in **Appendix E**.

3.5 Community Assessment Comparison Data

Community assessments were completed as described in **Section 2.4**. A summarized results table is presented as follows, the full table with specific benthic macroinvertebrate species identified can be found in **Table 7**.

Location	Collection Information				Biotic Index Score[2]	Biotic Health Score[3]
	Date	Number of Ponar Grabs	Approximate Collection Area (cm)[1]	Community Assessment Duration (min)		
BW16SR-001	9/21/2016	3	675	15	**1.6**	**Poor**
BW16SR-002	9/28/2016	3	675	15	**1.3**	**Poor**
BW16SR-003	9/22/2016	3	675	15	**1.7**	**Poor**
BW16SR-004	9/28/2016	3	675	15	**1.0**	**Poor**
BW16SR-005	9/28/2016	3	675	15	**1.0**	**Poor**
Boulder Lake Reservoir (Reference Sample)						
BW16BLR-001	9/20/2016	3	675	15	**0.0**	**Poor**

Notes:
[1]Each grab = 15.2 cm x 15.2 cm (225 cm^2)
[2]Biotic Index Score Calculation: http://watermonitoring.uwex.edu/pdf/level1/datasheets/data-Biotic2014.pdf
[3]Biotic Health Score: Good: 2.6–2.5, Fair: 2.1–2.5, and Poor: 2.0–1.0
cm = centimeters
min = minutes

4.0 DATA QUALITY REVIEW

4.1 Analytical Data QA/QC Review

In accordance with the St. Louis River Sediment AOC QAPP dated July 2014 and the QAPP Addendum dated February 2015, data verification was performed on the following organic and inorganic analyses: mercury, methylmercury, dioxin/furans, and TOC. A cursory review was performed on grain size. All data was collected and samples were analyzed by Pace, Axys, or GLEC, Minnesota Department of Health Environmental Laboratory Accreditation Program (ELAP)-accredited laboratories. The following table describes methods and percentage of total samples for each parameter.

Parameter	Media[b]	Total Samples	Percentage of Total Sampled	Analytical Method
Mercury	Sediment	22	100%	SW-846 Method 7471B
Dioxins/Furans	Sediment	22	100%	SW-846 Method 8290A
TOC	Sediment	22	100%	SW-846 Method 9060A
Grain size	Sediment	20	100%	ASTM D422
Percent Moisture	Sediment	22	100%	ASTM D2974-07
Total Mercury	Benthic Tissue	8[a]	100%	SW-846 7471B
Methylmercury	Benthic Tissue	8 [a]	100%	EPA Method 1630
Dioxins/Furans	Benthic Tissue	8 [a]	100%	SW-846 Method 8290A

Notes:
a = One bioaccumulation sample is a composite of samples 001, 003, 004, 005, and 007 due to limited sample mass.
b = Fish samples were not included in this table because the MPCA and USACE collected the fish samples and the USEPA was responsible for the QA/QC for fish tissue analysis.

In general, the areas covered by the data verification process included reviewing the following:

- CoC records;
- Technical holding times and preservation;
- Laboratory and field QC reporting forms (method blanks, rinsate blanks, surrogates, laboratory control samples [LCSs], laboratory control sample duplicates [LCSDs], and MS/MSDs, as appropriate);
- Required analytical methods;
- Reporting limits;
- Case narrative;
- Completeness of Results; and
- Data usability (compliance with data quality objectives [DQOs]).

Level II Laboratory reports were provided by the laboratory and reviewed, so the following areas were not covered by the data verification:

- Tune summaries (gas chromatograph/mass spectrometer only);
- Initial calibrations;
- Continuing calibrations;
- Internal standards;

- Target compound/analyte ID;
- Target Compound/analyte quantitation; and
- System performance.

As per the approved QAPP, data verification was performed by a Bay West Chemist and documented using the MPCA Laboratory Data Review Checklist. Data verification was performed by comparing the contents of the data packages and QA/QC results to the requirements in the QAPP, the respective analytical methods, and the laboratory SOPs. Additional qualifiers were added, as needed, and summarized in the MPCA Laboratory Data Review Checklists, included in **Appendix D**. All metals samples analyzed by SW-846 Method 6020A were analyzed at 20-fold dilution in accordance with the Pace SOP.

Field duplicates, MS/MSDs, method blanks, and rinsate blanks were collected and/or analyzed at required frequencies specified in the approved QAPP as follows. Field duplicates met or exceeded the required frequencies of 10% for the samples analyzed for mercury and dioxin/furans. MS analysis met or exceeded the required frequency of 5% for mercury and dioxin/furans. Rinsate blanks were collected daily (see **Section 2.5.2** for additional discussion), for mercury only as discussed in Section 4.6.4 of the FSP. Analytes detected in samples at concentrations less than 10% of the method blank or rinsate blank concentrations were qualified "U" as undetected.

Samples results were considered estimated if the sample results were associated with LCSs/LCSD or MS/MSD recoveries outside QC limits. When LCS or MS/MSD recoveries were biased low, both detected and undetected sample results were flagged with a "J" or "UJ" to indicate that the concentration or reporting limit is considered estimated. When LCS or MS/MSD recoveries were biased high, only the detected results were qualified "J" as estimated. Only detected results were qualified "J" when relative percent differences were high in field duplicates, MS/MSDs, and LCS/LCSDs. All non-detect values were flagged with a "U."

4.2 Interpretation of Concentrations Less Than Detection Limits

The MPCA Guidance: Laboratory Quality Control and Data Policy requires concentrations less than the reporting limit but above the method detection limits (MDLs) to be qualified with a "J" because they are considered estimated. Samples below the MDL were qualified with a "U." Bay West replaced all "E," "I," and "P" Pace qualifiers with a "J" flag to indicate that the sample concentrations are considered estimated.

Since guidance for calculations of toxicity quotients do not prescribe which scaling factor for non-detect results should be used, toxicity quotients were calculated for dioxin/furans using the methods described in **Section 2.6.1**.

4.3 Summary

Overall, no significant data quality discrepancies were observed. All data were verified and found acceptable, as qualified, and met DQOs. Additional information regarding data verification can be found in Laboratory Data Review Checklists in **Appendix D**.

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5.0 DISCUSSION AND CONCLUSION

The following section describes the results obtained during the limited field activities.

All Community Assessment comparisons completed for BW16SR–001 through BW16SR–005 and BW16BLR–001 (reference site) surface sediments indicated that the benthic community health at these locations was poor to fair. Macroinvertebrate species diversity was low and species consisted only of pollutant tolerant and semi-tolerant macroinvertebrates for the Site and the reference Site. This assessment was completed at the very end of the organism growing season, which likely skewed the outcome of the assessment; therefore, this data is considered inconclusive. Additional assessments would need to be completed during the growing season to develop a more definitive conclusion of benthic community quality at these locations.

Sediment samples were collected and analyzed for Site COCs to gather additional chemical data for delineation of extent and depth of contamination at the Site. Mercury sediment concentrations did not exceed Midpoint SQTs with the exception of one sample, indicating mercury contamination appears to be relatively limited throughout the Site. Dioxin/furan sediment concentrations exceeded Level II SQTs in 60% of the samples, focused within the back bay of the eastern half of SR. Exceedances were observed in both the 0.0 to 0.15 and 0.15 to 0.50-meter intervals indicating that deposition of contaminated sediment may still be occurring, or that sediment deposition in this area is minimal.

In situ macroinvertebrate tissue collection was completed at BW16SR-001 through BW16SR-005, and BW16SR-HD-001-MRCS, excluding BW16SR-004. Ex situ laboratory bioaccumulation testing was completed using sediment collected from BW16SR-004 and BW16SR-016.

Benthic macroinvertebrates do not appear to bioaccumulate mercury due to exposure to Site sediments significantly more compared to reference samples. Benthic macroinvertebrate tissue results for mercury for both in situ and ex situ samples were not observed to vary greatly between Site samples and reference/background samples. Based on these results, it appears that mercury would not migrate up the food chain to higher trophic levels significantly more than reference sites.

Benthic macroinvertebrates appear to bioaccumulate methylmercury due to exposure to Site sediments more compared to reference samples. The Site average for in situ methylmercury tissue concentration was over double the reference Site. Additionally, concentrations varied between in situ species sampled, indicating that some organisms are bioaccumulating methylmercury more than others; however, methylmercury concentration variations in in situ tissue may be attributed to contaminant distribution, benthic species type and lifecycle, and age. Methylmercury in tissue from organisms exposed to Site sediments under controlled laboratory conditions (ex situ tissue) also appears to be an order of magnitude greater than the background day 0 sample and double the reference sample, indicating that benthic organisms may be bioaccumulating methylmercury due to exposure to Site sediments. Methylmercury in site sediments appear to bioaccumulate in benthic tissue, indicating that this contaminant may migrate up the food chain to higher trophic levels significantly more than reference sites; however, there is limited data for methylmercury concentrations in sediment at the Site.

Fish tissue collection and testing of seven fish species within trophic levels 2 through 4, was completed by the MCPA, at the Site and reference Site. Concentrations of mercury in fish tissue were not observed to vary greatly between the Site and the reference Site. Statistically, mercury concentrations appear to be comparable to the reference sample and coincide trophic level for both Site fish tissue samples and reference samples. Concentrations of methylmercury in fish tissue were observed to have a statistically significant difference between fish collected from the

Site and the reference site within tropic Level 3. Bioconcentration of mercury and methylmercury appear to increase as trophic level increases, indicating that both mercury and methylmercury are bioconcentrating; however, methylmercury appears to be doing so significantly more at the Site compared to the reference site. Although methylmercury appears to be bioconcentrating in the fish tissue at the Site, there is limited data for methylmercury concentrations in sediment at the Site.

Benthic macroinvertebrates appear to bioaccumulate dioxins/furans due to exposure to Site sediments significantly more compared to reference samples. The dioxins/furans concentration in in situ tissue is over double the reference sample. Dioxins/furans in tissue from organisms exposed to Site sediments under controlled laboratory conditions (ex situ tissue) are over an order of magnitude greater than background results. These results indicate that these dioxins/furans may migrate up the food chain to higher trophic levels significantly more than reference sites.

Fish tissue results for dioxins/furans at the Site and the reference Site with results that showed trophic Level 3 exceeding Level 4 and Level 2 values for both the Site and the reference site. This may be attributed to a preferential uptake of dioxin/furans in fish species at trophic Level 3. Dioxins/furans concentrations in fish tissue were greater in tissue collected from the Site compared to the reference site and concentrations have a statistically significant difference in all trophic levels between Site samples and reference Site samples. Bioconcentration of dioxins/furans appear to increase as trophic level increases, indicating that dioxins/furans are bioconcentrating significantly more at the Site compared to the reference site.

Based on the sediment and tissue testing results, dioxins/furans should be retained as a COC for the Site. Methylmercury also appears to be bioconcentrating in tissue at the Site more than reference site and/or reference samples; however, there is insufficient analytical data to determine whether methylmercury should be considered a COC for the Site. Additionally, future sediment investigations and/or potential Site remedies may require analysis of methylmercury.

6.0 REFERENCES

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Tables

Table 1 - Sample Analysis Summary
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Location ID	Sample ID	Sediment											Tissue								
		Chemical/Physical							Toxicity		Bioaccumulation										
		Sample Interval (m)	Sample Type (G or C)	Dioxins and furans by SW-846 8290A	Mercury by SW-846 7471B	TOC by SW-846 9060A	Grain size by ASTM D422	Percent moisture by ASTM D2216	10-d	28-d	28-d	Community Assessment	In Situ (Mayfly, Dragonfly, & Crawfish)	In Situ Hester Dendy (Macrobenthos & Crawfish)	Laboratory Exposed (Lumbriculus)	Grams Collected	Dioxins and furans by SW-846 8290A	Mercury by SW-846 7471B	Methyl Mercury EPA 1630	% LIPIDS	Type
BW16SR-001	BW16SR-HD-001-MCRS	0.0-0.15	C										Mayfly			1.5		X	X		Composite (001, 007, 003, 004, & 005)
	BW16SR-001-0.0-0.15-M	0.0-0.15	G	X	X	X	X	X					Mayfly			40	X	X	X	X	
	BW16SR-001	0.0-0.15	G									X									
BW16SR-002	BW16SR-002-0.0-0.15-M	0.0-0.15	G	X	X	X	X	X					Mayfly			52	X	X	X	X	
	BW16SR-002	0.0-0.15	G									X									
	BW16SR-002-0.0-0.15-D	0.0-0.15	G										Dragonfly			40	X	X	X	X	
	BW16SR-102-0.0-0.15-D	0.0-0.15	G										Dragonfly			12	X			X	
BW16SR-003	BW16SR-003-0.0-0.15-M	0.0-0.15	G	X	X	X	X	X					Mayfly			38	X	X	X	X	
	BW16SR-003	0.0-0.15	G									X									
	BW16SR-103-0.0-0.15-M	0.0-0.15	G										Mayfly			10		X	X		
	BW16SR-003-0.0-0.15-D	0.0-0.15	G										Dragonfly			16	X			X	
BW16SR-004	BW16SR-004-0.0-0.15	0.0-0.15	G	X	X	X		X			X				X						
	BW16SR-004	0.0-0.15	G									X									
BW16SR-005	BW16SR-005-0.0-0.15-D	0.0-0.15	G	X	X	X	X	X					Dragonfly			36	X	X	X	X	
	BW16SR-005-0.0-0.15-C	0.0-0.15	G										Crawfish			35	X	X	X	X	
	BW16SR-005	0.0-0.15	G									X									
BW16SR-006	BW16SR-006-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-006-0.27-0.52	0.27-0.52	G	X	X	X	X	X													
BW16SR-007	BW16SR-007-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-007-0.06-0.31	0.06-0.31	G	X	X	X	X	X													
BW16SR-008	BW16SR-008-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-008-0.23-0.48	0.23-0.48	G	X	X	X	X	X													
BW16SR-009	BW16SR-009-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-009-0.17-0.42	0.17-0.42	G	X	X	X	X	X													
BW16SR-010	BW16SR-010-0.0-0.24	0.0-0.24	G	X	X	X	X	X													
BW16SR-011	BW16SR-011-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-011-0.16-0.41	0.16-0.41	G	X	X	X	X	X													
	BW16SR-111-0.0-0.15	0.0-0.15	G	X	X			X													
BW16SR-012	BW16SR-012-0.0-0.25	0.0-0.25	G	X	X	X	X	X													
	BW16SR-112-0.0-0.25	0.0-0.25	G	X	X			X													
BW16SR-013	BW16SR-013-0.0-0.15	0.0-0.25	G	X	X	X	X	X													
	BW16SR-013-0.11-0.36	0.11-0.36	G	X	X	X	X	X													
BW16SR-014	BW16SR-014-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
BW16SR-015	BW16SR-015-0.0-0.15	0.0-0.15	G	X	X	X	X	X													
	BW16SR-115-0.0-0.15	0.0-0.15	G	X	X			X													
BW16SR-016	BW16SR-016-0.15-0.60	0.15-0.60	G	X	X	X		X			X				X						
Boulder Lake Reservoir (Reference Sample)																					
BW16BR-001	BW16BR- HD-001-MCRS	0.0-0.015	C											Macrobenthos		17	X	X	X		Composite (001, 002, 003, 004, & 005)
	BW16BLR-001-0.0-0.15	0.0-0.015	C	X	X	X	X				X	X			X						Chemistry Composite from BW16BR-002 through 005
BW16BR-002	BW16BR-002	0.0-0.015	C																		
BW16BR-003	BW16BR-003	0.0-0.015	C																		
BW16BR-004	BW16BR-004	0.0-0.015	C																		
BW16BR-005	BW16BR-005	0.0-0.015	C																		

Table 2 - Sample Locations
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Sample Location	Sample ID	Easting	Northing	Water Depth (ft)	Date Sampled
BW16SR-001	BW16SR-HD-001-MCRS	-92.416369	46.711401	7.4	9/21/2016
	BW16SR-001-0.0-0.15-M				
	BW16SR-001				
BW16SR-002	BW16SR-002-0.0-0.15-M	-92.41845	46.709546	NR	9/28/2016
	BW16SR-002				
	BW16SR-002-0.0-0.15-D				
	BW16SR-102-0.0-0.15-D				
BW16SR-003	BW16SR-003-0.0-0.15-M	-92.416662	46.710885	7.2	9/22/2016
	BW16SR-003				
	BW16SR-103-0.0-0.15-M				
	BW16SR-003-0.0-0.15-D				
BW16SR-004	BW16SR-004-0.0-0.15	-92.41708	46.710086	NR	9/28/2016
	BW16SR-004				
BW16SR-005	BW16SR-005-0.0-0.15-D	-92.416137	46.70985	NR	9/28/2016
	BW16SR-005-0.0-0.15-C				
	BW16SR-005				
BW16SR-006	BW16SR-006-0.0-0.15	-92.416828	46.709685	8.4	9/22/2016
	BW16SR-006-0.27-0.52				
BW16SR-007	BW16SR-007-0.0-0.15	-92.418351	46.709768	0.5	9/22/2016
	BW16SR-007-0.06-0.31				
BW16SR-008	BW16SR-008-0.0-0.15	-92.422142	46.71115	2	9/22/2016
	BW16SR-008-0.23-0.48				
BW16SR-009	BW16SR-009-0.0-0.15	-92.422008	46.711771	0.5	9/22/2016
	BW16SR-009-0.17-0.42				
BW16SR-010	BW16SR-010-0.0-0.24	-92.418603	46.713449	1.4	9/22/2016
BW16SR-011	BW16SR-011-0.0-0.15	-92.419219	46.712286	0.7	9/22/2016
	BW16SR-011-0.16-0.41				
	BW16SR-111-0.0-0.15				
BW16SR-012	BW16SR-012-0.0-0.25	-92.41759	46.712343	5.5	9/22/2016
	BW16SR-112-0.0-0.25				
BW16SR-013	BW16SR-013-0.0-0.15	-92.416611	46.712004	2.9	9/22/2016
	BW16SR-013-0.11-0.36				
BW16SR-014	BW16SR-014-0.0-0.15	-92.418578	46.708766	NR	10/7/2016
BW16SR-015	BW16SR-015-0.0-0.15	-92.420848	46.711918	NR	10/7/2016
	BW16SR-115-0.0-0.15				
BW16SR-016	BW16SR-016-0.15-0.60	-92.416118	46.709931	NR	10/7/2016
Boulder Lake Reservoir (Reference Sample)					
BW16BR-HD	BW16BR-HD-001-MCRS	-92.208112	47.056288	8.0	9/20/2016
	BW16BLR-001-0.0-0.15				
BW16BR-002	BW16BR-002	-92.183069	47.076127	14.2	9/20/2016
BW16BR-003	BW16BR-003	-92.201496	47.070839	7.5	9/21/2016
BW16BR-004	BW16BR-004	NR	NR	NR	NR
BW16BR-005	BW16BR-005	NR	NR	NR	NR

NR- Not recorded

Table 3 - Core Summary
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Sample Location	Sample ID	Date Sampled	Sample Method	Depth of Push (m)	Depth of Push (ft)	Recovery (m)	Recovery (ft)	Percent Recovery
BW16SR-001	BW16SR-001-0.0-0.15	9/21/2016	Ponar	0.15	0.5	0.15	0.5	100
BW16SR-002	BW16SR-002-0.0-0.15	9/28/2016	Ponar	0.15	0.5	0.15	0.5	100
BW16SR-003	BW16SR-003-0.0-0.15	9/22/2016	Ponar	0.15	0.5	0.15	0.5	100
BW16SR-004	BW16SR-004-0.0-0.15	9/28/2016	Ponar	0.15	0.5	0.15	0.5	100
BW16SR-005	BW16SR-005-0.0-0.15	9/28/2016	Ponar	0.15	0.5	0.15	0.5	100
BW16SR-006	BW16SR-006-0.0-0.15	9/22/2016	Check Valve	0.98	3.2	0.52	1.7	53
	BW16SR-006-0.27-0.52							
BW16SR-007	BW16SR-007-0.0-0.15	9/22/2016	Check Valve	0.40	1.3	0.34	1.1	85
	BW16SR-007-0.06-0.31							
BW16SR-008	BW16SR-008-0.0-0.15	9/22/2016	Check Valve	0.61	2.0	0.50	1.65	83
	BW16SR-008-0.23-0.48							
BW16SR-009	BW16SR-009-0.0-0.15	9/22/2016	Check Valve	0.55	1.8	0.43	1.4	78
	BW16SR-009-0.17-0.42							
BW16SR-010	BW16SR-010-0.0-0.24	9/22/2016	Check Valve	0.37	1.2	0.27	0.9	75
BW16SR-011	BW16SR-011-0.0-0.15	9/22/2016	Check Valve	0.49	1.6	0.43	1.4	88
	BW16SR-011-0.16-0.41							
	BW16SR-111-0.0-0.15							
BW16SR-012	BW16SR-012-0.0-0.25	9/22/2016	Check Valve	1.37	4.5	0.40	1.3	29
	BW16SR-112-0.0-0.25							
BW16SR-013	BW16SR-013-0.0-0.15	9/22/2016	Check Valve	0.55	1.8	0.37	1.2	67
	BW16SR-013-0.11-0.36							
Boulder Lake Reservoir (Reference Sample)								
BW16BR-001	BW16BLR-001-0.0-0.15	9/20/2016	Ponar Grab	0.15	0.5	0.15	0.5	100
BW16BR-002	BW16BR-002	9/20/2016	Ponar Grab	0.15	0.5	0.15	0.5	100
BW16BR-003	BW16BR-003	9/21/2016	Ponar Grab	0.15	0.5	0.15	0.5	100

Table 4 - Poling Locations
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Location ID	Date Sampled	Poling Location		Polling ID Location	Depth of Water (cm)	Depth of Water (ft)	Depth to Resistance (cm)	Depth to Refusal (cm)	Depth to Refusal (ft)	Soft Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal	Sediment Surface Elevation (ft AMSL)	Refusal Elevation (ft AMSL)
		Longitude	Latitude											
PL-01	6/21/16	-92.421276	46.713414	NA	20	0.7	80	200	6.6	180	Sediment	Sandy silt	1119.1	1113.2
PL-02	6/21/16	-92.420893	46.71454	NA	270	8.9	340	340	11.2	70	Sediment	Sandy silt	1110.9	1108.6
PL-03	6/21/16	-92.41943	46.713882	NA	210	6.9	250	250	8.2	40	Sediment	Gravel/silt/sand/cobble	1112.9	1111.6
PL-04	6/21/16	-92.419554	46.713513	NA	150	4.9	160	160	5.2	10	Rock	Gravel/silt/sand/cobble	1114.9	1114.6
PL-04	6/21/16			NA	150	4.9	180	180	5.9	40	Rock	Gravel/silt/sand/cobble	1114.9	1113.9
PL-05	6/21/16	-92.419211	46.712289	NA	30	1.0	60	60	2.0	30	Sediment	Silt	1118.8	1117.8
PL-05	6/21/16			NA	50	1.6	60	150	4.9	110	Sediment	Silty clay	1118.2	1114.9
PL-06	6/21/16	-92.417789	46.712387	NA	180	5.9	280	310	10.2	130	Sediment	Silty clay	1113.9	1109.6
PL-07	6/21/16	-92.41843	46.713406	NA	50	1.6	90	120	3.9	70	Sediment	Silty clay w/ sand	1118.2	1115.9
PL-08	6/21/16	-92.417815	46.713236	NA	40	1.3	90	90	3.0	50	Sediment	Silty clay	1118.5	1116.8
PL-09	6/21/16	-92.416338	46.711502	NA	170	5.6	210	220	7.2	50	Rock	Gravel	1114.2	1112.6
PL-09	6/21/16			NA	180	5.9	210	210	6.9	20	Rock	Gravel	1113.9	1112.9
PL-10	6/21/16	-92.417091	46.71163	NA	190	6.2	270	300	9.8	110	Sediment	Silty clay w/ sand/org.	1113.6	1110.0
PL-11	6/21/16	-92.42002	46.711263	NA	40	1.3	130	220	7.2	180	Sediment	Silty clay w/ sand/org.	1118.5	1112.6
PL-12	6/21/16	-92.420234	46.711222	NA	30	1.0	100	110	3.6	80	Sediment	Silty clay w/ sand/org.	1118.8	1116.2
PL-13	6/21/16	-92.420368	46.711189	NA	20	0.7	130	140	4.6	120	Sediment	Silty fine sand	1119.1	1115.2
PL-14	6/21/16	-92.420538	46.711147	NA	330	10.8	410	450	14.8	120	Sediment	Clayey silt	1109.0	1105.0
PL-15	6/21/16	-92.420757	46.710841	NA	480	15.7	560	560	18.4	80	Sediment	Granular	1104.1	1101.4
PL-16	6/21/16	-92.422319	46.710596	NA	20	0.7	50	50	1.6	30	Sediment	Clayey silt	1119.1	1118.2
PL-17	6/21/16	-92.422136	46.710913	NA	20	0.7	30	120	3.9	90	Sediment	Clayey silt	1119.1	1115.9
PL-18	6/21/16	-92.42244	46.711966	NA	110	3.6	120	140	4.6	30	Rock	Clayey silt w/ sand	1116.2	1115.2
PL-19	6/21/16	-92.422037	46.711792	NA	20	0.7	20	20	0.7	0	Sediment	Sandbar	1119.1	1119.1
PL-20	6/21/16	-92.418529	46.709781	NA	40	1.3	60	70	2.3	30	Sediment	Silty sand	1118.5	1117.5
PL-21	6/21/16	-92.417697	46.709748	NA	40	1.3	60	90	3.0	50	Sediment	Sandy silt	1118.5	1116.8
PL-22	6/21/16	-92.416721	46.71	NA	210	6.9	280	280	9.2	70	Sediment	Clayey silt	1112.9	1110.6
PL-23	6/21/16	-92.416678	46.710748	NA	250	8.2	270	280	9.2	30	Sediment	Clayey silt w/ sand	1111.6	1110.6
PL-24	6/21/16	-92.416184	46.710726	NA	100	3.3	130	170	5.6	70	Sediment	Clayey silt	1116.5	1114.2
PL-25	6/21/16	-92.416295	46.710068	NA	230	7.5	280	280	9.2	50	Sediment	Clayey silt	1112.3	1110.6
BW16SR-006	9/22/16	-92.416828	46.709685	PL-01	256	8.4	416	416	13.6	160	Sediment	NA	1111.4	1106.2
BW16SR-012	9/22/16	-92.41759	46.712343	PL-01	170	5.6	170	312	10.2	142	Sediment	Peat	1114.2	1109.6
BW16SR-013	9/22/16	-92.416611	46.712004	PL-01	88	2.9	129	129	4.2	41	Sediment	Peat	1116.9	1115.6
Boulder Lake Reservoir (Reference Sample)														
BW16BR-001	9/20/16	-92.208112	47.056288	PL-01	254	NC	289	315	NC	61	Woody Debris	Silt Loam	NC	NC
BW16BR-002	9/20/16	-92.183069	47.076127	PL-01	432	NC	549	605	NC	173	Sediment	Silt	NC	NC
BW16BR-003	9/21/16	-92.201496	47.070839	PL-01	239	NC	249	272	NC	33	Sediment	Silt Loam	NC	NC

Note:
Water elevation maintained at 1119.80 ft +/- 0.50 ft
NC- Not Calculated

Table 5 - Analytical Parameters Summary
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Analytical Parameters	Chemical Abstract Number or Analyte Code	Analytical Method
Total Metals		
Mercury	7439-97-6	SW-846 7471B
Polychlorinated Dibenzo-p-dioxins (Dioxins)/Polychlorinated Dibenzofurans (Furans)		
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6	SW-846 8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4	SW-846 8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7	SW-846 8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6	SW-846 8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3	SW-846 8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9	SW-846 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9	SW-846 8290A
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9	SW-846 8290A
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6	SW-846 8290A
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4	SW-846 8290A
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9	SW-846 8290A
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9	SW-846 8290A
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9	SW-846 8290A
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5	SW-846 8290A
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4	SW-846 8290A
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7	SW-846 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0	SW-846 8290A
TCDD Equivalent	--	
General Chemistry		
Total Organic Carbon	--	SW-846 9060A
Physical Testing		
Grain Size	--	ASTM D422

Table 6 - Total Organic Carbon Results
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Sample Name	Sample Depth Start (m)	Sample Depth End (m)	Result (mg/kg)	Results Qualifier
BW16SR-001-0.0-0.15	0	0.15	39500	
BW16SR-002-0.0-0.15	0	0.15	47100	
BW16SR-003-0.0-0.15	0	0.15	31400	
BW16SR-004-0.0-0.15	0	0.15	39500	
BW16SR-005-0.0-0.15	0	0.15	33900	
BW16SR-006-0.0-0.15	0	0.15	27000	
BW16SR-006-0.27-0.52	0.27	0.52	7090	
BW16SR-007-0.0-0.15	0	0.15	19700	
BW16SR-007-0.06-0.31	0.06	0.31	33300	
BW16SR-008-0.0-0.15	0	0.15	25700	
BW16SR-008-0.23-0.48	0.23	0.48	48300	
BW16SR-009-0.0-0.15	0	0.15	11500	
BW16SR-009-0.17-0.42	0.17	0.42	20200	
BW16SR-010-0.0-0.24	0	0.24	51700	
BW16SR-011-0.0-0.15	0	0.15	22900	
BW16SR-011-0.16-0.41	0.16	0.41	30900	
BW16SR-012-0.0-0.25	0	0.25	60100	
BW16SR-013-0.0-0.15	0	0.15	54500	
BW16SR-013-0.11-0.36	0.11	0.36	18200	
BW16SR-014-0.0-0.15	0	0.15	14300	
BW16SR-015-0.0-0.15	0	0.15	13900	
BW16SR-016-0.15-0.60	0.15	0.6	36500	

Notes:
TOC - Total organic carbon
m - meters
mg/kg - milligrams per kilogram
TOC analyzed by EPA Method SW9060

Table 7 - Community Assessment
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Location	Collection Information				Benthic Macroinvertebrates												
	Date	Number of Ponar Grabs	Approximate Collection Area (cm)[1]	Community Assessment Duration (min)	Alderfly (Sensitive)	Mayfly (Semi-Sensitive)	Fingernail Clam (Semi-Sensitive)	Non-Red Midge (Semi-Tolerant)	Horsefly (Tolerant)	Horsehair Worm (Tolerant)	Thread Worm (Tolerant)	Snails (Semi-Tolerant)	Bloodworm (Tolerant)	Tubifex Worm (Tolerant)	Needleworm (Tolerant)	Biotic Index Score[2]	Biotic Health Score[3]
BW16SR-001	9/21/2016	3	675	15	0	4	1	0	0	1	6	2	3	0	0	**1.6**	**Poor**
				Weighted Group Score	0	12	3	0	0	1	6	2	3	0	0		
BW16SR-002	9/28/2016	3	675	15	0	1	0	0	0	1	0	4	2	0	0	**1.3**	**Poor**
				Weighted Group Score	0	3	0	0	0	1	0	4	2	0	0		
BW16SR-003	9/22/2016	3	675	15	2	5	0	0	0	0	8	4	3	0	0	**1.7**	**Poor**
				Weighted Group Score	8	15	0	0	0	0	8	4	3	0	0		
BW16SR-004	9/28/2016	3	675	15	0	0	0	0	0	1	3	0	3	0	0	**1.0**	**Poor**
				Weighted Group Score	0	0	0	0	0	1	3	0	3	0	0		
BW16SR-005	9/28/2016	3	675	15	0	0	0	0	0	0	4	3	5	0	0	**1.0**	**Poor**
				Weighted Group Score	0	0	0	0	0	0	4	3	5	0	0		
Boulder Lake Reservoir (Reference Sample)																	
BW16BLR-001	9/20/2016	3	675	15	0	0	0	0	0	0	0	0	0	0	0	**0.0**	**Poor**
				Weighted Group Score	0	0	0	0	0	0	0	0	0	0	0		

[1]Each grab = 15.2 cm x 15.2 cm (225 cm2)

[2]Biotic Index Score Calculation: http://watermonitoring.uwex.edu/pdf/level1/datasheets/data-Biotic2014.pdf

[3]Biotic Health Score:

Good	2.6-3.5
Fair	2.1-2.0
Poor	1.0-2.0

Table 8 - Metals Results
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Chemical					BW16SR-001-0.0-0.15		BW16SR-002-0.0-0.15		BW16SR-003-0.0-0.15		BW16SR-004-0.0-0.15		BW16SR-005-0.0-0.15		BW16SR-006-0.0-0.15		BW16SR-006-0.27-0.52		BW16SR-007-0.0-0.15	
	Sample Interval (meters)				0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.15-0.50		0.0-0.15	
	SQT Level 1	SQT Midpoint	SQT Level 2	Result unit	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Mercury	0.18	0.64	1.1	mg/kg	0.16		0.042		0.10		0.17		0.11		0.086		0.019	J	0.052	

Chemical	BW16SR-007-0.06-0.31		BW16SR-008-0.0-0.15		BW16SR-008-0.23-0.48		BW16SR-009-0.0-0.15		BW16SR-009-0.17-0.42		BW16SR-010-0.0-0.24		BW16SR-011-0.0-0.15		BW16SR-011-0.16-0.41		BW16SR-012-0.0-0.25	
Sample Interval (meters)	0.0-0.15 and 0.15-0.5		0.0-0.15		0.15-0.50		0.0-0.15		0.15-0.50		0.0-0.15 and 0.15-0.5		0.0-0.15		0.15-0.50		0.0-0.15 and 0.15-0.5	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Mercury	0.11		0.072		0.54		0.033		0.072		0.099		0.082		0.12		0.35	J

Chemical	BW16SR-013-0.0-0.15		BW16SR-013-0.11-0.36		BW16SR-014-0.0-0.15		BW16SR-015-0.0-0.15		BW16SR-016-0.15-0.60		BW16SR-111-0.0-0.15		BW16SR-112-0.0-0.25		BW16SR-115-0.0-0.15		BW16BLR-001-0.0-0.15	
Sample Interval (meters)	0.0-0.15		0.15-0.50		0.0-0.15		0.0-0.15		0.15-0.50		0.0-0.15		0.0-0.15 and 0.15-0.5		0.0-0.15		0.0-0.15	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Mercury	0.16		0.034		0.045		0.047		0.28		0.069		0.68	J	0.092		0.19	

Notes:
Q - Qualifiers
J - estimated value
mg/kg - milligrams per kilogram
NE - not established
SQT - Sediment Quality Target
U - concentration did not exceed laboratory reporting limit
Values highlighted in yellow indicate concentration exceeding SQT Level I
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II
Values highlighted in red indicate concentration exceeding SQT Level II
Mercury was analyzed by EPA Method SW7471B

Table 9 - Dioxin/Furan Results (Sediment)
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Chemical	Sample Name				BW16SR-001-0.0-0.15		BW16SR-002-0.0-0.15		BW16SR-003-0.0-0.15		BW16SR-004-0.0-0.15		BW16SR-005-0.0-0.15		BW16SR-006-0.0-0.15		BW16SR-006-0.27-0.52		BW16SR-007-0.0-0.15		BW16SR-007-0.06-0.31	
	Sample Interval (meters)				0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.0-0.15		0.15-0.50		0.0-0.15		0.0-0.15 and 0.15-0.5	
	SQT Level I	SQT Midpoint	SQT Level II	Units	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
1,2,3,4,6,7,8-HpCDD	NE	NE	NE	ng/Kg	440		1500	J	360		990		460		110		1	J	120		37000	J
1,2,3,4,6,7,8-HpCDF	NE	NE	NE	ng/Kg	1400		230	J	1100		870		1200		320		0.56	J	130		12000	J
1,2,3,4,7,8,9-HpCDF	NE	NE	NE	ng/Kg	9.1		2.9	J	9.2		15		8.9		1.9	J	0.87	J	1.4	J	630	
1,2,3,4,7,8-HxCDD	NE	NE	NE	ng/Kg	6.7		22		4.4	J	7.4		4.4	J	1.4		0.42		0.51		3.2	
1,2,3,4,7,8-HxCDF	NE	NE	NE	ng/Kg	15		5.1	J	12		15	J	15		3.7	J	0.15		1.9	J	150	
1,2,3,6,7,8-HxCDD	NE	NE	NE	ng/Kg	45		86		34		55		33		7.4		0.41		4	J	1800	
1,2,3,6,7,8-HxCDF	NE	NE	NE	ng/Kg	27		7.4		22		19		24		5.9	J	0.28		3	J	40	
1,2,3,7,8,9-HxCDD	NE	NE	NE	ng/Kg	28		59		18		16		18		3.5	J	0.4		1.6	J	69	
1,2,3,7,8,9-HxCDF	NE	NE	NE	ng/Kg	3.9	J	2.2	J	3.3	J	3.8	J	3.4	J	1.2	J	0.29		0.64	J	71	
1,2,3,7,8-PeCDD	NE	NE	NE	ng/Kg	10		6.5	J	5.9	J	4.2	J	5.9	J	2.4	J	0.22		0.39	J	5.4	J
1,2,3,7,8-PeCDF	NE	NE	NE	ng/Kg	2.3	J	0.89	J	1.7	J	1.2	J	1.7	J	0.73	J	0.16		0.27	J	2.4	J
2,3,4,6,7,8-HxCDF	NE	NE	NE	ng/Kg	11		3.8	J	7.5		7.9		7.9		2.7	J	0.35		1.1	J	94	
2,3,4,7,8-PeCDF	NE	NE	NE	ng/Kg	4.8	J	2.1	J	3.8	J	3.6	J	3.7	J	0.82	J	0.12		0.52	J	22	
2,3,7,8-TCDD	NE	NE	NE	ng/Kg	4		0.71		2.4		3.5		2		0.56		0.23		0.33	J	2.3	
2,3,7,8-TCDF	NE	NE	NE	ng/Kg	7.6		0.81	J	4.2		15		4.1		1.9		0.34	J	1.1	J	8.4	
OCDD	NE	NE	NE	ng/Kg	4100		3600	J	2900		11000	J	5200		1300		5.8	J	1000		160000	J
OCDF	NE	NE	NE	ng/Kg	550		87	J	470		860		560		160		0.9		80		63000	J
Total HpCDD	NE	NE	NE	ng/Kg	930		2700		700		2000		1000		250		2.1	J	250		58000	J
Total HpCDF	NE	NE	NE	ng/Kg	2500		600		2100		1900		2300		620		0.55		260		13000	J
Total HxCDD	NE	NE	NE	ng/Kg	370		860		260		350		270		70		3.7	J	33		4600	
Total HxCDF	NE	NE	NE	ng/Kg	710		290		540		560		780		180		0.27		82		18000	J
Total PeCDD	NE	NE	NE	ng/Kg	87		110		55		51		60		15		0.38	J	3	J	97	
Total PeCDF	NE	NE	NE	ng/Kg	100		19		64		58		56		12		0.14		3.4	J	160	
Total TCDD	NE	NE	NE	ng/Kg	33		14		19		22		19		6.6		1.4		2.4		38	
Total TCDF	NE	NE	NE	ng/Kg	35		4.1		17		43		23		7		3.1		3.4		55	
TEQ KM Fish[1]	0.85	11.2	21.5	ng TEQ/Kg	52.098		44.4641		36.148		45.034		37.569		9.7432	J	0.2712	J	4.0996	J	292.872	J

Notes:
Dioxins analyzed by EPA Method SW8290
Q - Qualifier
J - estimated value
NE - not established
ng TEQ/kg - nanograms of dioxin toxicity equivalency per kilogram
ng/kg - nanograms per kilogram
SQT - Sediment Quality Target
TEQ - dioxins/furans toxicity equivalency
U - concentration did not exceed laboratory reporting limit
Values highlighted in yellow indicate concentration exceeding SQT Level I
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II
Values highlighted in red indicate concentration exceeding SQT Level II
[1]The United States Environmental Protection Agency TEQ Kaplan Meier calculator was used to calculate TEQ FISH values, which utilized 1998 World Health Organization toxicity equivalency factors for fish.

Table 9 - Dioxin/Furan Results (Sediment)
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Chemical	Sample Name				BW16SR-008-0.0-0.15		BW16SR-008-0.23-0.48		BW16SR-009-0.0-0.15		BW16SR-009-0.17-0.42		BW16SR-010-0.0-0.24		BW16SR-011-0.0-0.15		BW16SR-011-0.16-0.41		BW16SR-012-0.0-0.25		BW16SR-013-0.0-0.15	
	Sample Interval (meters)				0.0-0.15		0.15-0.50		0.0-0.15		0.15-0.50		0.0-0.15 and 0.15-0.5		0.0-0.15		0.15-0.50		0.0-0.15 and 0.15-0.5		0.0-0.15	
	SQT Level I	SQT Midpoint	SQT Level II	Units	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
1,2,3,4,6,7,8-HpCDD	NE	NE	NE	ng/Kg	120		930		23		130		310		110		680		990	J	560	
1,2,3,4,6,7,8-HpCDF	NE	NE	NE	ng/Kg	120		3700		29		85		1700		320		390		790		1800	
1,2,3,4,7,8,9-HpCDF	NE	NE	NE	ng/Kg	1.8	J	33		0.61		2	J	12		2.2	J	8.7	J	16	J	13	
1,2,3,4,7,8-HxCDD	NE	NE	NE	ng/Kg	0.99		12		0.38		1.3	J	9.5		0.89	J	4.1	J	8.1	J	9.4	J
1,2,3,4,7,8-HxCDF	NE	NE	NE	ng/Kg	2.5	J	39		0.5	J	1.6	J	14		4.5	J	6.5		13	J	17	
1,2,3,6,7,8-HxCDD	NE	NE	NE	ng/Kg	5.6	J	63		0.92	J	7.1		44		6.5	J	31		60	J	59	
1,2,3,6,7,8-HxCDF	NE	NE	NE	ng/Kg	4.8	J	37		0.76	J	3.4	J	18		7.1	J	14		20		23	
1,2,3,7,8,9-HxCDD	NE	NE	NE	ng/Kg	2.3	J	40		0.59	J	3.1	J	29		2.5	J	14		27		33	
1,2,3,7,8,9-HxCDF	NE	NE	NE	ng/Kg	0.97	J	14		0.31		1.2	J	5.4	J	1	J	2.5	J	4.7	J	5.5	J
1,2,3,7,8-PeCDD	NE	NE	NE	ng/Kg	1.5	J	15		0.37	J	0.81	J	9.8		1.7	J	5.3	J	9.5	J	22	
1,2,3,7,8-PeCDF	NE	NE	NE	ng/Kg	0.77	J	4.6	J	0.17		0.59	J	1.9	J	0.78	J	1.6	J	3	J	2.7	J
2,3,4,6,7,8-HxCDF	NE	NE	NE	ng/Kg	1.5	J	17		0.58	J	1.7	J	8.4		2.4	J	6.1		11	J	13	
2,3,4,7,8-PeCDF	NE	NE	NE	ng/Kg	0.94	J	9.4		0.29	U	0.8	J	4.8	J	1.4	J	3.3	J	5.6	J	7	J
2,3,7,8-TCDD	NE	NE	NE	ng/Kg	0.77	J	4.8		0.25		0.58	J	2.2		0.67	J	2.5		6.5	J	5.6	
2,3,7,8-TCDF	NE	NE	NE	ng/Kg	2.4		6.1		0.41	J	2.1		3.2		2.1		7.6		23	J	10	
OCDD	NE	NE	NE	ng/Kg	1600		11000	J	240		1900		2300		1400		11000	J	12000	J	4200	
OCDF	NE	NE	NE	ng/Kg	76		1900		14		76		690		160		420		1100		730	
Total HpCDD	NE	NE	NE	ng/Kg	280		2000		53		300		610		250		1600		2100	J	1200	
Total HpCDF	NE	NE	NE	ng/Kg	270		7000		58		210		3100		610		990		2000	J	3400	
Total HxCDD	NE	NE	NE	ng/Kg	56		610		10		68		410		62		300		500	J	590	
Total HxCDF	NE	NE	NE	ng/Kg	89		1700		20		78		760		190		350		570		1100	
Total PeCDD	NE	NE	NE	ng/Kg	13		130		3.7	J	13		95		16		46		97	J	150	
Total PeCDF	NE	NE	NE	ng/Kg	11		90		1.5	J	8.7		60		16		52		89	J	130	
Total TCDD	NE	NE	NE	ng/Kg	6.5		45		1.9		5.7		24		7.5		24		48		46	
Total TCDF	NE	NE	NE	ng/Kg	9.6		28		1.8		9.2		21		10		34		86	J	51	
TEQ KM Fish[1]	0.85	11.2	21.5	ng TEQ/Kg	6.5947	J	96.035		1.2777	J	5.5461	J	51.999		9.5259	J	25.98		49.511	J	73.952	

Notes:
Dioxins analyzed by EPA Method SW8290
Q - Qualifier
J - estimated value
NE - not established
ng TEQ/kg - nanograms of dioxin toxicity equivalency per kilogram
ng/kg - nanograms per kilogram
SQT - Sediment Quality Target
TEQ - dioxins/furans toxicity equivalency
U - concentration did not exceed laboratory reporting limit
Values highlighted in yellow indicate concentration exceeding SQT Level I
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II
Values highlighted in red indicate concentration exceeding SQT Level II
[1]The United States Environmental Protection Agency TEQ Kaplan Meier calculator was used to

Table 9 - Dioxin/Furan Results (Sediment)
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Chemical	Sample Name				BW16SR-013-0.11-0.36		BW16SR-014-0.0-0.15		BW16SR-015-0.0-0.15		BW16SR-016-0.15-0.60		BW16BLR-001-0.0-0.15	
	Sample Interval (meters)				0.15-0.50		0.0-0.15		0.0-0.15		0.15-0.50		0.0-0.15	
	SQT Level I	SQT Midpoint	SQT Level II	Units	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
1,2,3,4,6,7,8-HpCDD	NE	NE	NE	ng/Kg	190		14		15		850		14	
1,2,3,4,6,7,8-HpCDF	NE	NE	NE	ng/Kg	1300		41		89		4300	J	3.50	J
1,2,3,4,7,8,9-HpCDF	NE	NE	NE	ng/Kg	8.9		0.38	J	0.44	J	34		0.64	
1,2,3,4,7,8-HxCDD	NE	NE	NE	ng/Kg	5	J	0.23	J	0.12		17		0.46	J
1,2,3,4,7,8-HxCDF	NE	NE	NE	ng/Kg	15		0.44	J	0.97		72		0.69	J
1,2,3,6,7,8-HxCDD	NE	NE	NE	ng/Kg	35		0.89	J	0.94	J	100		1.00	J
1,2,3,6,7,8-HxCDF	NE	NE	NE	ng/Kg	17		0.97	J	1.8	J	110		0.83	J
1,2,3,7,8,9-HxCDD	NE	NE	NE	ng/Kg	20		0.44	J	0.35	J	67		1.10	J
1,2,3,7,8,9-HxCDF	NE	NE	NE	ng/Kg	4	J	0.032		0.063		11		0.70	
1,2,3,7,8-PeCDD	NE	NE	NE	ng/Kg	6.6		0.19	J	0.2	J	23		.47	J
1,2,3,7,8-PeCDF	NE	NE	NE	ng/Kg	1.4	J	0.12	J	0.23	J	7.2	J	.97	J
2,3,4,6,7,8-HxCDF	NE	NE	NE	ng/Kg	9.5		0.38	J	0.47	J	19		0.68	J
2,3,4,7,8-PeCDF	NE	NE	NE	ng/Kg	5.9		0.26	J	0.28	J	17		0.97	J
2,3,7,8-TCDD	NE	NE	NE	ng/Kg	1.4		0.23		0.086		6.1		.47	
2,3,7,8-TCDF	NE	NE	NE	ng/Kg	1.3		0.5	J	0.37	J	12		1.70	J
OCDD	NE	NE	NE	ng/Kg	1100		160		180		6700		89.00	
OCDF	NE	NE	NE	ng/Kg	510		19		34		2000		5.40	J
Total HpCDD	NE	NE	NE	ng/Kg	390		31		37		1700		28	
Total HpCDF	NE	NE	NE	ng/Kg	2400		76		160		8300	J	5.50	J
Total HxCDD	NE	NE	NE	ng/Kg	280		5.2	J	9.1		900		12.0	J
Total HxCDF	NE	NE	NE	ng/Kg	700		20		39		2500		6.60	J
Total PeCDD	NE	NE	NE	ng/Kg	58		3.2	J	2.2	J	190		1.80	J
Total PeCDF	NE	NE	NE	ng/Kg	83		2.7	J	4.6	J	240		9	J
Total TCDD	NE	NE	NE	ng/Kg	11		2.1		1.4		53		0.82	J
Total TCDF	NE	NE	NE	ng/Kg	23		2		2.3		68		14	
TEQ KM Fish[1]	0.85	11.2	21.5	ng TEQ/Kg	39.475		1.2623	J	1.7427	J	139.49		3.13	

Notes:
Dioxins analyzed by EPA Method SW8290
Q - Qualifier
J - estimated value
NE - not established
ng TEQ/kg - nanograms of dioxin toxicity equivalency per kilogram
ng/kg - nanograms per kilogram
SQT - Sediment Quality Target
TEQ - dioxins/furans toxicity equivalency
U - concentration did not exceed laboratory reporting limit
Values highlighted in yellow indicate concentration exceeding SQT Level I
Values highlighted in orange indicate concentration exceeding the midpoint between SQT Level I and SQT Level II
Values highlighted in red indicate concentration exceeding SQT Level II
[1]The United States Environmental Protection Agency TEQ Kaplan Meier calculator was used to

Table 10 - Benthic Macroinvertebrate Tissue Results - Macrobenthos

Scanlon Reservoir

St. Louis River Area of Concern

Scanlon, Minnesota

Sample Information				Analytical Results					
Reservoir	Sample ID	Benthic Macroinvertebrate	Weight (g)	Total Mercury (mg/kg)	Results Qualifier	Methyl Mercury (µg/kg)	% Lipids (%)	TEQ FISH (ng TEQ/kg)	
Scanlon	BW16SR-001M	Mayfly	39.36	0.034	U	3.1	0.72	1.48	J
Scanlon	BW16SR-002M	Mayfly	40.43	0.031	U	3.3	0.56	0.33	J
Scanlon	BW16SR-003M	Mayfly	51.67	0.036	U	4.5	0.66	1.14	J
Scanlon	BW16SR-002D	Dragonfly	51.72	0.029	U	23	0.68	0.34	J
Scanlon	BW16SR-003D	Dragonfly	17.3	NA		NA	0.69	0.55	J
Scanlon	BW16SR-005D	Dragonfly	48.4	0.030	U	25	0.78	0.28	J
Scanlon	BW16SR-005C	Crawfish	37	0.030	U	18	0.39	0.43	J
Scanlon	BW16SR-HD-001-MRCS	Macro*	4.5	0.037	U	4.5	NA	NA	
Boulder Lake Reservoir (Reference Sample)									
Boulder	EPA16BR-HD-001-MRCS	Macro	23.7	0.032	U	4.3	0.72	0.11	J

Notes:

*combine EPA and BW samples into one sample

U - Not Detected

g - gram

mg/kg - milligram per kilogram

µg/kg - microgram per kilogram

ng TEQ/kg - nanogram per kilogram

NA- Not Applicable

Table 11 - Benthic Macroinvertebrate Tissue Results - Lumbriculus

Scanlon Reservoir

St. Louis River Area of Concern

Scanlon, Minnesota

Sample Information			Analytical Results					
Reservoir	Sample ID	Benthic Macroinvertebrate	Total Mercury (mg/kg)	Results Qualifier	Methyl Mercury (µg/kg)	% Lipids (%)	TEQ FISH[1] (ng TEQ/kg)	
Scanlon	BW16SR-004	Lumbriculus	0.036	U	0.24	0.71	0.98	J
Scanlon	BW16SR-016	Lumbriculus	0.037	U	0.32	0.74	3.98	J
Boulder Lake Reservoir (Reference Sample)								
Boulder	BW16BLR-001[1]	Lumbriculus	0.038	U	0.15	0.63	0.09	
Background Sample								
Background	Background Day 0	Lumbriculus	0.038	U	0.088	1.2	0.06	J

Notes:

*combine EPA and BW samples into one sample

U - Not Detected

g - gram

mg/kg - milligram per kilogram

µg/kg - microgram per kilogram

ng TEQ/kg - nanogram per kilogram

NA- Not Applicable

TEQ - dioxins/furans toxicity equivalent

[1] *The United States Environmental Protection Agency TEQ Kaplan Meier calculator was used to calculate TEQ FISH values, which utilized 1998 World Health Organization toxi*

Table 12 - Fish Tissue Results
Scanlon Reservoir
St. Louis River Area of Concern
Scanlon, Minnesota

Sample Information												Analytical Results					
Reservoir	Sample ID	Fish	Fish Trophic Level*	Date Collected	No of Fish Collected	Total Weight (g)	Duplicate	Date Received by GLEC	Sex	Otolith	No of Fish Aged	Total Mercury (mg/kg)	Results Qualifier	Methyl Mercury (µg/kg)	% Lipids	TEQ Fish[1] (ng TEQ/kg)	
Scanlon	MN16+SR-NP-A	Northern Pike	4.1	10/6/2016	3	2383		10/27/2016	Yes	Yes	NR	0.12	J	100	0.14	0.34	J
Scanlon	MN16+SR-GSH-A	Shiner Mix	2.1	10/6/2016	2	827		10/27/2016	Yes	Yes	2	0.054	J	41	0.55	0.10	J
Scanlon	MN16+SR-WAL-A	Walleye	4.5	10/6/2016	NR	61		10/27/2016	No	No	NA	0.12	J	110	0.37	0.32	J
Scanlon	MN16+SR-SMB-A	Smallmouth Bass	3.6	10/6/2016	3	1721		10/27/2016	Yes	Yes	3	0.22		230	1.3	0.93	J
Scanlon	MN16+SR-SMB-B	Smallmouth Bass	3.6	10/6/2016	3	1338		10/27/2016	Yes	Yes	3	0.2		170	1.1	0.86	J
Scanlon	MN16+SR-SMB-C	Smallmouth Bass	3.6	10/6/2016	3	858		10/27/2016	Yes	Yes	3	0.071	J	110	1.1	0.63	J
Scanlon	MN16+SR-WS-C	White Sucker	2.8	10/6/2016	3	620		10/27/2016	Yes	Yes	3	0.083	J	95	1.8	0.94	J
Scanlon	MN16+SR-WS-A	White Sucker	2.8	10/6/2016	3	2543		10/27/2016	Yes	Yes	3	0.075	J	96	3.1	1.25	J
Scanlon	MN16+SR-WS-B	White Sucker	2.8	10/6/2016	3	2120		10/27/2016	Yes	Yes	3	0.067	J	58	1.3	0.53	J
Scanlon	MN16+SR+YP-A	Yellow Perch	3.7	10/6/2016	4	500	MS/MSD	10/27/2016	Yes	Yes	4	0.086	J	89	0.72	0.39	J
Scanlon	MN16+SR+YP-B	Yellow Perch	3.7	10/6/2016	3	315		10/27/2016	Yes	Yes	3	0.079	J	98	0.56	0.67	J
Scanlon	MN16+SR+YP-C	Yellow Perch	3.7	10/6/2016	1	432		10/27/2016	Yes	Yes	1	0.092	J	74	0.4	0.58	J
Boulder Lake Reservoir (Reference Sample)																	
Boulder	MN16+BR+RB-A[12]	Rock Bass	3.4	10/6/2016	9	208		10/27/2016	No	No	NA	0.077	J B	76	1.2	0.06	
Boulder	MN16+BR+BLC-A[12]	Black Clappie	3.8	10/6/2016	6	116		10/27/2016	No	No	NA	0.068	J B	53	1	0.05	
Boulder	MN16+BR+YP-A	Yellow Perch	3.7	10/6/2016	13	378		10/27/2016	No	No	NA	0.073	J B	56	0.52	0.05	J
Boulder	MN16+BR+YP-B[12]	Yellow Perch	3.7	10/6/2016	13	311		10/27/2016	No	No	NA	0.068	J B	54	0.27	0.07	
Boulder	MN16+BR+YP-C[12]	Yellow Perch	3.7	10/6/2016	NR	152		10/27/2016	No	No	NA	0.077	J B	65	1.8	0.05	
Boulder	MN16+BR+GSH-A[12]	Shiner Mix	2.1	10/6/2016	NR	152		10/27/2016	No	No	NA	0.064	J B	62	1.8	0.06	
Boulder	MN16+BR+GSH-B	Shiner Mix	2.1	10/6/2016	NR	163		10/27/2016	No	No	NA	0.071	J B	65	0.52	0.49	J
Boulder	MN16+BR+GSH-C	Shiner Mix	2.1	10/6/2016	12	294		10/27/2016	No	No	NA	0.068	J B	62	2	0.04	
Boulder	MN16+BR+WAL-A	Walleye	4.5	10/6/2016	5	NR		10/27/2016	Yes	Yes	3	0.13	J B	140	2	0.16	J
Boulder	MN16+BR-WAL-B[12]	Walleye	4.5	10/6/2016	5	420		10/27/2016	Yes	Yes	5	0.11	J B	130	0.27	0.06	
Boulder	MN16+BR+WAL-C[12]	Walleye	4.5	10/6/2016	3	424		10/27/2016	Yes	Yes	3	0.098	J B	120	0.28	0.05	
Boulder	MN16+BR+WS-B	White Sucker	2.8	10/6/2016	3	3052		10/27/2016	Yes	Yes	3	0.071	J B	81	2.5	0.11	J
Boulder	MN16+BR+WS-A	White Sucker	2.8	10/6/2016	3	1847		10/27/2016	Yes	Yes	3	0.056	J B	57	2.2	0.15	J
Boulder	MN16+BR+WS-C	White Sucker	2.8	10/6/2016	3	4390		10/27/2016	Yes	Yes	3	0.051	J B	110	3.5	0.06	J

Notes:
NA - Not applicable
NR-Not Reported
J- The reported result is an estimation
B - The analyte is present in the associated method blank at a detectable level
MS/MSD - Matrix spike/Matrix spike duplicate
g - gram
mg/kg - milligram per kilogram
µg/kg - microgram per kilogram
ng TEQ/kg - nanogram per kilogram
TEQ - dioxins/furans toxicity equivalent
*http://fishbase.org/search.php

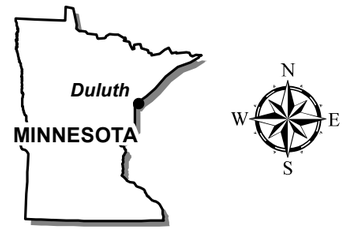
Figures

Figure 1

Site Location Map

Scanlon Reservoir
SLR Sediment AOCs

Duluth, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: National Geographic Society, i-cubed

0 2 4 Miles

0 3 6 Kilometers

Scanlon Reservoir Site Location

CLEAN WATER LAND & LEGACY AMENDMENT

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised:3/15/2017 Project No.J160749

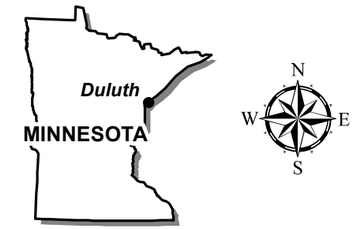
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_ReservoirMapDocs\J160749\J160749 FIG 1 Scanlon Reservoir Site Location Map.mxd

Figure 2

Site Map

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 200 400 Feet

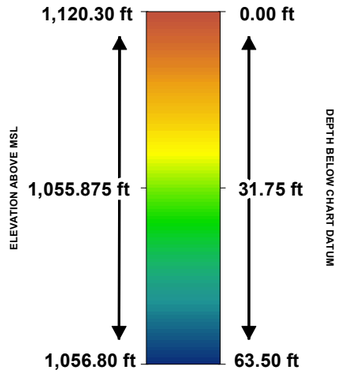
0 75 150 Meters

Bathymetry Contour Line (5ft Intervals)

Scanlon Reservoir Site Boundary

NOTE: Water elevation maintained at 1119.80 ft +/- 0.5 ft

Water Depth



(Based from MNDNR Lake Bathymetric Contours, 1996)

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Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:3/15/2017 Project No.J160749

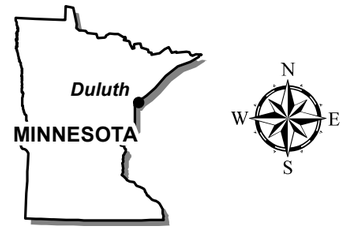
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\J160749 FIG 2 Scanlon Reservoir Site Map.mxd

Figure 3

Sample Locations

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 200 400 Feet

0 75 150 Meters

- 2016 Sediment Sample Locations
- 2016 Bioaccumulation Testing and Community Assessment Locations
- Scanlon Reservoir Site Boundary

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Drawn By: S.G. Date Drawn/Revised:4/4/2017 Project No.J160749

Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\J160749 FIG 3 Scanlon Reservoir Sample Locations.mxd

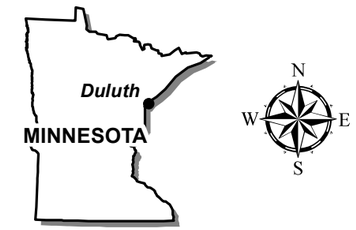
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\J160749 FIG 4 Scanlon Reservoir Poling Locations and Sediment Thicknesses.mxd

Figure 4

Poling Locations and Sediment Thicknesses

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

● Poling/Sample Location

Scanlon Reservoir Site Boundary

(0.98) Sediment Thickness in Feet

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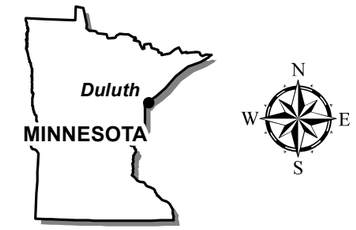
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\U160749\U160749 FIG 5 Scanlon Reservoir Mercury SQT Results.mxd

Figure 5

Mercury SQT Results

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 200 400 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Sample Type

- △ 2016 Sediment Sample, Including In-Situ Tissue
- ⛒ 2016 Sediment Sample and Lab Bioaccumulation Testing
- ○ 2016 Sediment Sample
- □ Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

Mercury SQT Comparison

- Does not exceed Level 1 SQT (0.18 mg/kg)
- Exceeds Level 1 SQT (0.18 mg/kg)
- Exceeds Midpoint SQT (0.64 mg/kg)
- Exceeds Level 2 SQT (1.1 mg/kg)

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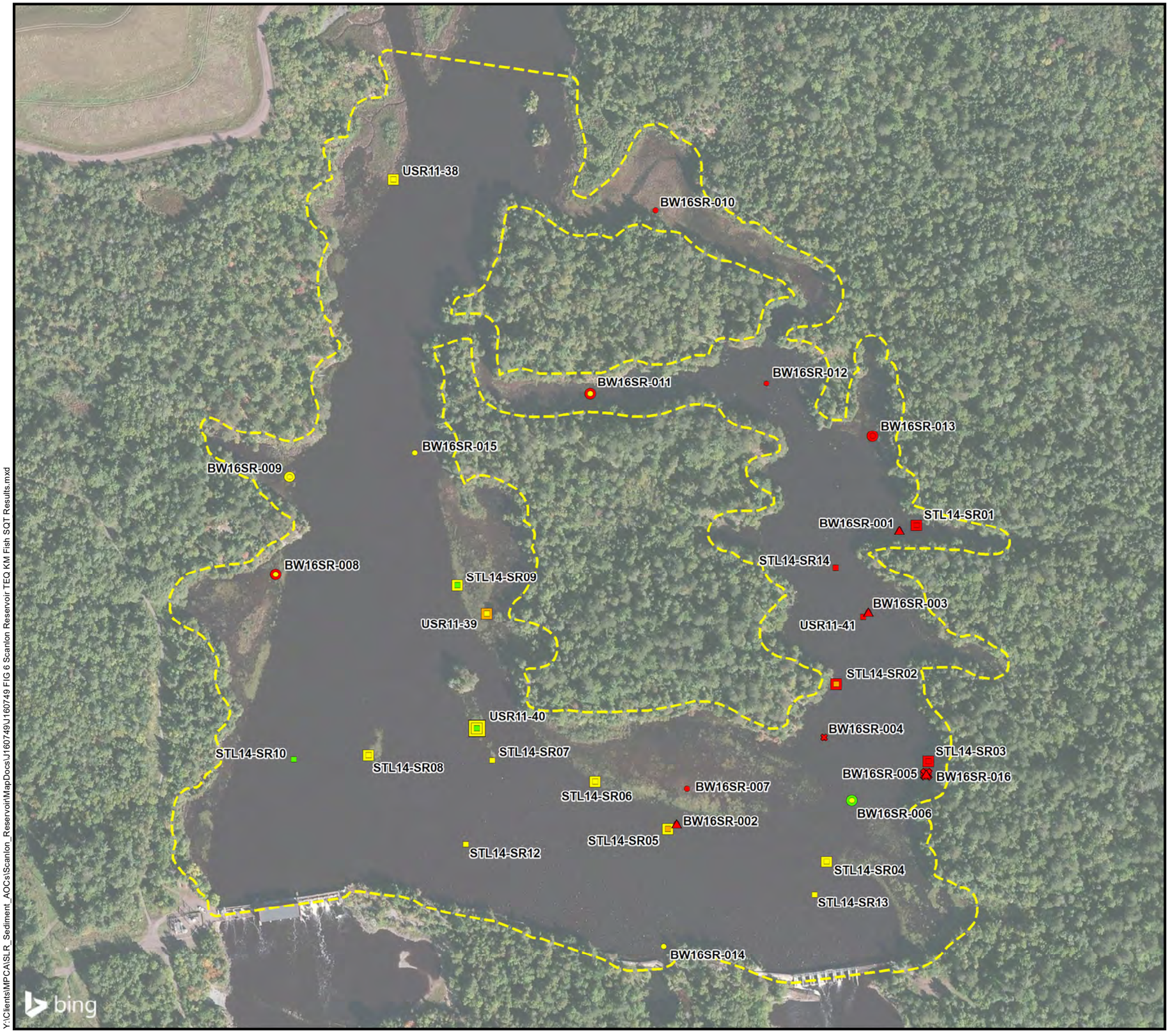
Drawn By: S.G. Date Drawn/Revised:6/13/2017 Project No.J160749



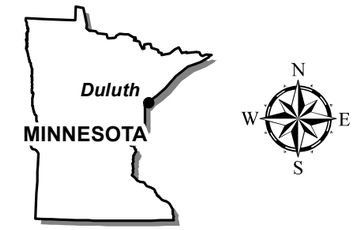
Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_ReservoirMapDocs\U160749\U160749 FIG 6 Scanlon Reservoir TEQ KM Fish SQT Results.mxd

Figure 6

TEQ KM Fish SQT Results

Scanlon Reservoir
SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:6/13/2017 Project No.J160749

Boulder Lake Reference	
HD-001-MRCS (Macrobenthos)	
Mercury Bioaccumulation (mg/kg)	0.032
Methyl Mercury Bioaccumulation (mg/kg)	4.3
BW16BLR-001 (Lumbriculus)	
Mercury Bioaccumulation (mg/kg)	0.032
Methyl Mercury Bioaccumulation (mg/kg)	0.038

Background Sample	
Background-O (Lumbriculus)	
Mercury Bioaccumulation (mg/kg)	0.038
Methyl Mercury Bioaccumulation (mg/kg)	0.088

1) U - Not detected
2) J - Estimated value
3) mg/kg - milligram/kilogram

BW16SR-001 (Mayfly)	
Mercury Bioaccumulation (mg/kg)	0.034
Methyl Mercury Bioaccumulation (mg/kg)	3.1

HD-001	
Mercury Bioaccumulation (mg/kg)	0.037
Methyl Mercury Bioaccumulation (mg/kg)	4.5

BW16SR-003 (Mayfly)	
Mercury Bioaccumulation (mg/kg)	0.036
Methyl Mercury Bioaccumulation (mg/kg)	4.5

BW16SR-005 (Dragonfly)	
Mercury Bioaccumulation (mg/kg)	0.03
Methyl Mercury Bioaccumulation (mg/kg)	25

BW16SR-004 (Lumbriculus)	
Mercury Bioaccumulation (mg/kg)	0.036
Methyl Mercury Bioaccumulation (mg/kg)	0.24

BW16SR-005 (Craw fish)	
Mercury Bioaccumulation (mg/kg)	0.03
Methyl Mercury Bioaccumulation (mg/kg)	18

BW16SR-016 (Lumbriculus)	
Mercury Bioaccumulation (mg/kg)	0.037
Methyl Mercury Bioaccumulation (mg/kg)	0.32

BW16SR-002 (Mayfly)	
Mercury Bioaccumulation (mg/kg)	0.031
Methyl Mercury Bioaccumulation (mg/kg)	0.029

BW16SR-002 (Dragonfly)	
Mercury Bioaccumulation (mg/kg)	0.029
Methyl Mercury Bioaccumulation (mg/kg)	23

USR11-38, BW16SR-010, BW16SR-011, BW16SR-012, BW16SR-013, BW16SR-015, BW16SR-009, BW16SR-008, SD5, BW16SR-001, STL14-SR01, STL14-SR14, STL14-SR09, USR11-39, BW16SR-003, USR11-41, STL14-SR02, USR11-40, BW16SR-004, SD2, STL14-SR10, STL14-SR08, STL14-SR07, STL14-SR03, BW16SR-016, BW16SR-005, BW16SR-007, STL14-SR06, SD4, BW16SR-006, BW16SR-002, SD1, STL14-SR05, STL14-SR12, STL14-SR04, STL14-SR13, BW16SR-014

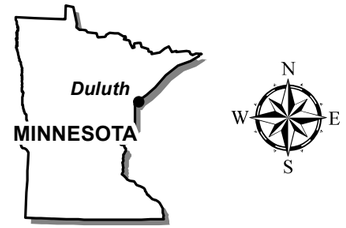
bing

Figure 7

Mercury and Methyl Mercury In Situ and Ex Situ Bioaccumulation Results

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 200 400 Feet
0 75 150 Meters

Scanlon Reservoir Site Boundary

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

Mercury SQT Comparison

- Does not exceed Level 1 SQT (0.18 mg/kg)
- Exceeds Level 1 SQT (0.18 mg/kg)
- Exceeds Midpoint SQT (0.64 mg/kg)
- Exceeds Level 2 SQT (1.1 mg/kg)

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:6/21/2017 Project No.J160749

Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\J160749\J160749 FIG 7 Scanlon Reservoir Mercury and Methyl Mercury Bioaccumulation Results.mxd

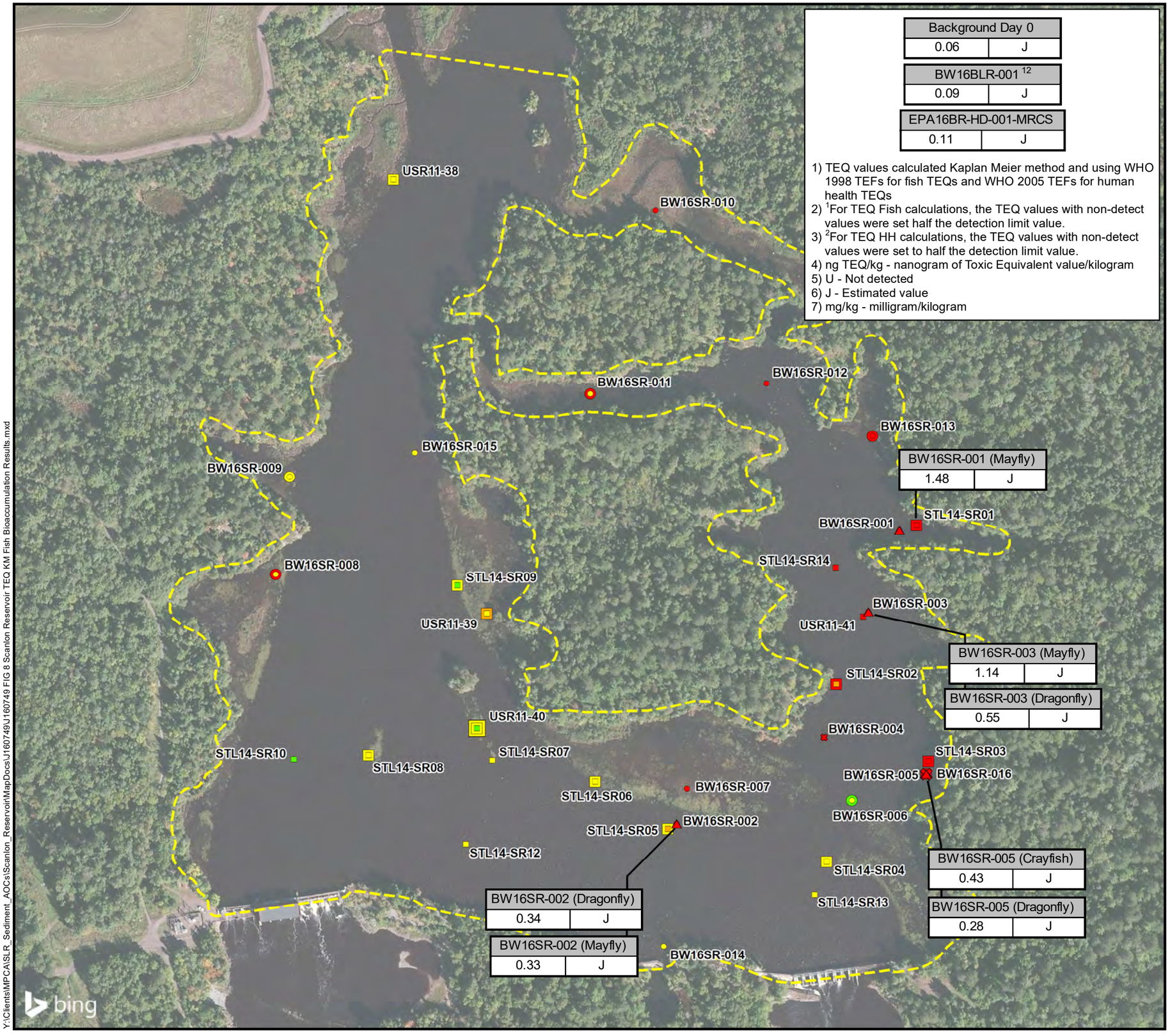
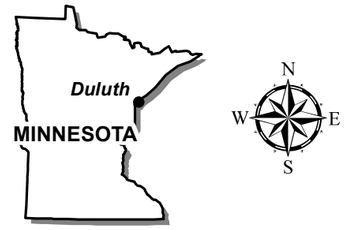


Figure 8

TEQ KM Fish In Situ and Ex Situ Bioaccumulation Results

Scanlon Reservoir SLR Sediment AOCs

Scanlon, MN



Map Projection: NAD 1983 UTM Zone 15 N
Basemap: Bing Aerial Imagery WMS (July-Sept 2011)

0 225 450 Feet

0 75 150 Meters

Scanlon Reservoir Site Boundary

Sample Type

- 2016 Sediment Sample, Including In-Situ Tissue
- 2016 Sediment Sample and Lab Bioaccumulation Testing
- 2016 Sediment Sample
- Historical Sediment Sample

Sample Interval

- 0-0.15 m
- 0.15-0.50 m
- 0.50-1.0 m
- >1.0 m

TEQ KM Fish SQT Comparison

- Does not exceed Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Level 1 SQT (0.85 ng TEQ/kg)
- Exceeds Midpoint SQT (11.2 ng TEQ/kg)
- Exceeds Level 2 SQT (21.5 ng TEQ/kg)

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: S.G. Date Drawn/Revised:6/21/2017 Project No.J160749

Y:\Clients\MPCA\SLR_Sediment_AOCs\Scanlon_Reservoir\MapDocs\U160749\U160749 FIG 8 Scanlon Reservoir TEQ KM Fish Bioaccumulation Results.mxd

Appendix A

Field Notes, Core Logs, and Photos

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name: SLR | Client: MPCA | Contractor: Bay West

Project #: J160139 | Site Location: Boulder Lake Reservoir | **Location ID:** BW16BLR-001

Core & Polling Collection Information

Sample Collectors: ACB, SMC, JMB

Date Collected: September 20, 2016 | Time Collected: 12:16 PM | Above/Below LWD (ft):

Water Elevation (ft): | Water Depth (ft): 8.0 | Sediment Elevation (ft):

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	254	289	315	61	Woody Debris	Silt Loam
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Ponar/Grab

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
-			0	—
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB, JMB, CJM

Length of Core (m): 0.15 | Date Processed: September 20, 2016 | Time Processed: 12:10 PM

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Sediment Characterization Log

Location ID: BW16BLR-001

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Very Dark Brown (10YR 2/2) | Secondary Color: Dark Brown (10YR 3/3)

USCS: PT | USDA: Peat | Grains: Rounded

Organics: Woody | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Very woody, 90%, some silt, <5%.

Layer 2:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Boulder Lake Reservoir **Sample/Location Name:** BW16BLR-001

Processors: ACB JMB **Date:** September 20, 2016 **Time:** 10:49 AM

Weather: Temperature (deg F): 70 Skies: Partly Cloudy Wind Speed (mph) & Direction: 5-10

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Multiple grabs

Habitat Information

Primary Color: Very Dark Brown (10YR 2/2) Secondary Color: Dark Brown (10YR 3/3)

USCS: PT USDA: Peat Grains: Well Rounded

Organics: Woody %: 75 - 100 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: None Cohesiveness: Loose

Description/Notes: Natural sheen, woody, 90%, some silt (<5%)

Notes:

Very woody organics, 90%, with some silt.

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*	Group 2 *(Semi-Sensitive)*	Group 3 *(Semi-Tolerant)*	Group 4 *(Tolerant)*
☐ Alderfly	☐ Caddisfly	☐ Black Fly	☐ Bloodworm Midge
☐ Dobsonfly	☐ Crane Fly	☐ Non-Red Midge	☐ Isopod/Sowbug
☐ Stonefly	☐ Crawfish	☐ Scud	☐ Leech
☐ Water Snipe Fly	☐ Damselfly	☐ Snails	☐ Tubifex Worm
	☐ Dragonfly		
	☐ Fingernail Clam		
	☐ Mayfly		
	☐ Riffle Beetle		
	☐ Water Penny		
Total # of Organisms: 0	Total # of Organisms: 0	Total # of Organisms: 0	Total # of Organisms: 0
Total # of Taxa:	Total # of Taxa:	Total # of Taxa:	Total # of Taxa:

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

☐ Other ☐ Other ☐ Other

Total # of Organisms: 0

Total # of Taxa:

Notes:

15 minute assessment performed no macroinvertebrates found.

TOTAL # of TAXA: 0

TOTAL # of ORGANISMS: 0

v.082016

Benthic Macroinvertebrate Sample Collection

Sample Location: BW16BLR-001

Target Macroinvertebrate Organism: Other (See notes)

Date: September 20, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)			0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	0	0

Notes:

No macroinvertebrates were submitted for analysis.

Sample Processing - Depuration

Start Date/Time:

End Date/Time:

Duration (hours):

Laboratory Sample Analysis

Sample ID:

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

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Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 20, 2016

Sample Location: BW16BLR-001

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name: SLR | Client: MPCA | Contractor: Bay West

Project #: J160139 | Site Location: Boulder Lake Reservoir | **Location ID:** BW16BLR-002

Core & Polling Collection Information

Sample Collectors: ACB, SC, JMB

Date Collected: September 20, 2016 | Time Collected: 1:25 PM | Above/Below LWD (ft):

Water Elevation (ft): | Water Depth (ft): 14.2 | Sediment Elevation (ft):

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	432	549	605	173	Sediment	Silt
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Ponar/Grab

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
—			0	—
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB, JMB, CJM

Length of Core (m): 0.15 | Date Processed: September 20, 2016 | Time Processed: 2:10 PM

v.072016

Sediment Characterization Log

Location ID: BW16BLR-002

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Very Dark Brown (10YR 2/2) | Secondary Color: Black (10YR 2/1)

USCS: ML | USDA: Silt Loam | Grains: Rounded

Organics: Woody | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Soft clayey silt, loose.

Layer 2:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 20, 2016

Location ID: BW16BLR-002

Photo 1:

Photo 2:

Photo 3:

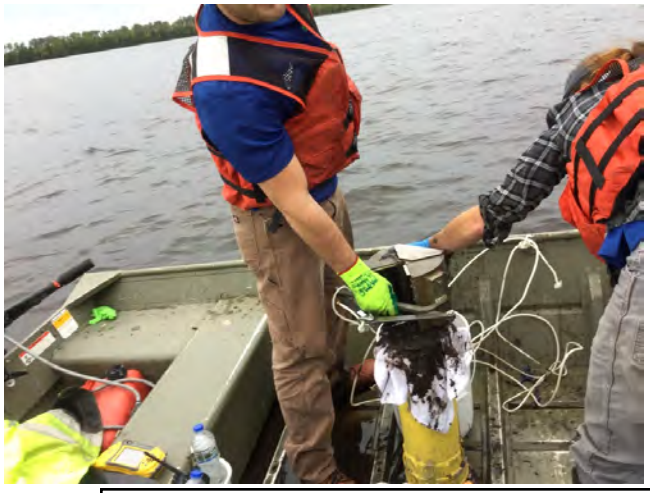

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name: SLR | Client: MPCA | Contractor: Bay West

Project #: J160139 | Site Location: Boulder Lake Reservoir | **Location ID:** BW16BLR-003

Core & Polling Collection Information

Sample Collectors: ACB | JMB | SC

Date Collected: September 21, 2016 | Time Collected: 10:17 AM | Above/Below LWD (ft):

Water Elevation (ft): | Water Depth (ft): 7.5 | Sediment Elevation (ft):

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	239	249	272	33	Sediment	Silt Loam
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Ponar/Grab

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
—			0	—
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | JMB | CJM

Length of Core (m): 0.15 | Date Processed: September 20, 2016 | Time Processed: 10:30 AM

v.072016

Sediment Characterization Log

Location ID: BW16BLR-003

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Very Dark Brown (10YR 2/2) | Secondary Color: Black (10YR 2/1)

USCS: ML | USDA: Silt Loam | Grains: Rounded

Organics: Fibrous | %: 10 - 25 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Silty with some fine sand, loose with long fibrous woody material.

Layer 2:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 21, 2016

Location ID: BW16BLR-003

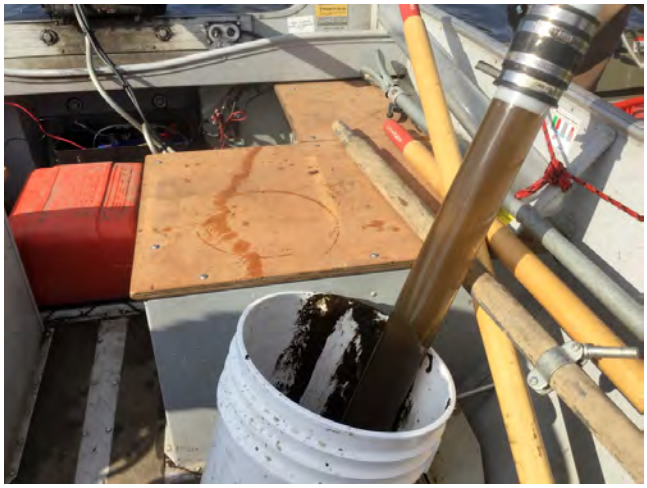

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Scanlon Reservoir **Sample/Location Name:** BW16SR-001

Processors: ACB JMB **Date:** September 21, 2016 **Time:** 3:46 PM

Weather: Temperature (deg F): 70 Skies: Partly Sunny Wind Speed (mph) & Direction: 10

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Habitat Information

Primary Color: Dark Brown (10YR 3/3) Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML USDA: Silt Loam Grains: Well Rounded

Organics: Fibrous %: 5 - 10 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: Sheen Cohesiveness: Loose

Description/Notes: Silty clay loam with some very fine sand. Stiff Clay plug below silty loam layer. Slight sheen.

Notes:

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*		Group 2 *(Semi-Sensitive)*		Group 3 *(Semi-Tolerant)*		Group 4 *(Tolerant)*	
	Alderfly		Caddisfly		Black Fly	3	Bloodworm Midge
	Dobsonfly		Crane Fly		Non-Red Midge		Isopod/Sowbug
	Stonefly		Crawfish		Scud		Leech
	Water Snipe Fly		Damselfly	2	Snails		Tubifex Worm
			Dragonfly				
		1	Fingernail Clam				
		4	Mayfly				
			Riffle Beetle				
			Water Penny				
Total # of Organisms:	0	Total # of Organisms:	5	Total # of Organisms:	2	Total # of Organisms:	3
Total # of Taxa:		Total # of Taxa:	2	Total # of Taxa:	1	Total # of Taxa:	1

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

						Total # of Organisms:	7
1	Other	Horsehair Worm		Other	Thread worm	Total # of Taxa:	2
				Other			

Notes:

15 minute assessment.

TOTAL # of TAXA: 6

TOTAL # of ORGANISMS: 17

v.082016

Benthic Macroinvertebrate Sample Collection

Sample Location: BW16SR-001

Target Macroinvertebrate Organism: Mayfly (Hexagenia limbata)

Date: September 21, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)		7.14	0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	7.14	0

Notes:

7.14g (mayfly) + 32.22g (151 mayflies) = 39.36g
33.8g (snail)

Sample Processing - Depuration

Start Date/Time: September 21, 2016 at 4:21 PM

End Date/Time: September 22, 2016 at 11:28 AM

Duration (hours): 19

Laboratory Sample Analysis

Sample ID: BW16SR-001

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 21, 2016

Sample Location: BW16SR-001

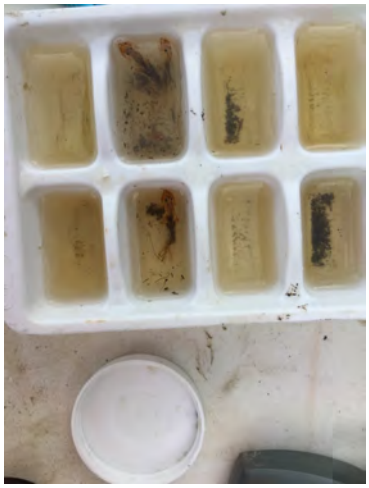

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Scanlon Reservoir **Sample/Location Name:** BW16SR-002

Processors: ACB JMB CJM **Date:** September 28, 2016 **Time:** 1:43 PM

Weather: Temperature (deg F): 65 Skies: Cloudy Wind Speed (mph) & Direction: 0-5

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Habitat Information

Primary Color: Very Dark Brown (10YR 2/2) Secondary Color: Dark Brown (10YR 3/3)

USCS: ML USDA: Silt Loam Grains: Well Rounded

Organics: Woody %: 25 - 50 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: None Cohesiveness: Loose

Description/Notes: Silty loam some very fine grain sand. Wood chunks 25%.

Notes:

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*		Group 2 *(Semi-Sensitive)*		Group 3 *(Semi-Tolerant)*		Group 4 *(Tolerant)*	
	Alderfly		Caddisfly		Black Fly	2	Bloodworm Midge
	Dobsonfly		Crane Fly		Non-Red Midge		Isopod/Sowbug
	Stonefly		Crawfish		Scud		Leech
	Water Snipe Fly		Damselfly	4	Snails		Tubifex Worm
			Dragonfly				
			Fingernail Clam				
		1	Mayfly				
			Riffle Beetle				
			Water Penny				
Total # of Organisms:	0	Total # of Organisms:	1	Total # of Organisms:	4	Total # of Organisms:	2
Total # of Taxa:		Total # of Taxa:	1	Total # of Taxa:	1	Total # of Taxa:	1

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

	Other			Other		Total # of Organisms:	1
1	Other	Horsetail		Other		Total # of Taxa:	1

Notes:

15 minute assessment

TOTAL # of TAXA: 4

TOTAL # of ORGANISMS: 8

v.082016

Benthic Macroinvertebrate Sample Collection

Sample Location: BW16SR-002

Target Macroinvertebrate Organism: Other (See notes)

Date: September 28, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)			0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	0	0

Notes:

40.43g (Mayfly)
51.72g (Dragon fly)
132g (Snails)

Sample Processing - Depuration

Start Date/Time: September 28, 2016 at 3:13 PM

End Date/Time: September 29, 2016 at 9:04 AM

Duration (hours): 18

Laboratory Sample Analysis

Sample ID:

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 28, 2016

Sample Location: BW16SR-002

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Scanlon Reservoir **Sample/Location Name:** BW16SR-003

Processors: ACB JMB **Date:** September 22, 2016 **Time:** 10:19 AM

Weather: Temperature (deg F): 62 Skies: Cloudy Wind Speed (mph) & Direction: 0-5

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Habitat Information

Primary Color: Dark Brown (10YR 3/3) Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML USDA: Silt Loam Grains: Well Rounded

Organics: Woody %: 5 - 10 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: Sheen Cohesiveness: Loose

Description/Notes: Slight sheen, silty clay loam, clay globs form when sifting

Notes:

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*		Group 2 *(Semi-Sensitive)*		Group 3 *(Semi-Tolerant)*		Group 4 *(Tolerant)*	
2	Alderfly		Caddisfly		Black Fly	3	Bloodworm Midge
	Dobsonfly		Crane Fly		Non-Red Midge		Isopod/Sowbug
	Stonefly		Crawfish		Scud		Leech
	Water Snipe Fly		Damselfly	4	Snails		Tubifex Worm
			Dragonfly				
			Fingernail Clam				
		5	Mayfly				
			Riffle Beetle				
			Water Penny				
Total # of Organisms:	2	Total # of Organisms:	5	Total # of Organisms:	4	Total # of Organisms:	3
Total # of Taxa:	1	Total # of Taxa:	1	Total # of Taxa:	1	Total # of Taxa:	1

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

	Other		8	Other	Thread worm	Total # of Organisms:	8
	Other			Other		Total # of Taxa:	1

Notes:

15 minutes assessment

TOTAL # of TAXA: 5

TOTAL # of ORGANISMS: 22

v.082016

Benthic Macroinvertebrate Sample Collection

Sample Location: BW16SR-003

Target Macroinvertebrate Organism: Other (See notes)

Date: September 22, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)			0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	0	0

Notes:

14.9g (54 mayfly) + 19.25g (mayfly)+ 17.52g = 51.67g
64.6g (snail)
17.35g (Dragonfly)

Sample Processing - Depuration

Start Date/Time: October 3, 2016 at 4:10 PM

End Date/Time: October 4, 2016 at 3:44 PM

Duration (hours): 23

Laboratory Sample Analysis

Sample ID: BW16SR-003

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

v.082016

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Sample Location: BW16SR-003

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Scanlon Reservoir **Sample/Location Name:** BW16SR-004

Processors: ACB JMB **Date:** September 28, 2016 **Time:** 3:28 PM

Weather: Temperature (deg F): 67 Skies: Cloudy Wind Speed (mph) & Direction: 0-5

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Habitat Information

Primary Color: Very Dark Brown (10YR 2/2) Secondary Color: Dark Brown (10YR 3/3)

USCS: ML USDA: Silt Loam Grains: Well Rounded

Organics: Fibrous %: 0 - 5 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: None Cohesiveness: Loose

Description/Notes: Silty loam with very fine sand, very few fibrous woody debris <5%

Notes:

BW16SR-004-0.0-0.15 @1115
TOC, Dioxin, Grain size, Mercury, moisture
5 jars

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*		Group 2 *(Semi-Sensitive)*		Group 3 *(Semi-Tolerant)*		Group 4 *(Tolerant)*	
	Alderfly		Caddisfly		Black Fly	3	Bloodworm Midge
	Dobsonfly		Crane Fly		Non-Red Midge		Isopod/Sowbug
	Stonefly		Crawfish		Scud		Leech
	Water Snipe Fly		Damselfly		Snails		Tubifex Worm
			Dragonfly				
			Fingernail Clam				
			Mayfly				
			Riffle Beetle				
			Water Penny				
Total # of Organisms:	0	Total # of Organisms:	0	Total # of Organisms:	0	Total # of Organisms:	3
Total # of Taxa:		Total # of Taxa:		Total # of Taxa:		Total # of Taxa:	1

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

1	Other	Horsetail	3	Other	Thread worm	Total # of Organisms:	4
				Other		Total # of Taxa:	2

Notes:

15 minute assessment

TOTAL # of TAXA: 3

TOTAL # of ORGANISMS: 7

v.082016

Benthic Macroinvertebrate Sample Collection

Sample Location: BW16SR-004

Target Macroinvertebrate Organism: Other (See notes)

Date: September 28, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)			0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	0	0

Notes:

No macroinvertebrates were submitted for analysis.

Sample Processing - Depuration

Start Date/Time:

End Date/Time:

Duration (hours):

Laboratory Sample Analysis

Sample ID:

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 30, 2016

Sample Location: BW16SR-004

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Benthic Macroinvertebrate Worksheet

Project/Site Information

Project Name: SLR **Project #:** J160139 **Client:** MPCA **Contractor:** Bay West

Site Name: Scanlon Reservoir **Sample/Location Name:** BW16SR-005

Processors: ACB JMB **Date:** September 28, 2016 **Time:** 9:48 AM

Weather: Temperature (deg F): 66 Skies: Partly Sunny Wind Speed (mph) & Direction: 0-5

Sample Collection Information

Method: Ponar

Number of Grabs: 3 **Approximate Collection Area (cm2):** 675

Notes: **Each grab = 15.2 cm x 15.2 cm (225 cm2)**

Habitat Information

Primary Color: Dark Brown (10YR 3/3) Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML USDA: Silt Loam Grains: Well Rounded

Organics: Fibrous %: 0 - 5 Odor: No Odor

Rocks: None %: N/A Moisture: Saturated

Petrochemical: None Cohesiveness: Loose

Description/Notes: Silty loam with very fine sand, very few fibrous woody debris <5%

Notes:

3.5g = 4 dragon fly, 9.2g dragonfly, 6.6g dragonfly-like

BW16SR-005-0.0-0.15 @1120
TOC, Dioxin, Grain size, Mercury, moisture
Dioxin & mercury dups (BW16SR-105-0.0-0.15) @1220
7 jars

Benthic Macroinvertebrate Community Assessment

Each grab = 15.2 cm x 15.2 cm (225 cm2)

Group 1 *(Sensitive)*		Group 2 *(Semi-Sensitive)*		Group 3 *(Semi-Tolerant)*		Group 4 *(Tolerant)*	
	Alderfly		Caddisfly		Black Fly	5	Bloodworm Midge
	Dobsonfly		Crane Fly		Non-Red Midge		Isopod/Sowbug
	Stonefly		Crawfish		Scud		Leech
	Water Snipe Fly		Damselfly	3	Snails		Tubifex Worm
			Dragonfly				
			Fingernail Clam				
			Mayfly				
			Riffle Beetle				
			Water Penny				
Total # of Organisms:	0	Total # of Organisms:	0	Total # of Organisms:	3	Total # of Organisms:	5
Total # of Taxa:	0	Total # of Taxa:	0	Total # of Taxa:	1	Total # of Taxa:	1

Miscellaneous Benthic Macroinvertebrates

(Not included in lists above.)

	Other		Other		Total # of Organisms:	4	
4	Other	Thread worm	Other		Total # of Taxa:	1	

Notes:

15 minutes assessment

TOTAL # of TAXA: 3

TOTAL # of ORGANISMS: 12

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Benthic Macroinvertebrate Sample Collection

Sample Location: BW16SR-005

Target Macroinvertebrate Organism: Other (See notes)

Date: September 28, 2016

Organism Size	Quantity	Wet Weight (g)	Individual Wet Weight (g)
Large (>/= 20 mm)			0
Medium (10-19 mm)			0
Small (< 9 mm)			0
	Total	**Total**	**Average**
	0	0	0

Notes:

88g (Snails)
48.4g (Dragon fly)
37g (Crayfish)

Sample Processing - Depuration

Start Date/Time: September 28, 2016 at 3:10 PM

End Date/Time: September 29, 2016 at 9:38 AM

Duration (hours):

Laboratory Sample Analysis

Sample ID:

Sample Date/Time:

Laboratory: —

☐ PAHs 17 ☐ VOCs ☐ Dioxins ☐ PCBs ☐ pH ☐ Moisture ☐ TOC ☐ Grain Size

☐ Select Metals ☐ Ar ☐ Cd ☐ Cr ☐ Cu ☐ Hg ☐ Ni ☐ Pb

☐ MS/MSD —

☐ Other Compound: —

☐ Duplicate —

Sample ID:

Dup Time:

Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 28, 2016

Sample Location: BW16SR-005

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name: SLR | Client: MPCA | Contractor: Bay West

Project #: J160139 | Site Location: Scanlon Reservoir | **Location ID:** BW16SR-001

Core & Polling Collection Information

Sample Collectors: ACB | CJM | JMB

Date Collected: September 21, 2016 | Time Collected: 1:35 PM | Above/Below LWD (ft):

Water Elevation (ft): | Water Depth (ft): 7.4 | Sediment Elevation (ft):

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Ponar/Grab

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
—			0	—
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | JMB | CJM

Length of Core (m): 0.5 | Date Processed: September 23, 2016 | Time Processed: 1:47 PM

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Sediment Characterization Log

Location ID: BW16SR-001

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Fibrous | %: 5 - 10 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: Sheen | Cohesiveness: Loose

Description/ Notes: Silty clay loam with some very fine sand. Stiff Clay plug below silty loam layer. Slight sheen.

Layer 2:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 21, 2016

Location ID: BW16SR-001

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name: SLR | Client: MPCA | Contractor: Bay West

Project #: J160139 | Site Location: Scanlon Reservoir | **Location ID:** BW16SR-003

Core & Polling Collection Information

Sample Collectors: ACB | JMB | CJM

Date Collected: September 22, 2016 | Time Collected: 10:09 AM | Above/Below LWD (ft):

Water Elevation (ft): | Water Depth (ft): 7.2 | Sediment Elevation (ft):

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Ponar/Grab

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
—			0	—
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | CJM | JMB

Length of Core (m): 0.15 | Date Processed: September 30, 2016 | Time Processed: 10:34 AM

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Sediment Characterization Log

Location ID: BW16SR-003

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Woody | %: 5 - 10 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: Sheen | Cohesiveness: Loose

Description/ Notes: Slight sheen, silty clay loam, clay globs form when sifting

Layer 2:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-003

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-006

Core & Polling Collection Information

Sample Collectors: CJM | ACB |

Date Collected:	September 22, 2016	Time Collected:	3:42 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	8.4	Sediment Elevation (ft):	

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	256	416	416	160	Sediment	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	3.2	1.7	53.13	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | CJM | SC

Length of Core (m):	0.52	Date Processed:	September 23, 2016	Time Processed:	9:37 AM

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Sediment Characterization Log

Location ID: BW16SR-006

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Woody | %: 5 - 10 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Crust at 144". Peat at 1.7 ft
Very loose silty clay

Layer 2:

Start Depth (m): 0.15 | End Depth (m): 0.2

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Woody | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Slightly more woody

Layer 3:

Start Depth (m): 0.2 | End Depth (m): 0.24

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Dark Grey Brown (10YR 3/2)

USCS: PT | USDA: Peat | Grains: Rounded

Organics: Woody | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

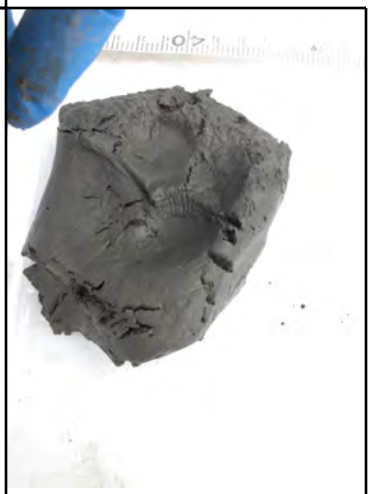
Petrochemical: None | Cohesiveness: Stiff

Description/ Notes:

Sediment Characterization Log

Location ID: BW16SR-006

Bay West



Layer 4:

Start Depth (m): 0.24 | End Depth (m): 0.52

Primary Color: Dark Grey Brown (10YR 3/2) | Secondary Color: Grey (10YR 6/1)

USCS: CL-ML | USDA: Silty Clay | Grains: Rounded

Organics: Fibrous | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: With very fine sand

Layer 5:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Layer 6:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-006

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-007

Core & Polling Collection Information

Sample Collectors: ACB | CJM |

Date Collected:	September 22, 2016	Time Collected:	4:03 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	0.5	Sediment Elevation (ft):	

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	1.3	1.1	84.62	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | JMB |

Length of Core (m):	0.31	Date Processed:	September 23, 2016	Time Processed:	9:58 AM

Sediment Characterization Log

Location ID: BW16SR-007

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.15

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: SM | USDA: Sandy Loam | Grains: Rounded

Organics: None | %: N/A | Odor: Petrochemical

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Medium Density

Description/ Notes: Fine sand, slight petro odor

Layer 2:

Start Depth (m): 0.15 | End Depth (m): 0.18

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: PT | USDA: Peat | Grains: Rounded

Organics: Woody | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: — | Cohesiveness: Loose

Description/ Notes: Peat layer

Layer 3:

Start Depth (m): 0.18 | End Depth (m): 0.31

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: SC-SM | USDA: Sandy Loam | Grains: Rounded

Organics: Woody | %: 10 - 25 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Fine sand with silt, some woody organics

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-007

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-008

Core & Polling Collection Information

Sample Collectors: ACB | CJM |

Date Collected:	September 22, 2016	Time Collected:	4:23 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	0.2	Sediment Elevation (ft):	

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	2.1	1.3	61.9	Yes
2	2.0	1.65	82.5	Yes
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | SC | CJM

Length of Core (m):	0.48	Date Processed:	September 23, 2016	Time Processed:	10:21 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-008

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.06

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: SM | USDA: Sandy Loam | Grains: Rounded

Organics: None | %: N/A | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Soft silty fine sand.

Layer 2:

Start Depth (m): 0.06 | End Depth (m): 0.30

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: SM | USDA: Sandy Loam | Grains: Rounded

Organics: Woody | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Snail shell at 0.10, wood chunk at 0.25. Silty sand. Stiffer

Layer 3:

Start Depth (m): 0.30 | End Depth (m): 0.48

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Light Brown (10YR 6/3)

USCS: SM | USDA: Sandy Loam | Grains: Rounded

Organics: Woody | %: 25 - 50 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Fine sand some. Stiff, breaks in chunks.

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-008

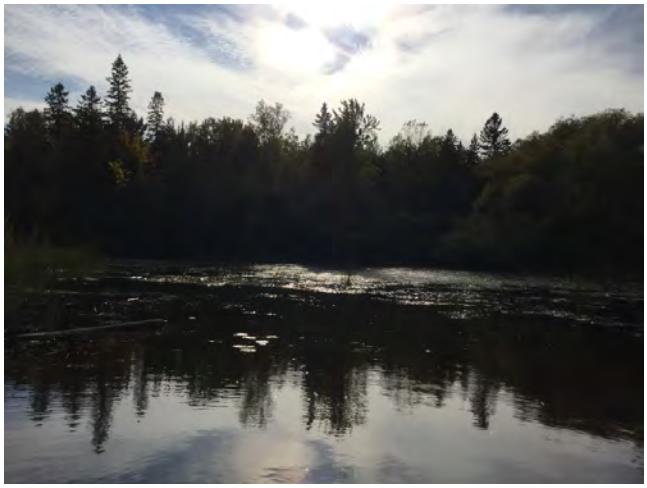


Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-009

Core & Polling Collection Information

Sample Collectors: ACB | CJM |

Date Collected:	September 22, 2016	Time Collected:	4:13 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	0.5	Sediment Elevation (ft):	

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	1.8	1.4	77.78	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | SMC | JMB

Length of Core (m):	0.42	Date Processed:	September 23, 2016	Time Processed:	10:36 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-009

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.25

Primary Color: Brown (10YR 5/3) | Secondary Color: Dark Brown (10YR 3/3)

USCS: SP | USDA: Sandy Loam | Grains: Rounded

Organics: Woody | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Medium sand, well sorted

Layer 2:

Start Depth (m): 0.25 | End Depth (m): 0.42

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Dark Grey Brown (10YR 3/2)

USCS: CL-ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Woody | %: 5 - 10 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Clayey silt with woody debris

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-009

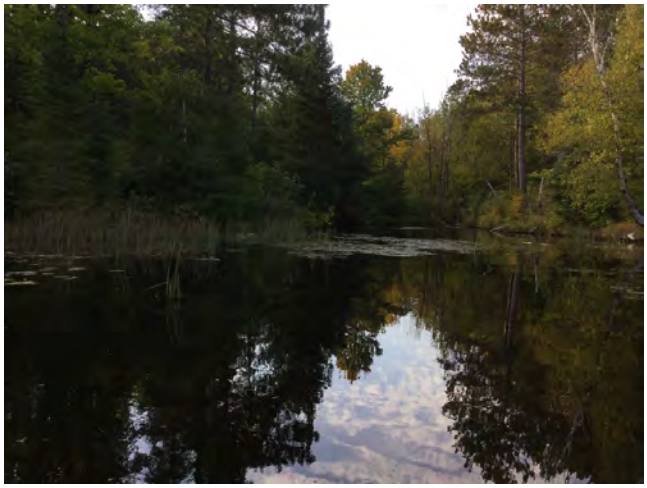


Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-010

Core & Polling Collection Information

Sample Collectors: ACB | CJM |

Date Collected:	September 22, 2016	Time Collected:	2:22 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	1.4	Sediment Elevation (ft):	

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	1.2	0.9	75	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | SMC | JMB

Length of Core (m):	0.24	Date Processed:	September 23, 2016	Time Processed:	10:43 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-010

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.2

Primary Color: Brown (10YR 5/3) | Secondary Color: Dark Brown (10YR 3/3)

USCS: ML | USDA: Silt Loam | Grains: Rounded

Organics: Woody | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Soft woody silt. Large wood chunk at 0.18

Layer 2:

Start Depth (m): 0.2 | End Depth (m): 0.25

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Very Dark Brown (10YR 2/2)

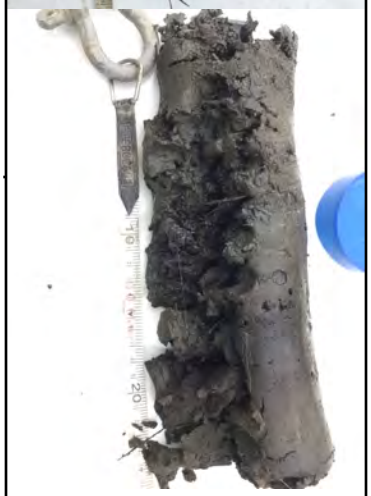
USCS: ML | USDA: Silt Loam | Grains: Rounded

Organics: Fibrous | %: 0 - 5 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Very fine sand-silt



Layer 3:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-010

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-011

Core & Polling Collection Information

Sample Collectors: ACB, CJM

Date Collected:	September 22, 2016	Time Collected:	2:44 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	0.7	Sediment Elevation (ft):	

Poling Collection Information

Equipment: N/A

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
				0	—	—
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	1.6	1.4	87.5	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB, CJM, JMB

Length of Core (m):	0.41	Date Processed:	September 23, 2016	Time Processed:	10:57 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-011

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.14

Primary Color:	Dark Brown (10YR 3/3)	Secondary Color:	Very Dark Brown (10YR 2/2)		
USCS:	ML	USDA:	Silt Loam	Grains:	Rounded
Organics:	Woody	%:	25 - 50	Odor:	No Odor
Rocks:	None	%:	N/A	Moisture:	Moist
Petrochemical:	None	Cohesiveness:	Loose		

Description/ Notes: Fine sand-silt with woody debris

Layer 2:

Start Depth (m): 0.14 | End Depth (m): 0.18

Primary Color:	Brown (10YR 5/3)	Secondary Color:	Dark Brown (10YR 3/3)		
USCS:	PT	USDA:	Peat	Grains:	Rounded
Organics:	Woody	%:	75 - 100	Odor:	No Odor
Rocks:	None	%:	N/A	Moisture:	Moist
Petrochemical:	None	Cohesiveness:	Stiff		

Description/ Notes: Peat with fine sand/silt

Layer 3:

Start Depth (m): 0.18 | End Depth (m): 0.41

Primary Color:	Dark Grey Brown (10YR 3/2)	Secondary Color:	Very Dark Brown (10YR 2/2)		
USCS:	CL-ML	USDA:	Silty Clay	Grains:	Rounded
Organics:	Woody	%:	5 - 10	Odor:	No Odor
Rocks:	None	%:	N/A	Moisture:	Moist
Petrochemical:	None	Cohesiveness:	Stiff		

Description/ Notes: Silty clay with some fibrous woòdy debris

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-011

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-012

Core & Polling Collection Information

Sample Collectors: ACB, CJM

Date Collected:	September 22, 2016	Time Collected:	2:59 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	5.5	Sediment Elevation (ft):	

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	170	170	312	142	Sediment	Peat
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	4.5	1.3	28.89	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB, CJM, JMB

Length of Core (m):	0.41	Date Processed:	September 23, 2016	Time Processed:	11:06 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-012

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.10

Primary Color: Dark Grey Brown (10YR 3/2) | Secondary Color: Dark Grey (10YR 4/1)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: None | %: N/A | Odor: No Odor

Rocks: None | %: N/A | Moisture: Saturated

Petrochemical: None | Cohesiveness: Loose

Description/ Notes: Couldn't get past peat layer plug
Silty loam, no structure

Layer 2:

Start Depth (m): 0.10 | End Depth (m): 0.17

Primary Color: Dark Grey Brown (10YR 3/2) | Secondary Color: Dark Brown (10YR 3/3)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: Woody | %: 50 - 75 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Silt loam with woody debris

Layer 3:

Start Depth (m): 0.17 | End Depth (m): 0.25

Primary Color: Dark Grey Brown (10YR 3/2) | Secondary Color: Dark Grey (10YR 4/1)

USCS: ML | USDA: Silt Loam | Grains: Well Rounded

Organics: None | %: N/A | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Same as layer 1 more structure

Sediment Characterization Log

Location ID: BW16SR-012

Bay West

Layer 4:

Start Depth (m): 0.25 | End Depth (m): 0.31

Primary Color: Dark Brown (10YR 3/3) | Secondary Color: Brown (10YR 5/3)

USCS: OH | USDA: Other (see Notes) | Grains: Well Rounded

Organics: Wood Chips | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Woody layer with some silt
Did not collect sample at bottom 25cm because of woody and peat layers

Layer 5:

Start Depth (m): 0.31 | End Depth (m): 0.41

Primary Color: Reddish Brown | Secondary Color: Very Dark Brown (10YR 2/2)

USCS: PT | USDA: Peat | Grains: Well Rounded

Organics: Other (see Notes) | %: 75 - 100 | Odor: No Odor

Rocks: None | %: N/A | Moisture: Moist

Petrochemical: None | Cohesiveness: Stiff

Description/ Notes: Peat with some silt

Layer 6:

Start Depth (m): | End Depth (m):

Primary Color: — | Secondary Color: —

USCS: — | USDA: — | Grains: —

Organics: — | %: — | Odor: —

Rocks: — | %: — | Moisture: —

Petrochemical: — | Cohesiveness: —

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160139 | Photographs taken on: September 22, 2016

Location ID: BW16SR-012

Photo 1:

Photo 2:

Photo 3:

Photo 4:

Photo 5:

Photo 6:

Sediment Collection & Characterization Core Log

Bay West
Customer-Focused Environmental & Industrial Solutions

Project/Site Information

Project Name:	SLR	Client:	MPCA	Contractor:	Bay West
Project #:	J160139	Site Location:	Scanlon Reservoir	**Location ID:**	BW16SR-013

Core & Polling Collection Information

Sample Collectors: ACB | CJM |

Date Collected:	September 22, 2016	Time Collected:	3:21 PM	Above/Below LWD (ft):	
Water Elevation (ft):		Water Depth (ft):	2.9	Sediment Elevation (ft):	

Poling Collection Information

Equipment: Rods

Location ID	Depth of Water (cm)	Depth to Resistance (cm)	Depth to Refusal (cm)	"Soft" Sediment Thickness (cm)	Refusal Type	Sediment Type Approaching Refusal
PL-01	*74*	*90*	*101*	*27*	*Sediment*	*Silty Clay*
PL-01	88	129	129	41	Sediment	Peat
				0	—	—
				0	—	—

Core Collection Information

Collection Method: Check Valve

Push Attempts	Push Depth (ft)	Push Recovery (ft)	% Recovery	Retained?
1	1.8	1.2	66.67	Yes
—			0	—
—			0	—
—			0	—
—			0	—

Core Processing Information

Sample Processors: ACB | CJM | JMB

Length of Core (m):	0.36	Date Processed:	September 23, 2016	Time Processed:	11:24 AM

v.072016

Sediment Characterization Log

Location ID: BW16SR-013

Bay West

Layer 1:

Start Depth (m): 0.0 | End Depth (m): 0.21

Primary Color:	Dark Brown (10YR 3/3)	Secondary Color:	Very Dark Brown (10YR 2/2)		
USCS:	PT	USDA:	Peat	Grains:	Rounded
Organics:	Woody	%:	75 - 100	Odor:	Other (see Notes)
Rocks:	None	%:	N/A	Moisture:	Saturated
Petrochemical:	Other (see Notes)	Cohesiveness:	Loose		

Description/ Notes: Woody with silt, slight sheen and petrochemical odor

Layer 2:

Start Depth (m): 0.21 | End Depth (m): 0.36

Primary Color:	Dark Grey Brown (10YR 3/2)	Secondary Color:	Dark Grey (10YR 4/1)		
USCS:	SM	USDA:	Sandy Loam	Grains:	Well Rounded
Organics:	None	%:	N/A	Odor:	No Odor
Rocks:	None	%:	N/A	Moisture:	Moist
Petrochemical:	None	Cohesiveness:	Other (see Notes)		

Description/ Notes: Very fine sand with silt increasing stiffness down core

Layer 3:

Start Depth (m): | End Depth (m):

Primary Color:	—	Secondary Color:	—		
USCS:	—	USDA:	—	Grains:	—
Organics:	—	%:	—	Odor:	—
Rocks:	—	%:	—	Moisture:	—
Petrochemical:	—	Cohesiveness:	—		

Description/ Notes:

Photographic Log

Project Name: SLR | Project Number: J160136 | Photographs taken on: September 22, 2016

Location ID: BW16SR-013

Photo 1:

Photo 2:

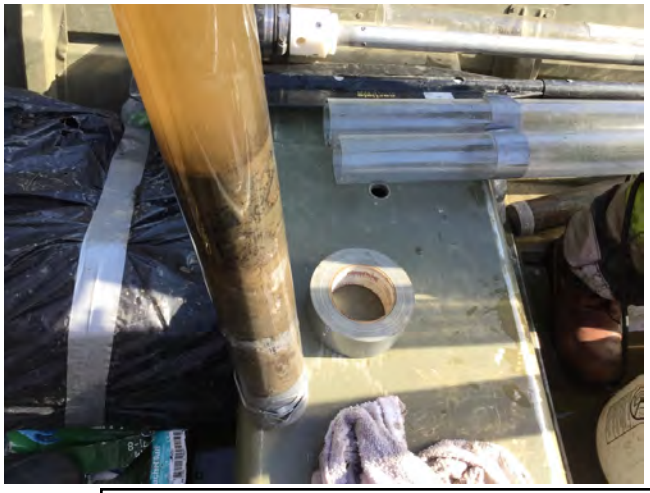


Photo 3:

Photo 4:

Photo 5:

Photo 6:

Appendix B

2016 Tissue Analysis Project Plan for Duluth Reservoirs Draft Report

2016 Tissue Analysis Project Plan for Duluth Reservoirs
Draft Report

Contract No. W911XK-16-D-0014-0005

Prepared for:
U.S. Army Corps of Engineers
Detroit District
477 Michigan Avenue
Detroit, Michigan 48226

Attn: Pam Horner

Prepared by:
Advanced Environmental Management Group
44339 Plymouth Oaks Blvd.
Plymouth, Michigan, 48170-2585
March 10, 2017
F16705

Table of Contents

Appendices

Appendix A – Scanlon Reservoir Whole Fish COCs and Fish Sampling Field Logs

Appendix B – Thomson Reservoir Whole Fish COCs and Fish Sampling Field Logs

Appendix C – Boulder Lake Reservoir Whole Fish COCs and Fish Sampling Field Logs

Appendix D – GLEC Fish and Macroinvertebrate Tissue Processing Report

Appendix E – GLEC Fish Information Summary Tables

Appendix F – EPA and MCPA Macroinvertebrate COCs and Instructions

Appendix G – Scanlon Reservoir Fish Samples Analytical Results Summary Table

Appendix H – Thomson Reservoir Fish Samples Analytical Results Summary Table

Appendix I – Boulder Lake Reservoir Fish Samples Analytical Results Summary Table

Appendix J – TA Report – J180-60593

Appendix K – TA Report – J180-60837

Appendix L – TA Report – J180-60852

Appendix M – Macroinvertebrate Analytical Results Summary Table

Appendix N – TA Report – J180-61461

Appendix O – Lumbriculus Variegatus Analytical Results Summary Table

Appendix P – TA Report – J180-62135

Appendix Q – TA Report – J180-60590

Appendix R – TA Report – J180-60831

Appendix S – TA Report – J180-61437

Abbreviations

AEM Group	Advanced Environmental Management Group, LLC
COC	Chain of Custody
EPA	Environmental Protection Agency
GLEC	Great Lakes Environmental Center, Inc.
MPCA	Minnesota Pollution Control Agency
MS	Matrix Spike
MSD	Matrix Spike Duplicate
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RFP	request for proposal
SOW	scope of work
USACE	U.S. Army Corps of Engineers

1.0 Introduction

The U.S. Army Corps of Engineers, Detroit District (USACE) contracted Advanced Environmental Management Group, LLC (AEM Group) to perform tissue analysis of organic samples collected from three reservoirs near Cloquet, Minnesota, in accordance with the USACE request for proposal (RFP) and statement of work (SOW) dated September, 2016.

The purpose of this project is part of an ongoing assessment to characterize the physical and chemical characteristics of fish and macro-organisms located in the Boulder Lake, Scanlon and Thomson Reservoirs for the purpose of evaluating human and wildlife exposure due to the current conditions of the reservoirs. The samples were provided by the Minnesota Pollution Control Agency (MPCA) and the Environmental Protection Agency (EPA).

For the purposes of aging the larger fish, the otoliths and fins were extracted from the fish prior to homogenization.

The SOW included the analysis of the selected fish and macro-organisms tissue provided by the MPCA and the EPA.

Laboratories subcontracted for analysis included:

- Test America (TA) for chemical and physical analyses, and
- Great Lakes Environmental Center, Inc. (GLEC) for biological handling and analyses.

This report provides documentation of the activities performed by GLEC and the laboratory results from Test America.

2.0 Scope of Work

The USACE SOW stated that MPCA and the EPA were to collect fish, mayflies and benthic organisms from three reservoirs near Cloquet, Minnesota for laboratory analysis.

The SOW estimated that MPCA and EPA would collect five fish species from each reservoir and three samples for each fish species collected. The fish samples were to consist of a total of: 5 fish species x 3 samples per reservoir x 3 reservoirs = 45 samples. Each fish sample would consist of a minimum of a 100 grams for analytical purposes.

The SOW estimated that MPCA and EPA would collect mayfly and other benthic organisms. The mayfly and benthic organism samples were to consist of a total of five samples per reservoir: 5 samples per reservoir x 3 reservoirs = 15 samples provided by MPCA and 15 additional samples provided by EPA.

The SOW estimated a total of 75 tissue samples be collected for chemical analysis. The samples were to be shipped under chain-of-custody (COC) with field logs to GLEC. GLEC would confirm the sample information collected in the field and preform the required homogenization. Each sample would be homogenized individually, including all fish in the same package. GLEC would send the samples to Test America, for analysis of dioxin, total mercury, methyl mercury, and % lipids.

For the fish samples, the EPA required otolith extraction from the larger fish species prior to homogenization. MPCA selected which fish would have the otolith extraction and included this information on the chain of custody form and field logs provided to GLEC. The SOW estimated that 100 fish would require otolith extraction. The extracted otoliths and a representative homogenated tissue samples of each fish sample would be preserved/frozen and sent to the EPA.

The samples being provided for chemical analysis for each reservoir are as follows:

- 5 Mayfly samples
- 5 Benthos samples
- 5 fish species samples will be selected from the following species:
    - Minnow or Shiner
    - Yellow Perch
    - Young of Year Bluegill
    - Smallmouth Bass
    - Walleye
    - Northern Pike

The actual number of samples processed and analyzed would be dependent on the number of fish and macro-organisms collected and the total weight of the fish and macro-organisms.

3.0 Sample Collection

3.1 Fish Sampling

Fish samples were collected by MPCA. No report has been provided to AEM Group documenting how the fish were collected, handled, measured, selected, processed, frozen, or shipped. All information provided in this section is based upon the field logs and whole fish chain-of-custody (COC) provided to GLEC with the fish samples. MPCA developed a standardized fish naming convention for the samples collected. The field logs record included:

- Sample ID including reservoir and fish species,
- Sample date and time,
- Fish species,
- Number of fish,
- Fish length in millimeters (mm),
- Fish weight in grams (g).

3.1.1 Scanlon Reservoir

Twelve fish samples were collected from the Scanlon Reservoir on October 6, 2016. The samples were sent to GLEC on October 26, 2016. Fish collected included:

- Northern Pike, 1 sample –2 fish
- Shiner Mix, 1 sample – number of individual fish not recorded, just total weight
- Smallmouth Bass, 3 samples – 3 fish, 3 fish, 3 fish
- Walleye, 1 sample – 3 fish
- White Sucker, 3 samples - 3 fish, 3 fish, 3 fish
- Yellow Perch, 3 samples – 4 fish, 3 fish, 19 fish

Appendix A includes copies of the Fish Sampling Field Log Sheets and the chain-of-custodies sent to GLEC for Scanlon Reservoir.

3.1.2 Thomson Reservoir

Thirteen fish samples were collected from the Thomson Reservoir on October 11, 2016. The samples were sent to GLEC on October 26, 2016. Fish collected included:

- Northern Pike, 1 sample – 3 fish
- Rock Bass, 2 sample – 3 fish, 8 fish
- Smallmouth Bass, 4 samples – 10 fish, 3 fish, 3 fish, 10 fish
- Walleye, 1 sample – 3 fish
- White Sucker, 3 samples - 3 fish, 3 fish, 3 fish
- Yellow Perch, 2 samples – 3 fish, 4 fish

Appendix B includes copies of the Fish Sampling Field Log Sheets and the chain-of-custodies sent to GLEC for Thomson Reservoir.

3.1.3 Boulder Lake Reservoir

Fourteen fish samples were collected from Boulder Lake Reservoir, the background samples, on October 6, 2016. The fish samples were sent to GLEC on October 26, 2016. Fish collected included:

- Black Crappie (species not confirmed), 1 sample – 6 fish
- Rock Bass, 1 sample - 9 fish
- Shiner Mix, 3 samples – number of individual fish not recorded, just total weight
- Walleye, 3 samples – 3 fish, 5 fish, 3 fish
- White Sucker, 3 samples – 3 fish, 3 fish, 3 fish
- Yellow Perch, 3 samples – 13 fish, 13 fish, 12 fish

Appendix C includes copies of the Fish Sampling Field Log Sheets and the chain-of-custodies sent to GLEC for Boulder Lake Reservoir.

3.2 Fish Handling and Homogenization

GLEC received a total of 39 fish samples out of the estimated 45 samples listed in the USACE SOW. GLEC logged the fish samples into their tracking system and issued the samples a GLEC sample ID number. MPCA also identified the samples to be used for Test America Quality Assurance/Quality Control (QA/QC) matrix spike/matrix spike duplicate (MS/MSD) samples and those samples to be duplicated by GLEC for QA/QC duplicate analysis.

GLEC prepared the fish samples based on the instructions on the COCs using the procedures outlined by EPA in EPA-841-R-14-007: National Coastal Condition Assessment, 2015 Field Operations Manual and EPA 841-R-14-008, National Coastal Condition Assessment, 2015 Laboratory Operations Manual. A copy of the GLEC report is included in Appendix D.

The information on the fish length, weight, and species were confirmed by GLEC in the laboratory, once the fish were partially thawed. This information is included in Appendix D, Tables 1 and 2. These tables are also included in Appendix E.

Once the fish samples were measured and weighed, the selected fish had the otolith bones and fins removed and sent to the EPA for fish aging. Fish that could have the sex determined were investigated to determine if the fish was male or female. The fish samples were then homogenized; samples were placed into three jars and sent to Test America Laboratories in Canton, Ohio; Pittsburg, Pennsylvania; and Knoxville, Tennessee for chemical and physical analysis. Samples were analyzed for methyl mercury, total mercury, % lipids, and dioxins/furans.

3.3 Macroinvertebrates Sample Collection

Macroinvertebrates samples were collected by MPCA and by the EPA. No report has been provided to AEM Group documenting how the macroinvertebrates samples were collected, handled, selected, processed, frozen, or shipped.

Copies of the COCs for the macroinvertebrates samples are included in Appendix F. Limited instructions on the homogenization of samples and the chemical and physical analysis of the

samples were included on the COCs. Additional instructions were provided by the EPA on how to composite and homogenize the EPA collected samples. These instructions are included in Appendix F.

3.4 Macroinvertebrates Handling and Homogenization

GLEC received seven samples from MPCA. Based on the sample ID, all seven samples were collected from the Scanlon Reservoir from September 19, 2017 to October 6, 2017. The samples included:

- 3 mayfly samples
- 3 dragonfly samples
- 1 crawfish sample

The third mayfly sample was run as a QA/QC duplicate for total mercury and methyl mercury. The first dragonfly sample was run as a QA/QC duplicate for % lipids and dioxins/furans. Not all samples were analyzed for all compounds based on the total weight of macroinvertebrates. The priority selected was dioxin and % lipids, then methyl mercury and total mercury.

GLEC received 19 samples from the USEPA for compositing and homogenization. The 19 samples were composited into 4 samples for analysis. The four samples were:

- EPA16-SR-HD-001-MCRS – Scanlon Reservoir composite sample
- EPA16-TR-HD-001-MCRS – Thomson Reservoir composite sample
- EPA16-BR-HD-001-MCRS – Boulder Reservoir composite sample
- EPA16-TR-HD-001-C - Thomson Reservoir composite sample for crawfish

No field identifications of the macroinvertebrates in the EPA samples were provided to AEM Group for inclusion in this report.

Only the Boulder Lake sample had enough material to be analyzed for dioxin, % lipids, methyl mercury, and total mercury. The Scanlon and Thomson samples were analyzed for methyl mercury, and total mercury

Copies of the COC and compositing instructions to GLEC are included in Appendix F.

Based on the amount of sample available, the samples were placed into one jar and shipped to Test America in Pittsburg, Pennsylvania for analysis based on the amount of material available.

3.5 *Lumbriculus variegatus* Handling and Homogenization

As part of this contract, it was decided since limited mass of macroinvertebrates were collected in September and October of 2016, that sediment from the reservoirs would be collected and used to test for biological toxicity in *Lumbriculus variegatus* grown and harvested in the lab.

Sediment was sent to GLEC under a separate contract for this purpose. The analysis of the *Lumbriculus variegatus* was performed under this contract. A copy of this report was not provided to AEM Group for this report.

According to the information that was provided, GLEC was sent seven sediment samples:

- Boulder Lake Reservoir – 1 sample
- Scanlon Reservoir – 2 samples
- Thomson Reservoir – 4 samples

GLEC following standard procedures for the growth of *Lumbriculus variegatus,* divided the sediment samples into five replicates to produce enough *Lumbriculus variegatus* for laboratory analysis and statistical analysis of the laboratory results. However, based on the amount of *Lumbriculus variegatus* tissue, it was decided to homogenize the five replicates into one sample for laboratory analysis by Test America. No sample was divided for QA/QC analysis.

According to the COC, the *Lumbriculus variegatus* samples were homogenized on December 22, 2016 and shipped to Test America on December 26, 2016.

The *Lumbriculus variegatus* tissue samples were placed into three jars and sent to Test America Laboratories in Canton, Ohio; Pittsburg, Pennsylvania; and Knoxville, Tennessee for chemical and physical analysis. Samples were analyzed for methyl mercury, total mercury, % lipids, and dioxins/furans.

4.0 Tissue Sample Analysis

The tissue samples were delivered under chain-of-custody to Test America Laboratories in Canton, Ohio; Pittsburg, Pennsylvania; and Knoxville, Tennessee for chemical and physical analysis. Samples were analyzed for methyl mercury, total mercury, % lipids, and dioxins/furans.

- Canton, Ohio – methyl mercury
- Pittsburg, Pennsylvania – total mercury and % lipids
- Knoxville, Tennessee – dioxins/furans

All coordination between the three labs was managed out of the Pittsburg laboratory.

4.1 Fish Samples

4.1.1 Scanlon Reservoir

Thirteen fish samples were sent from GLEC to Test America and were analyzed in two laboratory batches.

- 180-60837 – 9 samples (includes 1 QA/QC duplicate sample)
- 180-60852 – 4 samples

A summary table of the results is included in Appendix G.

4.1.2 Thompson Reservoir

Fifteen fish samples were sent from GLEC to Test America and were analyzed in two laboratory batches.

- 180-60593 – 3 samples
- 180-60837 – 12 samples (includes 2 QA/QC duplicate sample)

A summary table of the results is included in Appendix H.

4.1.3 Boulder Lake Reservoir

Fifteen fish samples were sent from GLEC to Test America and were analyzed in one laboratory batch.

- 180-60593 – 15 samples (includes 1 QA/QC duplicate sample)

A summary table of the results is included in Appendix I.

4.1.4 Test America Laboratory Reports

Copies of the Test America Laboratory Reports are included in Appendices J, K, and L.

- Appendix J – J180-60593 – 1 report

- Appendix K – J180-60837 – 2 reports
- Appendix L – J180-60852 – 2 reports

The number of reports was based on the amount of time required to perform the dioxin/furan analyses and get the information into the Test America reporting database. The dioxin/furan reports were originally run using the World Health Organization (WHO) 2005 Toxic Equivalency Factors (TEF) for human health risks to calculate the Toxic Equivalence (TEQ) for the total dioxin/furans identified in the laboratory reports. These values were calculated using zero (0) as the concentration in the equation for all samples that had no detection levels for the analyte.

Upon review, the client requested the TEQs be recalculated using the WHO 1998 TEF for fish. These values were calculated using the equipment detection limit (EDL) as the concentration in the equation for all samples that had no detection levels for the analyte.

4.2 Macroinvertebrate Samples

Thirteen macroinvertebrate samples were sent from GLEC to Test America and were analyzed in one laboratory batch.

- 180-61461 – 13 samples (includes 2 QA/QC duplicate samples)

A summary table of the results is included in Appendix M.

Copies of the Test America Laboratory Reports are included in Appendix N. Two reports were issued for these samples, one for dioxin/furans, and one for % lipids, methyl mercury, and total mercury. The dioxin/furan report was reissued using the WHO 1998 TEF for fish and the EDL for non-detects.

4.3 *Lumbriculus variegatus* Samples

Eight *Lumbriculus variegatus* samples were sent from GLEC to Test America and were analyzed in one laboratory batch.

- 180-62135 – 8 samples (includes no QA/QC duplicate samples)

A summary table of the results is included in Appendix O.

Copies of the Test America Laboratory Reports are included in Appendix P. Two reports were issued for these samples, one for dioxin/furans, and one for % lipids, methyl mercury, and total mercury. The dioxin/furan report was reissued using the WHO 1998 TEF for fish and the EDL for non-detects.

4.4 GLEC QA/QC Samples

As part of the homogenization process, GLEC sent equipment rinsate blanks to Test America for analysis to document the cleaning decontamination process that occurred between the fish homogenization activities.

The activities are described in Appendix D on page 2, and the page below.

Table 1: GLEC Rinsate Blanks

Date	Tissue Type	GLEC Sample Number	Project Sample Designation
11-2-16	Fish	H2O Rinsate collected for MeHG following GLEC 5041	MN16 BR WAL-A
11-2-16	Fish	H2O Rinsate collected for total Hg following GLEC 5031	MN16 BR YP-A
11-3-16	Fish	Hexane Rinse collected for dioxin following GLEC 5045	MN16 BR GS-C
11-8-16	Fish	H2O Rinsate collected for MeHG following GLEC 5036	MN16 TR SMB-B
11-8-16	Fish	Hexane Rinse collected for dioxin following GLEC 5004	MN16 TR SMB-C
11-9-16	Fish	H2O Rinsate collected for total Hg following GLEC 5015	MN16 TR WS-B
11-10-16	Fish	Hexane Rinse collected for dioxin following GLEC 5016	MN16 SR WS-C
11-11-16	Fish	H2O Rinsate collected for total Hg following GLEC 5022	MN16 SR WS-A
11-14-16	Fish	H2O Rinsate collected for MeHG following GLEC 5020	MN16 SR YP-B
11-29-16	Macroinvertebrates	H2O Rinsate collected for total Hg following EPA-HD-TR-001-C	EPA HD TR 001-C
11-30-16	Macroinvertebrates	H2O Rinsate collected for MeHG following BW16 SR 003 D	BW16 SR 003 D

Copies of these reports are located in Appendices Q, R, and S.

- Appendix Q - 180-60590
- Appendix R - 180-60831
- Appendix S - 180-61437

APPENDIX A

Scanlon Reservoir Whole Fish COCs and Fish Sampling Field Logs

Cooler # 1
Scanlon 16-A

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-SR-WS-C (Label # 5016)

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	452	X	
.02		410	X	
.03		425	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-SR-NP-A (Label # 5019)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Northern Pike	340	X	Duplicate
.02		487	X	
.03			○	
.04			○	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-SR-GSH-A

	Common Name	Total Length (mm)	Frozen	Comments
.01	Golden Shiner - mixed shiner		X	bulk sample 61 grams
.02	5024		○	
.03			○	
.04			○	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-SR-WAL-A

	Common Name	Total Length (mm)	Frozen	Comments
.01	Walleye	305	X	
.02		290	X	
.03	5023	279	X	
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: Y
Print Name & Organization: Mark Elliott/MPCA Greg Peterson EPA	Time: 16:00	Print Name & Organization: John Bachman	Time: 10:19
Signature: Mark Elliott Greg Peterson	Date: 10-25-16	Signature: John Bachman	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Reservoir **Sample Date:** 10/6/16 **Time:** 15:43 **Recorded by:** CSM

Weather: ~~Sunny~~, 55°C dusk **Comments:**

Fish Species: WAL walleye **Composite Sample Group ID:** A **Group Sample ID#:** ~~5023.~~ MN16-SR-WAL-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	305	237	WAL	Y	Y	5023.1
A2	290	215	WAL	Y	Y	5023.2
A3	279	168	WA	Y	Y	5023.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Reservoir **Sample Date:** 10/6/16 **Time:** 14:43 **Recorded by:** CSM

Weather: Sunny, 55°F **Comments:**

Fish Species: WS White Sucker **Composite Sample Group ID:** C **Group Sample ID#:** MN16-SR-WS-C

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
C1	452	791	WS	Y	Y	5016.1
C2	410	781	WS	Y	Y	5016.2
C3	425	817	WS	Y	Y	5016.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon **Sample Date:** 10/6/16 **Time:** 17:43 **Recorded by:** CSM

Weather: Sunny, 55°C **Comments:**

Fish Species: NP Northern Pike **Composite Sample Group ID:** A **Group Sample ID#:** ~~5019~~ MN16-SR-NP-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	417	340	NP	X	Y	5019.1
A2	462	487	NP	X	Y	5019.2

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Reservoir **Sample Date:** 10/6/16 **Time:** 17:41 **Recorded by:** GSH

Weather: clear, sunny, 55°C **Comments:** many individuals n=17

Fish Species: GSH **Composite Sample Group ID:** A **Group Sample ID#:** ~~5024~~ MN16-SR-GSH-A

Golden Shiner – Shiner Mix

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
17 ~~NA~~	NA	61g	Shiner			5024.1 – many individuals

Notes:

WHOLE FISH CHAIN OF CUSTODY Scanlon Cooler #2

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Scanlon Reservoir			Date Collected: 10 / 6 / 16	
SAMPLE ID: MN16-SR-SMB-A (Label # 5001)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	~~544~~ 344	X	
.02		~~627~~ 341	X	
.03		340	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir			Date Collected: 10 / 6 / 16	
SAMPLE ID: MN16-SR-SMB-B (Label # 5002)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	326	X	
.02		345	X	Duplicate
.03		272	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir			Date Collected: 10 / 6 / 16	
SAMPLE ID: MN16-SR-SMB-C (Label # 5021)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	251	X	
.02		295	X	
.03		265	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir			Date Collected: 10 / 6 / 16	
SAMPLE ID: MN16-SR-WS-A (Label # 5022)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	439	X	
.02		399	X	
.03		399	X	
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: Y
Print Name & Organization: Mark Elliott/MPCA Greg Peterson ECA	Time: 16:00	Print Name & Organization: John Bachman	Time: 11:00 am
Signature: [signature] [signature]	Date: 10-25-16	Signature: [signature]	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Res. **Sample Date:** 10/6/16 **Time:** 14:57 **Recorded by:** CSM

Weather: Sunny, 55°C **Comments:**

Fish Species: SMB Smallmouth Bass **Composite Sample Group ID:** A **Group Sample ID#:** ~~5001~~ MN16-SR-SMB-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	344	547	SMB	Y	Y	5001.1
A2	341	627	SMB	Y	Y	5001.2
A3	340	547	SMB	Y	Y	5001.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR **Sample Date:** 10/6/16 **Time:** 15:10 **Recorded by:** CSM

Scanlon Reservoir

Weather: Sunny, 55°C **Comments:**

Fish Species: SMB **Composite Sample Group ID:** B **Group Sample ID#:** ~~5002~~

MN16-SR-SMB-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
B1	326	473	SMB	Y	Y	5002.1
B2	345	587	SMB	Y	Y	5002.2
B3	272	278	SMB	Y	Y	~~5002~~. 5002.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR **Sample Date:** 10/6/16 **Time:** 17:26 **Recorded by:** CSM

Scanlon Reservoir

Weather: Sunny, 55°C **Comments:**

Fish Species: SMB **Composite Sample Group ID:** C **Group Sample ID#:** ~~5021~~

Smallmouth Bass MN16-SR-SMB-C

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
C1	251	213	SMB	Y	Y	5021.1
C2	295	371	SMB	Y	Y	5021.2
C3	265	274	SMB	Y	Y	5021.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR **Sample Date:** 10/6/16 **Time:** 14:11 **Recorded by:** CSM

Scanlon Reservoir

Weather: Sunny, 55°F **Comments:**

Fish Species: WS **Composite Sample Group ID:** A **Group Sample ID#:** MN16-SR-WS-A

White Sucker

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	439	1015	WS	Y	Y	5022.1
A2	399	736	WS	Y	Y	5022.2
A3	399	792	WS	Y	Y	5022.3

Notes:

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in cooler)

Sender: Mark Elliott / MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+SR+WS-B (label # 5017)

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	421	X	
.02		452	X	
.03		304	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+SR+YP-A (label # 5025)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch A	239	X	
.02		226	X	MS/MSD
.03		217	X	
.04		185	X	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+SR+YP-B (label # 5020)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch	202	X	
.02		169	X	
.03		223	X	
.04			○	
.05			○	

Site ID: Scanlon Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+SR+YP-C (label # 5018)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch	~~45~~	○	Length not recorded
.02		Not recorded	○	
.03			○	Weight = 432 grams
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: Y
Print Name & Organization: Mark Elliott/MPCA, Greg Peterson EPA	Time: 16:00	Print Name & Organization: John Bachman	Time: 10:30a
Signature:	Date: 10/25/16	Signature:	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Reservoir **Sample Date:** 10/6/16 **Time:** 14:32 **Recorded by:** CSM

Weather: Sunny, 55°F **Comments:**

Fish Species: WS white sucker **Composite Sample Group ID:** B **Group Sample ID#:** MN16-SR-WS-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
B1	421	844	WS	Y	Y	5017.1
B2	452	952	WS	Y	Y	5017.2
B3	304	324	WS	Y	Y	5017.3

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Res. **Sample Date:** 10/6/16 **Time:** 13:17 **Recorded by:** CSM

Weather: clear, sunny, 55°F **Comments:**

Fish Species: YP Yellow Perch **Composite Sample Group ID:** A **Group Sample ID#:** MN16-SR-YP-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	239	166	YP	Y	Y	5025.1
A2	226	136	YP	Y	Y	5025.2
A3	217	124	YP	Y	Y	5025.3
A4	185	74	YP	Y	Y	5025.4

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon RES **Sample Date:** 10/6/16 **Time:** 13:33 **Recorded by:** CSM

Weather: clear, sunny, 55°F **Comments:**

Fish Species: YP Yellow Perch **Composite Sample Group ID:** B **Group Sample ID#:** MN16-SR-YP-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
B1	202	98	YP	Y	Y	5020.1
B2	189	76	YP	Y	Y	5020.2 (no 5020.3)
B3	223	141	YP	Y	Y	5020.4

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: SR Scanlon Res. **Sample Date:** 10/6/16 **Time:** 14:26 **Recorded by:** CSM

Weather: ~~Cot~~ Sunny, 55°F **Comments:** did not individually measure fish
19 individuals / 432g

Fish Species: YP Yellow Perch **Composite Sample Group ID:** C **Group Sample ID#:** ~~5018~~ MN16-SR-YPC

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
C		432	YP	Y	~~C~~ Y	5018.1

Notes:

APPENDIX B

Thomson Reservoir Whole Fish COCs and Fish Sampling Field Logs

Thomson - Cooler #1

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Thomson Reservoir			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16-TR-WS-B (label # 5015)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	425	X	
.02		420	X	
.03		448	X	
.04			O	
.05			O	

Site ID: Thomson Reservoir			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16+TR-WS-C (label # 5014)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	435	X	
.02		405	X	
.03		392	X	
.04			O	
.05			O	

Site ID: Thomson Reservoir			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16+TR-WAL-A (label # 5007)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Walleye	219	X	
.02		346	X	MS/MSD
.03		334	X	
.04			O	
.05			O	

Site ID: Thomson Reservoir			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16-TR-RB-A (label # 5009)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Rock Bass	135	X	
.02		145	X	
.03		190	X	
.04			O	
.05			O	

Released By / Organization		Received By / Organization	Received Frozen:
Print Name & Organization: Mark Elliott/MPCA Greg Peterson IERF	Time: 16:00	Print Name & Organization: John Bachman	Time: 16:45
Signature:	Date: 10-25-16	Signature:	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott — Date Sent: 10 / 26 / 16

Email Address: — Phone Number:

Note: Record information for minnows on back

Site ID: Thompson Reservoir — **Date Collected:** 10 / 11 / 16

SAMPLE ID: MN16 TR RB-B

	Common Name	Total Length (mm)	Frozen	Comments
.01	Rock Bass		○	8 individuals
.02			○	total = 150g
.03	5010		○	
.04			○	
.05	sample not on COC, entered info on receipt. JB 10/27/16		○	

Site ID: — **Date Collected:** / /

SAMPLE ID:

	Common Name	Total Length (mm)	Frozen	Comments
.01			○	
.02			○	
.03			○	
.04			○	
.05			○	

Site ID: — **Date Collected:** / /

SAMPLE ID:

	Common Name	Total Length (mm)	Frozen	Comments
.01			○	
.02			○	
.03			○	
.04			○	
.05			○	

Site ID: — **Date Collected:** / /

SAMPLE ID:

	Common Name	Total Length (mm)	Frozen	Comments
.01			○	
.02			○	
.03			○	
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: ☑
Print Name & Organization	Time	Print Name & Organization: John Bachman	Time 11:30
Signature	Date	Signature John Bachman	Date 10/27/16
Print Name & Organization	Time	Print Name & Organization	Time
Signature	Date	Signature	Date

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res **Sample Date:** 10/11/16 **Time:** 12:35 **Recorded by:** HB

Weather: Cloudy **Comments:** _____

Fish Species: WS White Sucker **Composite Sample Group ID:** B **Group Sample ID#:** 5015 MN16-TR-WS-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	425	965	WS	Yes	Yes	
2	420	820	↓	Y	Y	
3	448	923	↓	Y	Y	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/4/16 **Time:** 12:45 **Recorded by:** H Bauman

Weather: Cloudy **Comments:**

Fish Species: WS White Sucker **Composite Sample Group ID:** C **Group Sample ID#:** MN16-TR-WS-C 5014

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	435	1070	WS	Yes	Yes	
2	405	618	↓	↓	↓	
3	392	633	↓	↓	↓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Reservoir **Sample Date:** 10/11/16 **Time:** 15:00 **Recorded by:** H. Bauman

Weather: Cloudy **Comments:**

Fish Species: WAL Walleye **Composite Sample Group ID:** A **Group Sample ID#:** MN16-TR-WAL-A 5007

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	~~261~~ 319	261	WAL	Yes	Yes	
2	346	360	↓	↓	↓	
3	334	311	↓	↓	↓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/11/16 **Time:** 15:3 **Recorded by:** H. Bauman

Weather: Cloudy **Comments:** Small 1-3 yr Rock Bass Individual fish

Fish Species: Rock Bass **Composite Sample Group ID:** A **Group Sample ID#:** MN16-TR-RB-A 5009

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	135	50	RB	Y	Y	
2	145	58	RB	Y	Y	
3	190	142	RB	Y	Y	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Reservoir **Sample Date:** 10/4/16 **Time:** 15:30 **Recorded by:** H. Bauman

Weather: Cloudy **Comments:** Small 1-2 yr Rock Bass

Fish Species: Rock Bass **Composite Sample Group ID:** B **Group Sample ID#:** MNHo-TR-RB-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	90			NO	NO	
2	100					
3	100					
4	100					
5	100					
6	100					
7	105					
8	105					
		150		↓	↓	Total wt.

Notes:

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Thomson Reservoir			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16-SMB+A (label # 5003)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass		X	bulk sample w/
.02			X	10 small/juven. fish
.03			X	size fish
.04			○	130-164 mm
.05			○	

Site ID: Thomson Res			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16-TR-SMB-B (label # 5036)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	364	X	
.02		327	X	
.03		365	X	
.04			○	
.05			○	

Site ID: Thomson Res			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN 16+TR-SMB-C (label # 5004)				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	389	X	
.02		394	X	
.03		392	X	
.04			○	
.05			○	

Site ID: Thomson Res.			Date Collected: 10 / 11 / 16	
SAMPLE ID: MN16-TR+SMB-D (label # 5038				
	Common Name	Total Length (mm)	Frozen	Comments
.01	Smallmouth Bass	bulk sample	X	bulk sample
.02		total weight	○	103-170 mm
.03		358 gr	○	small juvenile
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: [ ]
Print Name & Organization: Mark Elliott/MPCA Greg Peterson EA	Time: 16:00	Print Name & Organization: John Bachman	Time: 10:30
Signature: Greg Peterson	Date: 10-25-16	Signature: John Bachman	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/11/16 **Time:** 13:05 **Recorded by:** H Bauman

Weather: Cloudy **Comments:** _____

Fish Species: ~~FB~~ SMB **Composite Sample Group ID:** ~~B~~ A **Group Sample ID#:** MN16-TR-~~FB~~ 5003 SMB-BA

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	143	39	~~FB~~ SMB	No	No	
2	139	36		↓	↓	
3	145	41				
4	143	41				
5	153	47				
6	130	30				
7	164	64				
8	142	37				
9	131	30				
10	130	29		✓	✓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR **Sample Date:** 10/11/16 **Time:** _____ **Recorded by:** H Bauman

Thomson Reservoir

Weather: Cloudy **Comments:** _____

Fish Species: SMB **Composite Sample Group ID:** B **Group Sample ID#:** MN16-TR-SMB-B 50360

Smallmouth Bass

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	364	763	SMB	Yes	Yes	
2	327	468		Y	Y	
3	365	714		Y	Y	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/11/16 **Time:** _____ **Recorded by:** H. Bauman

Weather: Cloudy **Comments:** _____

Fish Species: SMB Smallmouth Bass **Composite Sample Group ID:** C **Group Sample ID#:** MN16-TR-SMB-C 5004

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	389	1090	SMB	Yes	Yes	
2	389	1012	↓	↓	↓	
3	392	936	↓	↓	↓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/11/16 **Time:** _____ **Recorded by:** H. Bauman

Weather: cloudy **Comments:** _____

Fish Species: SMB Smallmouth Bass **Composite Sample Group ID:** D **Group Sample ID#:** MN16-TR-SMB-D

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	170			NO	NO	
2	160			↓	↓	
3	159					
4	155					
5	143					
6	139					
7	127					
8	112					
9	103					
		358				Total wt.

Notes:

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** ___/___/___

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Thomson Reservoir **Date Collected:** 10 / 11 / 16

SAMPLE ID: MN16-TR-NP-A (label # 5006)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Northern Pike	374	X	
.02		348	X	
.03		342	X	
.04			○	
.05			○	

Site ID: Thomson Res. **Date Collected:** 10 / 11 / 16

SAMPLE ID: MN16-TR-YP-A (label # 5005)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch	270	X	
.02		278	X	Duplicate
.03		256	X	
.04			○	
.05			○	

Site ID: Thomson Res. **Date Collected:** 10 / 11 / 16

SAMPLE ID: MN16-TR-YP-B (label # 5008)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch	238	X	
.02		239	X	
.03		231	X	
.04		230	X	
.05			○	

Site ID: Thomson RES **Date Collected:** 10 / 11 / 16

SAMPLE ID: MN16-TR-WS-A (label # 5011)

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	490	X	
.02		485	X	Duplicate
.03		480	X	
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen: [ ]
Print Name & Organization: Mark Elliott/MPCA Greg Peterson	Time: 1515	Print Name & Organization: John Bachman	Time: 11:00
Signature: Mark Elliott Greg Peterson	Date: 10/25/16	Signature: John Bachman	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson RES **Sample Date:** 10/11/16 **Time:** _____ **Recorded by:** H. Bauman

Weather: Cloudy **Comments:** Small 1 yr Pike

Fish Species: NP Northern Pike **Composite Sample Group ID:** A **Group Sample ID#:** MN16-TR-NP-A 5006

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	374	275	NP	Yes	Yes	
2	348	178	↓	↓	↓	
3	342	186	↓	↓	↓	

539g

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Reservoir **Sample Date:** 10/11/16 **Time:** 13:00 **Recorded by:** K Bauman

Weather: Cloudy **Comments:**

Fish Species: YP Yellow Perch **Composite Sample Group ID:** A **Group Sample ID#:** 5005 MN16-TR-YP-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	270	268	YP	Yes	Yes	
2	278	352	↓	↓	↓	
3	256	239	↓	↓	↓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/6/16 **Time:** 15:15 **Recorded by:** H. Bauman

Weather: Cloudy **Comments:**

Fish Species: YP Yellow Perch **Composite Sample Group ID:** B **Group Sample ID#:** MN16-TR-YP-B 5008

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	238	210	YP	YES	YES	
2	239	211	YP	↓	↓	
3	231	188	YP	↓	↓	
4	230	172	YP	↓	↓	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: TR Thomson Res. **Sample Date:** 10/11/16 **Time:** 12:25 **Recorded by:** H. Bauman

Weather: Cloudy **Comments:**

Fish Species: WS White Sucker **Composite Sample Group ID:** A **Group Sample ID#:** MN16-TR-WS-A 5011

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	490	1204	WS	Yes	Yes	
2	485	1184		Y	Y	
3	480	1064		Y	Y	

Notes:

APPENDIX C

Boulder Lake Reservoir Whole Fish COCs and Fish Sampling Field Logs

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott/MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+BR-RB-A (Lab # 5035)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Rock Bass		Y	9 individual fish
.02			Y	see attached log
.03			Y	YOY or small juvenile
.04			Y	
.05			Y	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+BLC-A (Label # 5044)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Black Crappie	107	Y	Small juvenile
.02		88	Y	or YOY 6
.03		102	Y	individual
.04		109	Y	
.05		99, 99	Y	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+YP-A (Label # 5031)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch		Y	13 individuals - See
.02			Y	log sheet (112-140 mm)
.03			Y	juvenile
.04			Y	
.05			Y	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+YP-B (Label # 5032)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch		N	13 individual fish
.02			N	see log sheet (110-185 mm)
.03			N	juvenile perch
.04			N	
.05			N	

Released By / Organization			Received By / Organization		Received Frozen: [N]
Print Name & Organization: Mark Elliott/MPCA Greg Peterson	Time: 16:00		Print Name & Organization: John Bachman	Time: 11:00am	
Signature:	Date: 10-25-16		Signature:	Date: 10/27/16	
Print Name & Organization:	Time:		Print Name & Organization:	Time:	
Signature:	Date:		Signature:	Date:	

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** 14:45 **Recorded by:** H Bauman

Weather: P Cloudy **Comments:**

Fish Species: RB Rock Bass **Composite Sample Group ID:** A **Group Sample ID#:** MN16-BR-RB-A 5035

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	102	22	RB	No	No	
2	114	27				
3	101	18				
4	105	22				
5	98	17				
6	101	19				
7	102	21				
8	121	35				
9	110	27		↓	↓	
~~10~~						
~~11~~						

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** 14:50 **Recorded by:** H Bauman

Weather: PCloudy **Comments:** BLC - Black Crappie ? check

Fish Species: BLC Black Crappie ? **Composite Sample Group ID:** A **Group Sample ID#:** MN16-BR-BLC-A 5044

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	107	23	BLC	No	No	
2	88	13				
3	102	20				
4	109	25				
5	99	17				
6	99	18		↓	↓	

Notes: Check Species

Black Crappie

or Rock Bass

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** ~~17:00~~ 12:30 **Recorded by:** H. Bauman

Weather: Sun, breezy **Comments:** Bulk 5031.1

Fish Species: YP Yellow Perch **Composite Sample Group ID:** A **Group Sample ID#:** MN16-BR-YP-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
~~1~~ 1	112	15	YP	No	No	
2	190	77				
3	180	63				
4	160	49				
5	124	15				
6	115	14				
7	115	15				
8	115	13				
9	120	16				
10	114	14				
11	173	64				
12	112	12				
13	115	11				

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** 12:45 **Recorded by:** H. Bauman

Weather: P. Cloudy **Comments:** Bulk

~~5031.5~~ 5030.1

Fish Species: YP Yellow Perch **Composite Sample Group ID:** B **Group Sample ID#:** MN16-BR-YP-B

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	160	46	YP	No	No	
2	110	12	"	"	"	
3	150	36				
4	115	11				
5	154	41				
6	105	11				
7	113	14				
8	121	18			✓	
9	185	68		✓		
10	111	13	✓			
11	106	12				
12	119	17				
13	106	12				

Notes:

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MCA **Date Sent:** 10 / ___ / ___

Email Address: ___ **Phone Number:** 248-302-6649

Note: Record information for minnows on back

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+YP-C (Label # 5034)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Yellow Perch		X	12 individual fish
.02				see attached log
.03				103-173 mm
.04				juvenile size
.05				

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+GSH+A (Label # 5033)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Golden Shiner / Shiner Mix		X	Bulk Sample 152 gr
.02				# minnows - 8 Golden
.03				Shiner mixed w/
.04				spot-tail + Common Shiner
.05				see log sheet attached

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR+GSH-B (Label # 5032)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Golden Shiner / Shiner mix			Bulk Sample (152 grams)
.02				
.03				mix of Golden Shiner +
.04				spot-tail & Common Shiner
.05				see log sheet

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR-GSH-C

	Common Name	Total Length (mm)	Frozen	Comments
.01	Golden Shiner / Shiner Mix			Bulk Sample (163 grams)
.02	5045			
.03				mix of Golden + Spot-tail
.04				Shiner
.05				see log sheet

Released By / Organization		Received By / Organization	Received Frozen: [ ]
Print Name & Organization:	Time:	Print Name & Organization: John Bachman	Time: 10:45
Signature:	Date:	Signature: John Bachman	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** 15:00 **Recorded by:** H Bauman

Weather: P Cloudy **Comments:** Bulk Sp ID

Fish Species: GSH (Golden Shiner / Mixed Shiner) **Composite Sample Group ID:** A **Group Sample ID#:** MN16-BR-GSH-A 5033

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
		152	GSH	No	No	

Notes: 10% spot tail/ shiners by weight

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR **Sample Date:** 10/6/16 **Time:** 15:05 **Recorded by:** HBauman

Boulder Reservoir

Weather: P Cloudy **Comments:** Bulk Sple

5032

Fish Species: GSH **Composite Sample Group ID:** B **Group Sample ID#:** MN16-BR-GSH-B

Golden Shiner (Shiner Mix)

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
		152	GSH	No	No	

Notes: 10% spot tail shiner by weight

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR **Sample Date:** 10/4/16 **Time:** 15:10 **Recorded by:** H Bauman

Boulder reservoir

Weather: P Cloudy **Comments:** Bulk Smpl

Fish Species: GSH **Composite Sample Group ID:** C **Group Sample ID#:** MN16-BR-GSH-C 5045

Golden Shiner / Shiner Mix

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
		163	GSH	No	No	

Notes: 10% spot tail Shiner by weight

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Reservoir **Sample Date:** 10/6/16 **Time:** 14:30 **Recorded by:** H Bauman

Weather: P Cloudy **Comments:** Bulk Smpl

Fish Species: YP Yellow Perch **Composite Sample Group ID:** C **Group Sample ID#:** MN16-BR-YP-C 5034

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
1	173	58	YP	No	No	
2	172	58				
3	157	45				
4	166	47				
5	103	10				
6	107	12				
7	106	12				
8	110	13				
9	105	12				
10	110	14				
11	103	11				
12	109	12				

Notes:

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** 10 / 26 / 16

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+BR+WAL-A (Lab # 5041)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Walleye	420	X	Duplicate
.02		396	X	
.03		396	X	
.04			○	
.05			○	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+BR+WAL-C (Lab # 5043)

	Common Name	Total Length (mm)	Frozen	Comments
.01	walleye	226	X	
.02		214	X	
.03		222	X	
.04		203	X	
.05		211	X	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16+BR+WS-B 5029

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	370	X	
.02		466	X	
.03		456	X	
.04			○	
.05			○	

Site ID: **Date Collected:** / /

SAMPLE ID:

	Common Name	Total Length (mm)	Frozen	Comments
.01			○	
.02			○	
.03			○	
.04			○	
.05			○	

Released By / Organization			Received By / Organization		Received Frozen: Y
Print Name & Organization: Mark Elliott MPCA Greg Peterson EPA	Time: 16:00		Print Name & Organization: John Bachman	Time: 11:00 am	
Signature:	Date: 10/25/16		Signature:	Date: 10/27/16	
Print Name & Organization:	Time:		Print Name & Organization:	Time:	
Signature:	Date:		Signature:	Date:	

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder res. **Sample Date:** 10/6/16 **Time:** 11:00 **Recorded by:** H. Bauman

Weather: Sun + Breeze **Comments:**

Fish Species: WAL walleye **Composite Sample Group ID:** A **Group Sample ID#:** MN16 - BR - WAL - A 5041

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	420	664	WAL	Yes	Yes	
A2	396	578	WAL	Yes	Yes	
A3	396	577	WAL	Yes	Yes	
A4			WAL	Yes	Yes	?
A5			WAL	Yes	Yes	?

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Res. **Sample Date:** 10/6/16 **Time:** 14:15 **Recorded by:** H Bauman

Weather: P Cloudy **Comments:** _____

Fish Species: WAL Walleye **Composite Sample Group ID:** C **Group Sample ID#:** 5043 MN16-BR-WAL-C

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
C1	206	58	WAL	Yes	Yes	
C2	214	84	WAL	Yes	Yes	
C3	222	83	WAL	Yes	Yes	
C4	203	72	WAL	Yes	Yes	
C5	211	73	WAL	Yes	Yes	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Res. **Sample Date:** 10/16/16 **Time:** 13:35 **Recorded by:** H. Bauman

Weather: Pcloudy **Comments:**

Fish Species: WS white sucker **Composite Sample Group ID:** B **Group Sample ID#:** MN16-BR-WS-B 5029

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
B1	370	622	WS	Yes	Yes	
B2	466	1230	WS	Yes	Yes	
B3	456	1150	WS	Yes	Yes	

Notes:

Boulder Cooler #1

WHOLE FISH CHAIN OF CUSTODY

(Complete for the samples to be included in one cooler)

Sender: Mark Elliott / MPCA **Date Sent:** ___/___/___

Email Address: mark.elliott@state.mn.us **Phone Number:** 218-302-6649

Note: Record information for minnows on back

Site ID: Boulder Lake Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR-WS-A (Lab # 5027)

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	279	X	MS/MSD
.02		438	X	
.03		365	X	
.04			○	
.05			○	

Site ID: Boulder Lake Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR-WS-C (Lab # 5028)

	Common Name	Total Length (mm)	Frozen	Comments
.01	White Sucker	528	X	
.02		470	X	
.03		482	X	
.04			○	
.05			○	

Site ID: Boulder Reservoir **Date Collected:** 10 / 6 / 16

SAMPLE ID: MN16-BR-WAL-B (Lab # 5042)

	Common Name	Total Length (mm)	Frozen	Comments
.01	Walleye	250	X	
.02		295	X	
.03		225	X	
.04			○	
.05			○	

Site ID: **Date Collected:** ___/___/___

SAMPLE ID:

	Common Name	Total Length (mm)	Frozen	Comments
.01			○	
.02			○	
.03			○	
.04			○	
.05			○	

Released By / Organization		Received By / Organization	Received Frozen:
Print Name & Organization: Mark Elliott/MPCA Greg Peterson EPA	Time: 16:00	Print Name & Organization: John Bachman	Time: 10:30
Signature: Mark Elliott Greg Peterson	Date: 10/24/16	Signature: John Bachman	Date: 10/27/16
Print Name & Organization:	Time:	Print Name & Organization:	Time:
Signature:	Date:	Signature:	Date:

Ship coolers to: GLEC
Attn: John Bachman
739 Hastings Street
Traverse City, MI 49686

GLEC
Great Lakes Environmental Center

Questions regarding sampling, packing, and shipping:
Call Jim Stricko (GLEC)
231-499-5947

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Res **Sample Date:** 10/6/16 **Time:** 11:00 **Recorded by:** H. Bauman

Weather: Sun Breezy **Comments:**

5027.1

Fish Species: WS white sucker **Composite Sample Group ID:** A **Group Sample ID#:** MN16-BR-WS-A

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
A1	279	221	WS	Yes	Yes	
A2	438	1007	WS	Yes	Yes	
A3	365	619	WS	Yes	Yes	
		1847				

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Res **Sample Date:** 10/6/16 **Time:** 1345 **Recorded by:** H. Bauman

Weather: PCloudy **Comments:** _____

Fish Species: WS White Sucker **Composite Sample Group ID:** C **Group Sample ID#:** MN16-BR-WS-C 5028

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
C1	528	1779	WS	Yes	Yes	
C2	470	1285	WS	Yes	Yes	
C3	482	1326	WS	Yes	Yes	

Notes:

Fish Sampling Field Log Sheet

Project: SLRAOC Reservoir whole fish sampling conducted by MPCA, October 2016

Location: BR Boulder Res. **Sample Date:** 10/6/16 **Time:** 14:10 **Recorded by:** H Bauman

Weather: P Cloudy **Comments:**

Fish Species: WAL walleye **Composite Sample Group ID:** B **Group Sample ID#:** MN16-BR-WAL-B 5042

Individual Fish #	Wet Field Length (mm)	Wet Field Weight (grams)	Species	Sex ID	Perform Otolith Extraction	Comments
B1	280	131	WAL	Yes	Yes	
B2	295	212	WAL	Yes	Yes	
B3	225	81 / 424	WAL	Yes	Yes	

Notes:

APPENDIX D

GLEC Fish and Macroinvertebrate Tissue Processing Report

Great Lakes Environmental Center
Southeast Michigan Office
31700 West Thirteen Mile Road, Suite 215
Farmington Hills, Michigan 48334
Phone: 248.538.0900
Fax: 248.538.0906

January 19, 2017

Mariah Hope
Advanced Environmental Management Group
44339 Plymouth Oaks Boulevard
Plymouth, Michigan 48170-2585
Phone: 734-354-9070

SUBJECT: **Fish and Macroinvertebrate Tissue Processing**
Scanlon, Thomson, and Boulder Reservoirs
Cloquet, Minnesota
USACE AEM Group Contract W911XK-16-D-0014
GLEC Project Number: 5148

Dear Ms. Hope:

Great Lakes Environmental Center, Inc. (GLEC) provided fish and macroinvertebrate tissue processing and fish otolith extraction services to Advance Environmental Management Group (AEM Group) and the U.S. Army Corps of Engineers in conjunction with the 2016 Tissue Analysis of Scanlon, Thomson, and Boulder Reservoirs located near Cloquet, Minnesota.

The purpose of this project is to characterize the tissue samples provided by the Minnesota Pollution Control Agency (MPCA) from three reservoirs located near Cloquet, Minnesota (Boulder Lake, Scanlon and Thomson Reservoirs). The tissue samples were used to evaluate human and wildlife exposure due to dioxin, total and methyl mercury within these three reservoirs.

Tissue Processing Procedures

GLEC prepared the 39 fish and 11 macroinvertebrate tissue samples using the procedures outlined by EPA in EPA-841-R-14-007: National Coastal Condition Assessment, 2015 Field Operations Manual and EPA 841-R-14-008, National Coastal Condition Assessment, 2015 Laboratory

Operations Manual. The whole body fish tissue procedure is described in the 2015 National Coastal Condition Assessment Laboratory Operations Manual (Appendix A); this document references the fish tissue preparation procedures that are located in the Quality Assurance Project Plan for Sample Preparation for the 2013-2014 National Rivers and Streams Assessment Fish Fillet Indicator (Appendix B).

In accordance with the 2015 National Coastal Condition Assessment Laboratory Operations Manual USEPA Laboratory, the following rinsate blanks were collected.

Date	Tissue Type	GLEC Sample Number	Project Sample Designation
11-2-16	Fish	H2O Rinsate collected for MeHG following GLEC 5041	MN16 BR WAL-A
11-2-16	Fish	H2O Rinsate collected for total Hg following GLEC 5031	MN16 BR YP-A
11-3-16	Fish	Hexane Rinse collected for dioxin following GLEC 5045	MN16 BR GS-C
11-8-16	Fish	H2O Rinsate collected for MeHG following GLEC 5036	MN16 TR SMB-B
11-8-16	Fish	Hexane Rinse collected for dioxin following GLEC 5004	MN16 TR SMB-C
11-9-16	Fish	H2O Rinsate collected for total Hg following GLEC 5015	MN16 TR WS-B
11-10-16	Fish	Hexane Rinse collected for dioxin following GLEC 5016	MN16 SR WS-C
11-11-16	Fish	H2O Rinsate collected for total Hg following GLEC 5022	MN16 SR WS-A
11-14-16	Fish	H2O Rinsate collected for MeHG following GLEC 5020	MN16 SR YP-B
11-29-16	Macroinvertebrates	H2O Rinsate collected for total Hg following EPA-HD-TR-001-C	EPA HD TR 001-C
11-30-16	Macroinvertebrates	H2O Rinsate collected for MeHG following BW16 SR 003 D	BW16 SR 003 D

In conjunction with the USEPA Operations Manuals, triplicate percent lipid testing was conducted on three of the 39 fish tissue samples to evaluate homogenization of the tissue, the percent lipids test results follow:

Mariah Hope, AEM Group **January 19, 2017**
2016 Tissue Analysis, Scanlon, Thomson, and Boulder Reservoirs

GLEC Sample Number	Reservoir	Description	# of Individuals	Field Weight 1 (g)	Field Weight 2 (g)	Field Weight 3 (g)	Tissue Mass (g)	% Lipid	Standard Deviation	Sample Mean	Relative Standard Deviation (%)
5007-1	Thomson	Walleye A	3	261	360	311	10.00	0.75	0.12	0.85	15
5007-2							10.21	0.85			
5007-3							11.93	0.98			
5021-1	Scanlon	SMB C	3	213	371	274	11.21	0.85	0.09	0.95	9.1
5021-2							8.90	0.99			
5021-3							11.49	1.00			
5028-1	Boulder	White Sucker C	3	1775	1285	1326	12.38	2.78	0.43	2.31	18
5028-2							10.86	1.96			
5028-3							11.54	2.17			

Review of the percent lipids data for each of the three tissue samples reveals that the relative standard deviation was less than 20 percent and meets the homogenization requirements of the USEPA Laboratory Operation Manual. Note that there was insufficient tissue mass to complete percent lipids testing on the macroinvertebrate samples.

Let us know if you have other questions or require additional information.

Sincerely,
GREAT LAKES ENVIRONMENTAL CENTER, INC.

John Bachman
Principal Research Scientist

John H. Barkach, CPG, CHMM
Senior Program Manager

Table 1. Fish Tissue Processing Field Data
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Reservoir	Description	# of Individuals	Field Weight 1	Field Weight 2	Field Weight 3	Field Weight 4	Field Weight 5	Total Mass (mg)	Perform Sex ID?	Perform Otolith?	SexID 1	SexID 2	SexID 3	SexID 4	SexID 5	Processed Tissue Shipped to Lab
5035		Boulder	Rock Bass A	9						368	No	No						11/7/2016
5044		Boulder	Black Crappie A	6						116	No	No						11/7/2016
5031		Boulder	Yellow Perch A	13						378	No	No						11/7/2016
5030		Boulder	Yellow Perch B	13						311	No	No						11/7/2016
5034		Boulder	Yellow Perch C	12						304	No	No						11/7/2016
5033		Boulder	Shiners A	Many						152	No	No						11/7/2016
5032		Boulder	Shiners B	Many						152	No	No						11/7/2016
5045		Boulder	Shiners C	Many						163	No	No						11/7/2016
5041	Dupe	Boulder	Walleye A	3	664	578	577			1819	1,2,3	1,2,3	M	M	M			11/7/2016
5043		Boulder	Walleye C	5	58	84	83	72	73	370	1,2,3,4,5	1,2,3,4,5	IND	IND	IND	IND	IND	11/7/2016
5029		Boulder	White Sucker B	3	622	1230	1150			3002	1,2,3	1,2,3	M	M	M			11/7/2016
5027	MSD	Boulder	White Sucker A	3	221	1007	619			1847	1,2,3	1,2,3	IND	M	M			11/7/2016
5028		Boulder	White Sucker C	3	1779	1285	1326			4390	1,2,3	1,2,3	F	F	F			11/7/2016
5042		Boulder	Walleye B	3	131	212	81			424	1,2,3	1,2,3	M	M	M			11/7/2016
5015		Thomson	White Sucker B	3	965	820	923			2708	1,2,3	1,2,3	F	F	F			11/14/2016
5014		Thomson	White Sucker C	3	1070	618	633			2321	1,2,3	1,2,3	F	F	F			11/14/2016
5007	MSD	Thomson	Walleye A	3	261	360	311			932	1,2,3	1,2,3	M	M	M			11/7/2016
5009		Thomson	Rock Bass A	3	50	58	142			250	1,2,3	1,2,3	M	F	M			11/14/2016
5010		Thomson	Rock Bass B	8						150	No	No						11/7/2016
5003		Thomson	Small Mouth Bass A	10						394	No	No						11/14/2016
5036		Thomson	Small Mouth Bass B	3	763	768	714			2245	1,2,3	1,2,3	F	M	F			11/14/2016
5004		Thomson	Small Mouth Bass C	3	1090	1012	936			3038	1,2,3	1,2,3	F	M	M			11/14/2016
5038		Thomson	Small Mouth Bass D	9						358	No	No						11/7/2016
5006		Thomson	North Pike A	3	275	178	186			639	1,2,3	1,2,3	F	F	F			11/14/2016
5005	Dupe	Thomson	Yellow Perch A	3	268	352	239			859	1,2,3	1,2,3	F	F	F			11/14/2016
5008		Thomson	Yellow Perch B	4	210	211	188	172		781	1,2,3,4	1,2,3,4	F	M	M	F		11/14/2016
5011	Dupe	Thomson	White Sucker A	3	1204	1144	1064			3412	1,2,3	1,2,3	F	F	F			11/14/2016
5016		Scanlon	White Sucker C	3	791	781	817			2389	1,2,3	1,2,3	F	F	F			11/14/2016
5019	Dupe	Scanlon	Northern Pike A	2	340	487				827	1,2	1,2	F	F				11/14/2016
5024		Scanlon	Shiners A	Many						61	No	No						11/15/2016
5023		Scanlon	Walleye A	3	237	215	168			620	1,2,3	1,2,3	M	M	M			11/14/2016
5001		Scanlon	SMB A	3	547	627	547			1721	1,2,3	1,2,3	M	F	M			11/14/2016
5002	Dupe	Scanlon	SMB B	3	473	587	278			1338	1,2,3	1,2,3	F	M	F			11/15/2016
5021		Scanlon	SMB C	3	213	371	274			858	1,2,3	1,2,3	F	F	M			11/14/2016
5022		Scanlon	White Sucker A	3	1015	736	792			2543	1,2,3	1,2,3	F	F	F			11/14/2016
5017		Scanlon	White Sucker B	3	844	952	324			2120	1,2,3	1,2,3	F	F	M			11/14/2016
5025	MSD	Scanlon	Yellow Perch A	4	166	136	124	74		500	1,2,3,4	1,2,3,4	F	F	F	M		11/15/2016
5020		Scanlon	Yellow Perch B	3	98	76	141			315	1,2,3	1,2,3	M	M	F			11/15/2016
5018		Scanlon	Yellow Perch C	19						432	No	No						11/14/2016

Table 2. Fish Tissue Processing Laboratory Data
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Reservoir	Description	# of Individuals	Metric	1	2	3	4	5	SexID 1	SexID 2	SexID 3	SexID 4	SexID 5
5027	MSD	Boulder	White Sucker A	3	Length (mm)	272	399	347			IND	M	M		
5027	MSD	Boulder	White Sucker A	3	Mass (g)	221	1013	615			IND	M	M		
5028		Boulder	White Sucker C	3	Length (mm)	502	468	457			F	F	F		
5028		Boulder	White Sucker C	3	Mass (g)	1884	1368	1326			F	F	F		
5029		Boulder	White Sucker B	3	Length (mm)	351	464	455			M	M	M		
5029		Boulder	White Sucker B	3	Mass (g)	616	1232	1147			M	M	M		
5030		Boulder	Yellow Perch B	13	For greyed-out samples, see small species spreadsheet tab for individual mass and length										
5031		Boulder	Yellow Perch A	13											
5032		Boulder	Shiners B	Many											
5033		Boulder	Shiners A	Many											
5034		Boulder	Yellow Perch C	12											
5035		Boulder	Rock Bass A	9											
5041	Dupe	Boulder	Walleye A	3	Length (mm)	418	394	393			M	M	M		
5041	Dupe	Boulder	Walleye A	3	Mass (g)	671	599	591			M	M	M		
5042		Boulder	Walleye B	3	Length (mm)	248	288	219			M	M	M		
5042		Boulder	Walleye B	3	Mass (g)	127	213	79			M	M	M		
5043		Boulder	Walleye C	5	Length (mm)	204	213	220	200	211	IND	IND	IND	IND	IND
5043		Boulder	Walleye C	5	Mass (g)	56	83	82	72	73	IND	IND	IND	IND	IND
5044		Boulder	Black Crappie A	6											
5045		Boulder	Shiners C	Many											
5001		Scanlon	SMB A	3	Length (mm)	343	338	341			M	F	M		
5001		Scanlon	SMB A	3	Mass (g)	552	631	555			M	F	M		
5002	Dupe	Scanlon	SMB B	3	Length (mm)	321	345	272			F	M	F		
5002	Dupe	Scanlon	SMB B	3	Mass (g)	481	594	282			F	M	F		
5016		Scanlon	White Sucker C	3	Length (mm)	415	410	414			F	F	F		
5016		Scanlon	White Sucker C	3	Mass (g)	785	743	799			F	F	F		
5017		Scanlon	White Sucker B	3	Length (mm)	420	432	306			F	F	M		
5017		Scanlon	White Sucker B	3	Mass (g)	826	939	320			F	F	M		
5018		Scanlon	Yellow Perch C	19											
5019	Dupe	Scanlon	Northern Pike A	2	Length (mm)	415	459				F	F			
5019	Dupe	Scanlon	Northern Pike A	2	Mass (g)	343	481				F	F			
5020		Scanlon	Yellow Perch B	3	Length (mm)	201	186	219			M	M	F		
5020		Scanlon	Yellow Perch B	3	Mass (g)	100	87	142			M	M	F		
5021		Scanlon	SMB C	3	Length (mm)	250	289	265			F	F	M		
5021		Scanlon	SMB C	3	Mass (g)	218	379	279			F	F	M		
5022		Scanlon	White Sucker A	3	Length (mm)	436	389	395			F	F	F		
5022		Scanlon	White Sucker A	3	Mass (g)	1016	736	796			F	F	F		
5023		Scanlon	Walleye A	3	Length (mm)	307	290	276			M	M	M		

Table 2. Fish Tissue Processing Laboratory Data
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Reservoir	Description	# of Individuals	Metric	1	2	3	4	5	SexID 1	SexID 2	SexID 3	SexID 4	SexID 5
5023		Scanlon	Walleye A	3	Mass (g)	241	216	172			M	M	M		
5024		Scanlon	Shiners A	Many											
5025	MSD	Scanlon	Yellow Perch A	4	Length (mm)	232	219	214	187		F	F	F	M	
5025	MSD	Scanlon	Yellow Perch A	4	Mass (g)	170	137	125	75		F	F	F	M	
5003		Thomson	Small Mouth Bass A	10											
5004		Thomson	Small Mouth Bass C	3	Length (mm)	393	348	393			F	M	M		
5004		Thomson	Small Mouth Bass C	3	Mass (g)	1083	1001	924			F	M	M		
5005	Dupe	Thomson	Yellow Perch A	3	Length (mm)	271	276	259			F	F	F		
5005	Dupe	Thomson	Yellow Perch A	3	Mass (g)	267	345	238			F	F	F		
5006		Thomson	North Pike A	3	Length (mm)	371	347	339			F	F	F		
5006		Thomson	North Pike A	3	Mass (g)	278	179	183			F	F	F		
5007	MSD	Thomson	Walleye A	3	Length (mm)	317	332	330			M	M	M		
5007	MSD	Thomson	Walleye A	3	Mass (g)	261	360	309			M	M	M		
5008		Thomson	Yellow Perch B	4	Length (mm)	216	215	228	230		F	M	M	F	
5008		Thomson	Yellow Perch B	4	Mass (g)	138	138	190	168		F	M	M	F	
5009		Thomson	Rock Bass A	3	Length (mm)	136	145	192			M	F	M		
5009		Thomson	Rock Bass A	3	Mass (g)	50	58	132			M	F	M		
5010		Thomson	Rock Bass B	8											
5011	Dupe	Thomson	White Sucker A	3	Length (mm)	468	468	480			F	F	F		
5011	Dupe	Thomson	White Sucker A	3	Mass (g)	1023	1108	1169			F	F	F		
5014		Thomson	White Sucker C	3	Length (mm)	431	404	392			F	F	F		
5014		Thomson	White Sucker C	3	Mass (g)	1030	591	620			F	F	F		
5015		Thomson	White Sucker B	3	Length (mm)	419	412	438			F	F	F		
5015		Thomson	White Sucker B	3	Mass (g)	949	794	892			F	F	F		
5036		Thomson	Small Mouth Bass B	3	Length (mm)	366	311	363			F	M	F		
5036		Thomson	Small Mouth Bass B	3	Mass (g)	1083	1001	924			F	M	F		
5038		Thomson	Small Mouth Bass D	9											

Table 2. Fish Tissue Processing Laboratory Data (continued)
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Reservoir	Species	Individual	Mass (g)	Length (mm)
5030	13	Boulder	Yellow Perch B	1	12	102
				2	46	162
				3	12	107
				4	35	143
				5	18	110
				6	11	96
				7	12	104
				8	11	100
				9	17	109
				10	12	99
				11	14	104
				12	41	147
				13	67	176
5031	13	Boulder	Yellow Perch A	1	77	181
				2	64	172
				3	48	156
				4	65	169
				5	16	109
				6	14	107
				7	15	111
				8	14	108
				9	15	106
				10	17	116
				11	15	111
				12	10	97
				13	12	103
5032	many	Boulder	Shiners B	Min	<1	39
				Max	12	110
5033	many	Boulder	Shiners A	Min	<1	18
				Max	24	125
5034	12	Boulder	Yellow Perch C	1	11	99
				2	12	103
				3	12	104
				4	13	105

Table 2. Fish Tissue Processing Laboratory Data (continued)
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Reservoir	Species	Individual	Mass (g)	Length (mm)
				5	14	108
				6	11	99
				7	13	103
				8	12	103
				9	45	154
				10	59	168
				11	47	146
				12	59	171
5035	9	Boulder	Rock Bass A	1	29	108
				2	36	117
				3	23	102
				4	20	96
				5	22	101
				6	28	110
				7	24	103
				8	18	94
				9	19	100
5044	6	Boulder	Black Crappie A	1	18	95
				2	14	86
				3	18	94
				4	21	99
				5	26	103
				6	25	104
5045	many	Boulder	Shiners C	Min	<1	9
				Max	32	101
5018	19	Scanlon	Yellow Perch C	1	132	216
				2	82	178
				3	43	153
				4	25	132
				5	20	118
				6	16	111
				7	15	109
				8	14	109
				9	18	116

Table 2. Fish Tissue Processing Laboratory Data (continued)
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Reservoir	Species	Individual	Mass (g)	Length (mm)
				10	14	103
				11	12	103
				12	12	103
				13	10	97
				14	3	68
				15	2	66
				16	3	65
				17	3	70
				18	3	70
				19	2	59
5024	many	Scanlon	Shiners A	Min	<1	15
				Max	33	120
5003	10	Thomson	Small Mouth Bass A	1	30	132
				2	30	135
				3	29	133
				4	35	142
				5	39	141
				6	41	138
				7	40	139
				8	47	151
				9	38	140
				10	64	161
5010	8	Thomson	Rock Bass B	1	20	97
				2	14	91
				3	19	97
				4	19	101
				5	23	107
				6	22	104
				7	18	95
				8	18	95
5038	9	Thomson	Small Mouth Bass D	1	57	156
				2	55	155
				3	63	169
				4	50	149

Table 2. Fish Tissue Processing Laboratory Data (continued)
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Reservoir	Species	Individual	Mass (g)	Length (mm)
				5	39	141
				6	35	133
				7	28	124
				8	19	110
				9	16	100

Table 3. **Macroinvertebrate Tissue Processing Data**
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnesota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

Sample Description	Date Processed	Sample ID	Homogenate Mass (g)	Notes
Boulder Reservoir, HD collection, macro invertebrates	11/30/2016	EPA16-HD-BR-001-MCRS	17	
Scanlon Reservoir, field collected crayfish, 005	11/30/2016	BW16-SR-005-C	35	
Scanlon Reservoir, field collected dragon fly nymphs, 002	11/29/2016	BW16-SR-002-D/ BW16-SR-102-D	40/12	Homogenate split into 2 samples (002D = 40g, 102D = 12g)
Scanlon Reservoir, field collected dragon fly nymphs, 003	11/30/2016	BW16-SR-003-D	16	
Scanlon Reservoir, field collected dragon fly nymphs, 005	11/30/2016	BW16-SR-005-D	36	
Scanlon Reservoir, field collected mayfly nymphs, 001	11/29/2016	BW16-SR-001-M	40	
Scanlon Reservoir, field collected mayfly nymphs, 002	11/29/2016	BW16-SR-002-M	52	
Scanlon Reservoir, field collected mayfly nymphs, 003	11/29/2016	BW16-SR-003-M/ BW16-SR-103-M	38/10	Homogenate split into 2 samples (003M = 38g, 103M = 10g)
Scanlon Reservoir, HD collected macro invertebrates	12/1/2016	BW16-HS-SR-001-MCRS	1.5	
Thomson Reservoir, HD collected macro invertebrates	11/29/2016	EPA16-HD-TR-001-MCRS	4	
Thomson Reservoir, HD collected crayfish	11/29/2016	EPA16-HD-TR-001-C	9	

APPENDIX A

2015 National Coastal Condition Assessment Laboratory Operations Manual

United States Environmental Protection Agency
Office of Water
Washington, DC
EPA 841-R-14-008

National Coastal Condition Assessment 2015 Laboratory Operations Manual

Version 2.1 May 2016

NOTICE

The goal of the National Coastal Condition Assessment (NCCA) is to provide a comprehensive assessment of the Nation's freshwater, marine shoreline and estuarine waters. The complete documentation of overall project management, design, methods, and standards is contained in four companion documents, including:

National Coastal Condition Assessment: Quality Assurance Project Plan EPA 841-R-14-005
National Coastal Condition Assessment: Site Evaluation Guidelines EPA 841-R-14-006
National Coastal Condition Assessment: Field Operations Manual EPA 841-R-14-007
National Coastal Condition Assessment: Laboratory Methods Manual EPA 841-R-14-008

This document (*Laboratory Operations Manual*) contains information on laboratory methods for analyses of the samples collected during the National Coastal Condition Assessment (NCCA). It also provides quality assurance objectives, sample handling procedures, and data reporting requirements. Methods described in this document are to be used specifically in work relating to the NCCA 2015. All NCCA Cooperator laboratories must follow the guidelines presented in the document.

With the exception of the requirements in Chapter 4 for evaluating algal toxics, mention of trade names or commercial products in this document does not constitute endorsement or recommendation for use. Chapter 4 requires use of a specific kit and supplemental materials manufactured by a single firm.

More details on specific methods for site evaluation, sampling, and sample processing can be found in the appropriate companion document.

The suggested citation for this document is:

USEPA. National Coastal Condition Assessment 2015: Laboratory Operations Manual. EPA-841-R-14-008. U.S. Environmental Protection Agency, Office of Water, Washington, DC. 2016.

Contents

NOTICE

LIST OF ACRONYMS

ADT	analysis decision tree
AFDM	ash-free dry mass
ANC	acid neutralizing capacity
ANS	Academy of Natural Sciences
AQM	absolute quantitation method
ASTM	American Society for Testing and Materials
Avg	Average
BHI	brain heart infusion
BV	biovolume
Ca	Calcium
CAS	Chemical Abstracts Service assigns unique identifiers to chemicals
CCE	calibrator cell equivalents
CEQ	cell equivalent
Chl-*a*	chlorophyll-a
Cl	Chloride
CO_2	carbon dioxide
Ct	threshold cycle
CPR	cardiopulmonary resuscitation
cv	curriculum vitae
DCF	dilution/concentration factor
DDT	dichloro-diphenyl-trichloroethane
DI	de-ionized
DIC	differential interference contrast
DL	detection limit
DNA	Deoxyribo-nucleic Acid
DO	dissolved oxygen
DOC	dissolved organic carbon
DTH	depositional targeted habitat
DW	distilled water
ELISA	enzyme-linked Immunosorbent assay
EMAP	Environmental Monitoring and Assessment Program
ENT	enterococci
EPA	Environmental Protection Agency
ETOH	ethyl alcohol
FOM	Field Operations Manual
g	grams
GEQ	genomic equivalent
GIS	geographic information system
GPS	global positioning device
HCl	hydrogen chloride
HDPE	high density polyethylene
HNO_3	nitric acid
HRP	antibody-Horseradish Peroxidase
H2S	hydrogen sulfide
$H2SO_4$	sulphuric acid
IBD	ionic balance difference

ID	Identification
IM	information management
IPC	internal positive control
ISBN	International Standard Book Number
ISO	International Organization for Standardization
IT IS	Integrated Taxonomic Information System (IT IS)
K	potassium
kg	kilograms
L	Liters
LCR	Labeled Compound Recovery
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blanks
LFM	Laboratory Fortified Matrices
LIMS	Laboratory Information Management System
LOM	Laboratory Operations Manual
LRL	Laboratory Reporting Limit
mg	milligrams
mg/kg	milligrams per kilogram
Mg	magnesium
mL	milliliters
MDL	method detection limit
Mn	manganese
MPCA	Minnesota Pollution Control Agency
MSDS	Materials Safety Data Sheet
N	nitrogen
Na	sodium
NABS	North American Benthological Society
NALMS	North American Lakes Management Society
NARS	National Aquatic Resource Surveys
NAWQA	National Water Quality Assessment Program
ND	non-detect
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
ng	nanograms
NH_4	ammonium
NIST	National Institute of Standards
NO_2	nitrite
NO_3	nitrate
NRSA	National Rivers and Streams Assessment
NTL	no template control
NTU	Nephelometric Turbidity Units
OD	optical density
ORD	EPA's Office of Research and Development
OSHA	Occupational Safety and Health Administration
OW	EPA's Office of Water
PAH	Polycyclic Aromatic hydrocarbons
PAR	Photosynthetically Active Radiation

PBS	phosphate buffered saline
PCB	polychlorinated biphenyl
PctDIFF	percent difference
PDE	percent disagreement in enumeration
PCR	polymerase chain reaction
PE	performance evaluation
PES	performance evaluation samples
PHab	physical habitat
P-M	Palmer-Maloney (P-M) count
PDE	percent difference in enumeration
ppb	parts per billion
ppm	parts per million
ppt	parts per trillion
PSE	percent sorting efficiency
PT	performance testing
PTD	percent taxonomic disagreement
QA	quality assurance
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QC	quality control
QCCS	Quality Control Check Sample
QMP	Quality Management Plan
qPCR	quantitative polymerase chain reaction
QRG	Quick Reference Guide
RL	reporting limit
RMSE	root mean square error
RO	reverse-osmosis
RPD	Relative Percent Difference
RQM	relative quantitation method
RSD	Relative Standard Deviation
RTH	richest targeted habitat
Sb	antimony
SEG	Site Evaluation Guidelines
SFS	Society of Freshwater Science
SiO_2	silica
SO_4	sulphate
SOPs	Standard Operating Procedures
SPC	sample processing control
S-R	Sedgewick-Rafter count
SRM	standard reference material
SS	salmon sperm
TMB	tetramethylbenzidine
TN	total nitrogen
TOC	total organic carbon
TP	total phosphorus
TRANS	transect

TSN	taxonomic serial number
TSS	total suspended solids
TVS	total volatile solids
µg	micrograms
µg/g	micrograms per gram
µg/L	micrograms per liter
UNK	unknown
USGS	United States Geological Survey
WSA	Wadeable Streams Assessment
WQX	Water Quality Exchange

1.0 INTRODUCTION

This manual describes methods for laboratory analyses of the samples to be collected during the National Coastal Condition Assessment (NCCA). The manual includes quality assurance objectives, sample handling specifications, and data reporting requirements.

The NCCA is one of a series of water assessments conducted by States, Tribes, the U.S. Environmental Protection Agency (EPA), and other partners. In addition to coastal waters, the National Aquatic Resource Surveys (NARS) also focuses on rivers and streams, lakes, and wetlands in a revolving sequence. The purpose of these assessments is to generate statistically-valid reports on the condition of our Nation's water resources and identify key stressors to these systems.

The goal of NCCA is to address two key questions about the quality of the Nation's coastal waters:

- What percent of the Nation's coastal waters are in good, fair, and poor condition for key indicators of water quality, ecological health, and recreation?
- What is the relative importance of key stressors such as nutrients and contaminated sediments?

The NCCA is a probability-based survey of our Nation's coastal and estuarine waters, and designed to:

- Assess the condition of the Nation's coastal and estuarine waters at national and regional scales, including the Great Lakes;
- Identify the relative importance of selected stressors to coastal and estuarine water quality;
- Evaluate changes in condition from previous National Coastal Assessments (NCA) starting in 2000; and
- Help build State and Tribal capacity for monitoring and assessment and promote collaboration across jurisdictional boundaries.

EPA selected the sampling locations using a probability based survey design. Sample surveys have been used in a variety of fields (e.g., monthly labor estimates, forest inventory analysis) to determine the status of populations or resources of interest using a representative sample of a relatively few members or sites. Using this survey design allows data from the subset of sampled sites to be applied to the larger target population, and assessments with known confidence bounds to be made.

The NCCA field sampling season will be during the index period of June through the end of September. Field crews will collect a variety of measurements and samples from the statistically selected sampling locations identified by geographical coordinates. The samples are shipped to laboratories to evaluate the indicators identified in Table 1.1. The indicators are similar to those evaluated in previous NCA.

Table 1.1 NCCA: Indicators

Measure/Indicator		Assessment outcome
Water Quality	Dissolved oxygen	Hypoxia/anoxia
	pH Temperature Depth Conductivity (freshwater) Salinity (marine)	Water column characterization
	Secchi/light measurements PAR	Societal value and ecosystem production
	Nutrients: • Dissolved inorganic NO_2 , NO_3 NH_4 ,PO_4; • Total N and P	Nutrient enrichment
	Chlorophyll *a*	
Sediment Quality	Grain size (Silt/Clay content)	Influencing factor for extent and severity for contamination
	Total Organic Carbon (TOC)	Influencing factor for extent and severity for contamination
	Sediment chemistry • 15 metals • 25 PAHs • 20 PCBs • 14 pesticides • 6 DDT metabolites	Risk of biological response to sediment contamination
	Sediment toxicity (10-day static bioassay with *Leptocheirus* or *Hyalella)*	Biological response to sediment exposure
Biological Quality	Whole body fish contaminants • 13 metals (no Sb or Mn) • 20 PCBs • 14 pesticides • 6 DDT metabolites • Optional: PAHs (national lab only)	Environmentally available contaminant exposure
	Benthic community structure	Biological response to site conditions

2.0 GENERAL LABORATORY GUIDELINES

This chapter describes the general laboratory guidelines with an overview to the quality assurance / quality control (QA/QC) requirements. Each of the following chapters describes a different procedure and the relevant QA/QC requirements for that particular procedure. In addition, the Quality Assurance Project Plan (QAPP) provides a comprehensive consolidation of the QA/QC requirements for NCCA 2015.

2.1 Responsibility and Personnel Qualifications

Each laboratory shall train its laboratory personnel in advance in the use of equipment and procedures used for the standard operating procedure (SOP) in which they are responsible. All personnel are responsible for complying with all of the QA/QC requirements that pertain to the samples to be analyzed. Each laboratory follows its institutional or organizational requirements for instrument maintenance. Appendix A identifies the specific documentation that each laboratory must submit to demonstrate its qualifications for performing the analyses.

2.2 Roles and Contact Information

The **EPA Headquarters Project Management Team** consists of the Project Leader, Alternate Project Leaders, Project QA Lead, and Laboratory Review Coordinator. The Team is responsible for overseeing all aspects of the project and ensuring that the laboratories properly adhere to the technical and quality assurance requirements. The Team is the final authority on all decisions regarding laboratory analysis.

The **NARS Information Management (IM) Coordinator** tracks the location of each NCCA sample that involves post-processing. The coordinator will be the labs main point of contact in regards to sample tracking and data submission.

Table 2.1 NCCA: Contact Information

Title*	Name	Contact Information
EPA HQ NCCA Project Lead, Acting	Hugh Sullivan, OW	**sullivan.hugh@epa.gov** 202-564-1763
EPA HQ NCCA Project QA Coordinator	Sarah Lehmann, OW	**lehmann.sarah@epa.gov** 202-566-1379
EPA HQ NCCA Laboratory Review Coordinator	Kendra Forde, OW	**forde.kendra@epa.gov** 202-564-0417
EPA HQ NARS Team Leader	Sarah Lehmann, OW	**lehmann.sarah@epa.gov** 202-566-1379
Information Management Center Coordinator	Marlys Cappaert, SRA International Inc.	**cappaert.marlys@epa.gov** 541-754-4467 541-754-4799 (fax)

*For any technical direction, laboratories under contract to EPA must contact the Task Order's Contracting Officer's Representative (TOCOR) instead of the contacts provided in this table. For any technical information or sample tracking, the laboratories are permitted to contact these persons.

2.3 Sample Tracking

Samples are collected by a large number of different field crews during the index period (May through September). The actual number of sites sampled on a given day will vary widely during this time. Field crews will submit electronic forms when they have shipped samples and the NARS IM Center will input each sample into the NARS IM database. Laboratories can track sample shipment from field crews by accessing the NARS IM database. Participating laboratories will be given access to the NARS IM system, where they can acquire tracking numbers and information on samples that have been shipped to them by field crews (either by overnight shipment for perishable samples or batch shipments for preserved samples). Upon sample receipt, the laboratory must immediately log in to the database and confirm that samples have arrived. Each laboratory will make arrangements with the NARS IM Coordinator, listed above, to ensure access is granted.

When the samples arrive from the field crews, the shipments will include tracking forms (refer to the NCCA FOM). These forms will list the samples included in the shipment. Laboratory personnel must cross check the forms with the samples received to verify that there are not any inconsistencies. If any sample is missing or damaged, contact the NARS IM Coordinator immediately.

2.4 Reporting

All labs must provide data analysis information to the HQ Project Management Team and the NARS IM Center by **March 30, 2016** or as stipulated in contractual agreements. These reports must include the data elements specified for each analytical method in this manual. The submitted filename must use the following naming convention:

- Indicator name (ex: microcystins)
- Date of files submission to NARS IM Center by year, month, and day (ex: 2015_11_01)
- Laboratory name (ex: MyLab)

Combined, the file name would look as follows: Microcystins_2015_11_01_MyLab.xlsx
Before the laboratory submits the batch data to EPA, the analyst who generated the data and an experienced data reviewer independently check and review the data, as follows:

The analyst shall review the data to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate method and standard operating procedures were followed;
- Analytical results are correct and complete;
- Quality control samples were within established control limits;
- Blanks (where appropriate) were within the appropriate QC limits; and
- Documentation is complete.

The data reviewer shall review the data package to verify that:

- Calibration data (where appropriate) are scientifically sound and appropriate;
- QC samples were within established control limits;
- Qualitative and quantitative results are correct; and
- Documentation is complete.

Accompanying its data submission for each batch, the laboratory shall provide a short narrative that includes the following information:

- Project summary referencing the batch QC identification number, total number of samples in the batch and their sample numbers, and the analytical methodology used for analysis;
- Discussion of any protocol deviations that may have occurred during sample testing;
- Discussion of QC questions or issues that were encountered and the corrective measures taken;
- Definitions of any laboratory QC codes used in the data;
- Summary and discussion of samples that are diluted by the presence of an interference, non-target analyte, or target analyte; and
- QC samples exceeding established control limits or parameters required by laboratory internal analytical SOPs and an explanation of why, if known.

As specified in the QAPP, remaining sample material and specimens must be maintained by the EPA's designated laboratory or facilities as directed by the NCCA 2015 Project Lead. Unless otherwise authorized by the Project Lead, the laboratory shall retain:

- The sample materials, including vials, for a minimum of three (3) years from the date the EPA publishes the 2015 NCCA report. During this time, the laboratory shall maintain the materials at the temperature specified in its laboratory method. The laboratory shall periodically check the sample materials for degradation. Unless the Project Lead arranges for transfer of sample materials to EPA, at the end of the retention period, the laboratory shall follow its internal protocols for disposal.
- Original records, including laboratory notebooks and raw data files (including logbooks, bench sheets, and instrument tracings), for a minimum of ten (10) years from the date that EPA publishes the final report.

The Project Lead is responsible for maintaining the following:

- Deliverables from contractors and cooperators, including raw data, which are permanent as per EPA Record Schedule 258.
- EPA's project records which under Schedule 501 are permanent.

3.0 ALGAL TOXIN (MICROCYSTIN) IMMUNOASSAY PROCEDURE

This chapter describes an immunoassay procedure that measures concentrations of total microcystins in water samples. In applying the procedure, the laboratory uses Abraxis' Microcystins-ADDA Test Kits (Figure 3.1; "kits"). Each kit is an enzyme-linked immunosorbent assay (ELISA) for the determination of microcystins and nodularins in water samples. Microcystins refers to the entire group of toxins, all of the different congeners, rather than just one congener. Algae can produce one or many different congeners at any one time, including Microcystin-LR (used in the kit's calibration standards), Microcystin-LA, and Microcystin-RR. The different letters on the end signify the chemical structure (each one is slightly different), which makes each congener different.

Figure 3.1 Microcystins: Abraxis Test Kit
(Converted from color to grayscale from James, page 3, 2010)

3.1 Summary of the Procedure

The procedure is an adaptation of the instructions provided by Abraxis for determining total microcystins concentrations using its ELISA-ADDA kits.[1] For samples samples with salinity<3.5 parts per thousand (ppt), the procedure's reporting range is 0.15 µg/L to 5.0 µg/L, although, theoretically, the procedure can detect, not quantify, microcystins concentrations as

1 Abraxis, "Microcystins-ADDA ELISA (Microtiter Plate): User's Guide R021412." Retrieved on January 14, 2014 from http://www.abraxiskits.com/uploads/products/docfiles/278_Microcystin%20PL%20ADDA%20users%20R120214.pdf.

low as 0.10 µg/L. For samples with higher concentrations of microcystins, the procedure includes the necessary dilution steps. The procedure also provides additional sample preparation steps for samples with salinities≥3.5 ppt. The results then are adjusted by a factor of 1.75 for a reporting range of 0.263 µg/L to 8.75 µg/L.

3.2 Health and Safety Warnings

The laboratory must require its staff to abide by appropriate health and safety precautions, because the kit substrate solution contains tetramethylbenzidine (TMB) and the stop solution contains diluted sulfuric acid. In addition to the laboratory's usual requirements such as a Chemical Hygiene Plan, the laboratory must adhere to the following health and safety procedures:

1. Laboratory facilities must properly store and dispose of solutions of weak acid.

2. Laboratory personnel must wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear, gloves).

3. When working with potential hazardous chemicals (e.g., weak acid), laboratory personnel must avoid inhalation, skin contact, eye contact, or ingestion. Laboratory personnel must avoid contacting skin and mucous membranes with the TMB and stopping solution. If skin contact occurs, remove clothing immediately. Wash and rinse the affected skin areas thoroughly with large amounts of water.

3.3 Definitions and Required Resources (Personnel, Laboratories, and Equipment)

This section provides definitions and required resources for using the procedure.

3.3.1 Definitions

The procedure uses the following terms:

Absorbance (A) is a measure of the amount of light absorbed by a sample at a specific wavelength. A standard statistical curve is used to convert the absorbance value to the concentration value of microcystins.

Brackish and Seawater Samples, for the purposes of the ABRAXIS microcystins test procedure, are samples with salinity greater than or equal to 3.5 parts per thousand (ppt). (EPA is using different definitions for the water chemistry samples.) EPA recognizes that brackish water is usually defined as 0.5 ppt, and seawater as 35 ppt, but for this immunoassay procedure, it is important to use additional steps described in Section 3.5.2 for any sample with salinity greater than or equal to 3.5 ppt. The sample labels provide the salinity levels.

Calibration Range is the assay range for which analysis results can be reported with confidence. For example, assays of undiluted samples with salinities<3.5 ppt range from the reporting limit of 0.15 µg/L to a maximum value of 5.0 µg/L.

Coefficient of Variation (CV): The precision for a sample is reported in terms of the percent CV of its absorbance values. To calculate the %CV, first calculate the standard deviation, *S*, as follows:

$$S = \left[\frac{1}{n-1}\sum_{i=1}^{n}(A_i - \bar{A})^2\right]^{1/2}$$

where *n* is the number of replicate samples, A_i, is the absorbance measured for the i^{th} replicate. Per Section 3.5.4, samples are evaluated in duplicate (i=1 or 2); controls are either evaluated in duplicate or triplicate (i=1, 2, 3). $\bar{A}$ is the average absorbance of the replicates. Then, calculate %CV as:

$$\%CV = \left|\frac{S}{\bar{A}}\right| \times 100$$

Dark or Dimly Lit: Away from sunlight, but under incandescent lighting is acceptable.

Detection Limit is the minimum concentration at which the analyte can be *detected* with confidence. In other words, the outcome can be reported with confidence that it is greater than zero (i.e., present in the sample). The detection limit is less than the reporting limit at which the *measured* value of the analyte can be reported with confidence. Also see "Sample-Specific Detection Limit."

Duplicates are defined as two aliquots of the same sample which are analyzed separately using identical procedures. The results are used to evaluate the precision of the laboratory analyses. Per Section 3.5.4, controls are evaluated in duplicate or triplicate (i.e., three aliquots).

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management System (NARS IM): The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. Freshwater and coastal samples will be collected during the field stage of NCCA.

Relative Standard Deviation (RSD) is the same as the coefficient of variation (%CV). Because many of the plate reader software programs provides the CV in their outputs, the procedure presents the quality control requirement in terms of %CV instead of RSD.

Reporting Limit: A reporting limit is the point at which the measured value of the analyte can be reported with confidence.

Sample-Specific Detection Limit: Most samples will have a sample-specific detection equal to the method's detection limi. For diluted samples, the sample-specific detection limit will be the product of the method's detection limit and the dilution factor. Typical values for the dilution factor will be 10 or 100.

Seawater Sample: See definition for brackish and seawater samples.

3.3.2 General Requirements for Laboratories

Expertise. To demonstrate its expertise, the laboratory shall provide EPA with one or more of the following:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
- Documentation detailing the expertise of the organization, including professional certifications for water-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.

Quality assurance and quality control requirements.
To demonstrate its expertise in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).
To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

3.3.3 Personnel

The procedure refers to the following personnel:

Laboratory Technician: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual (which differs from the Abraxis instructions). The laboratory technician also must be familiar with the use of a multichannel pipette and plate readers.

External QC Coordinator is an EPA staff person who is responsible for selecting and managing the "**QC contractor**." To eliminate the appearance of any inherent bias, the QC contractor must be dedicated to QA/QC functions, and thus, must not be a primary laboratory or a field sampling contractor for NCCA. The QC contractor is responsible for complying with instructions from the External QC Coordinator; coordinating and paying for shipments of the performance samples to participating laboratories; comparing immunoassay results from the laboratories; and preparing brief summary reports.

3.3.4 Equipment/Materials

The procedures require the following equipment and information:

- Abraxis ADDA Test Kit, Product #520011 (see items in Section 3.5.2)
- Adhesive Sealing Film (Parafilm) for Micro Plates (such as Rainin, non-sterile, Cat. No. 96-SP-100): Used to cover plates during incubation.
- Data Template – See Figure 3.2
- Distilled or Deionized Water: For diluting samples when necessary.
- ELISA evaluation software
- Glass scintillation, LC, vials (two vials of 20 mL each)
- Glass vials with Teflon-lined caps of size:
  - 20 mL
  - 4 mL (for dilutions)
- Multichannel Pipette & Plastic Tips: A single-channel and an 8-channel pipette are used for this method.
- Norm-ject syringes (or equivalent)
- Paper Towels: For blotting the microtiter plates dry after washing.
- Permanent Marker (Sharpie Fine Point): For labeling samples, bottles, plates and covers.
- Plate Reader (e.g., Metertech Model M965 AccuReader; ChroMate®; or equivalent readers with software to read the microtiter plates and measure absorbances).
- Reagent Reservoirs (e.g., Costar Cat Number 4870): Plain plastic reservoir for reagents that accommodate the use of a multi-channel pipette.
- Test tubes (glass): For dilutions, if needed.
- Timer: For measuring incubation times.
- Vortex Genie: For mixing dilutions.
- Whatman Glass fiber syringe filter (25mm, GF 0.45 µm filter)

Analysis of samples with salinity≥3.5 ppt require additional equipment and supplies, as follows:

- Microcystins-ADDA Seawater Sample Clean-Up Kit (Product #529912) which includes the following supplies:
  - Disposable 5 ¾" glass Pasteur pipettes
  - Disposable 9" glass Pasteur pipettes
  - Glass wool
  - Pasteur pipette bulb
  - Microcystins-ADDA Seawater Sample Treatment Solution
  - Microcystins-ADDA Seawater Sample Clean-up Resin
- 12x75 mm test tubes
- Scoopula
- Micropipettes with disposable plastic tips
- Vortex mixer

3.4 Sample Receipt

Field crews hold the microcystins samples on ice while in the field and then pack the samples in ice for delivery to a central facility ("batching laboratory") or the State's laboratory. The batching and State laboratories freeze the samples upon receipt. Periodically, the batching laboratory ships samples to the microcystins laboratory. The batching and microcystins laboratory may retain the frozen samples for several months before analysis.

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery.

1. Report receipt of samples in the NARS IM sample tracking system (within 24 clock hours). Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Inspect each sample THE SAME DAY THEY ARE RECEIVED:
   a. Verify that the sample IDs in the shipment match those recorded on the:
      i. Chain of custody forms when the batching laboratory sends the samples to the microcystins laboratory; or
      ii. Sample tracking form if the field crew sends the shipment directly to the State laboratory.
   b. Record the information in Table 3.1 into NARS IM, including the Condition Code for each sample:
      i. *OK*: Sample is in good condition
      ii. *C*: Sample container was cracked
      iii. *L*: Sample container is leaking
      iv. *ML*: Sample label is missing
      v. *W*: Sample is warm (>8^{o}), record the temperature in the comment field, and perform the assay
   c. If any sample is damaged or missing, contact the EPA HQ Laboratory Review Coordinator to discuss whether the sample can be analyzed. (See contact information in Chapter 2 of the Manual).

3. Store samples in the freezer until sample preparation begins.

4. Maintain the chain of custody or sample tracking forms with the samples.

Table 3.1 Microcystins Login: Required Data Elements

FIELD	FORMAT	DESCRIPTION
LAB ID	text	Name or abbreviation for QC laboratory
DATE RECEIVED	MMDDYY	Date sample was received by lab
SITE ID	text	NCCA site id as used on sample label
VISIT NUMBER	numeric	Sequential visits to site (1 or 2)

FIELD	FORMAT	DESCRIPTION	
SAMPLE ID	numeric	Sample id as used on field sheet (on sample label)	
DATE COLLECTED	MMDDYY	Date sample was collected	
CONDITION CODE	text	Condition codes describing the condition of the sample upon arrival at the laboratory.	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		W	Sample is warm (>8°)
		Q	Other quality concerns, not identified above
CONDITION COMMENT	text	Comments about the condition of the sample. If the condition code='W' then provide the temperature	

3.5 Procedure

The following sections describe the sample and kit preparation and analysis.

3.5.1 Sample Preparation: Freeze-Thaw Steps

For each frozen sample (500 mL per sample), the laboratory technician runs it through a freeze-thaw cycle three times to lyse the cells as follows:

1. All cycles: Keep the samples in dark or dimly lit areas (i.e., away from sunlight, but under incandescent lighting is acceptable).

2. First freeze-thaw cycle:
    a. Start with a frozen 500 ml sample.
    b. Thaw the sample to room temperature (approximately 25° C). Swirl the sample to check for ice crystals. At this temperature, no ice crystals should be present in the sample.
    c. Shake well to homogenize the sample, then transfer 10 mL to an appropriately labeled clean 20 mL glass vial.

3. Second freeze-thaw cycle:
    a. Freeze the vial.
    b. Keep the large sample bottle (from the 500 mL initial sample) frozen for future use.
    c. Thaw the sample vial contents to room temperature.

4. Third freeze-thaw cycle:
    a. Freeze the vial.
    b. Thaw the vial contents to room temperature.
    c. Filter the vial contents through a new, syringe filter (0.45 µm) into a new, labeled 20 mL glass scintillation vial. Norm-ject syringes and Whatman Glass fiber

syringe filters (25mm, GF 0.45 µm filter) or other similar alternative are acceptable. Use one new syringe and filter per sample.

3.5.2 Additional Sample Preparation for Samples with Salinity>3.5 parts per thousand

For any sample with salinity of 3.5 parts per thousand (ppt) or greater (the salinity will be marked on sample vials), the laboratory technician needs to perform the following additional steps provided by Abraxis. [2] For all other samples (i.e. with salinity less than 3.5 ppt), the technician skips this section (i.e., Section 3.5.2) and goes directly to kit preparation as described in Section 3.5.3. For samples with salinity 3.5 ppt the technician:

1. Prepares the column as follows:
    a. Place a small amount of glass wool into the top of a 5 ¾" glass Pasteur pipette. Using a 9" glass Pasteur pipette, push the glass wool into to the bottom of the 5 ¾" pipette to form the base of the column. The depth of the glass wool should be approximately 5 mm. Place the column into a 12x75 mm test tube.
    b. Each column will require approximately 1.5 g of Seawater Sample Clean-Up Resin. Calculate and add the appropriate amount of Microcystins-ADDA Seawater Sample Clean-Up Resin to a 20 mL glass vial.
    c. Add distilled or deionized water at an approximately 2:1 ratio to the Microcystins-ADDA Seawater Sample Clean-Up Resin (for example, 10 mL of deionized or distilled water per 5 g of Resin). Shake or vortex.
    d. Pipette the Resin in water solution into the column using the 9" Pasteur pipette. Avoid the formation of air bubbles in the column bed by keeping the tip of the pipette at the surface of the bed being created. Fill the column to the indentation approximately 2 cm from the top of the pipette. This will create an approximately 8 cm column.
    e. Allow the deionized or distilled water to drain from the column. [3]. Lift the tip of the column at least 1 cm above the surface of the water in the tube. Place the pipette bulb against the top of the column (do not attach the bulb to the column) and push the remaining water out of the column. Avoid allowing the tip of the column to come into contact with the water in the tube to prevent aspiration of water back into the column.
    f. Place the column into an appropriately labeled 4 mL glass vial.

2. Cleans up the sample as follows:
    a. Add 1 mL of the sample to a clean, appropriately labeled 4 mL glass vial. Add 50 µL of Microcystins-ADDA Seawater Sample Treatment Solution. Vortex.

2 Reformatted from Abraxis, "Microcystins in Brackish Water or Seawater Sample Preparation" Retrieved on January 14, 2014 from http://abraxiskits.com/uploads/products/docfiles/385_MCT-ADDA%20in%20Seawater%20Sample%20Prep%20%20Bulletin%20R041112.pdf. Reproduced with permission. Except for Abraxis' solutions labeled as seawater, EPA has removed references to "brackish" and "seawater" which typically are defined as having different cutpoints than 3.5 ppt for salinity.

3 Additional correspondence between EPA and Abraxis notes that this step leaves the resin in the column.

b. Add 375 µL of the treated sample to the top of the column. Allow the sample to drain through the column and collect in the vial.
c. Add a second 375 µL aliquot of the treated sample to the column. Allow to drain through the column.
d. Lift the tip of the column at least 1 cm above the surface of the sample in the vial. Place the pipette bulb against the top of the column (do not attach the bulb to the column) and push the remaining sample out of the column. Avoid allowing the tip of the column to come into contact with the sample in the vial to prevent aspiration of the sample back into the column.
e. Lower the column back into the vial. Add 500 µL of distilled or deionized water to the top of the column. Allow the rinse to drain through the column and collect with the sample.
f. Lift the tip of the column at least 1 cm above the surface of the sample/rinse in the vial. Place the pipette bulb against the top of the column (do not attach the bulb to the column) and push the remaining rinse out of the column. Avoid allowing the tip of the column to come into contact with the sample in the vial to prevent aspiration of the sample back into the column.
g. Remove the column and discard (columns are single use only). Cap vial and vortex. The sample can then be analyzed using the Abraxis Microcystins-ADDA ELISA Kit beginning with the next section (3.5.3).

3.5.3 Kit Preparation

The technician prepares the kits using the following instructions:

1. Check the expiration date on the kit box and verify that it has not expired. If the kit has expired, discard and select a kit that is still within its marked shelf life. (Instead of discarding the kit, consider clearly labelling it as expired and keeping it for training activities.)

2. Verify that each kit contains all of the required contents:
    - Microtiter plate
    - Standards (6) referenced in this procedure as follows with the associated concentration:
        - S0: 0 µg/L
        - S1: 0.15 µg/L
        - S2: 0.40 µg/L,
        - S3: 1.0 µg/L
        - S4: 2.0 µg/L
        - S5: 5.0 µg/L
    - Kit Control (KC): 0.75 µg/L
    - Antibody solution
    - Anti-Sheep-HRP Conjugate
    - Wash Solution 5X Concentrate
    - Color Solution
    - Stop Solution
    - Diluent
    - Foil bag with 12 microtiter plate strips

3. If any bottles are missing or damaged, discard the kit. This step is important because Abraxis has calibrated the standards and reagents separately for each kit.

4. Adjust the microtiter plate, samples, standards, and the reagents to room temperature.

5. Remove 12 microtiter plate strips (each for 8 wells) from the foil bag for each kit. The plates contain 12 strips of 8 wells. If running less than a whole plate, remove unneeded strips from the strip holder and place in the foil bag, ziplocked closed, and store in the refrigerator (4-8^{o} C).

6. Prepare a negative control (NC) using distilled water.

7. The standards, controls, antibody solution, enzyme conjugate, color solution, and stop solutions are ready to use and do not require any further dilutions.

8. Dilute the wash solution with deionized water. (The wash solution is a 5X concentrated solution.) In a 1L container, dilute the 5X solution 1:5 (i.e., 100 mL of the 5X wash solution plus 400 mL of deionized water). Mix thoroughly. Set aside the diluted solution to wash the microtiter wells later.

9. Handle the stop solution containing diluted H_2SO_4 with care.

3.5.4 Insertion of Contents into Wells

This section describes the steps for placing the different solutions into the 96 wells. Because of the potential for cross contamination using a shaker table, the following steps specify manual shaking of the kits instead mechanized shaking.

1. While preparing the samples and kit, turn the plate reader on so it can warm up. The plate reader needs a minimum of 30 minutes to warm up.

2. Turn on the computer so that it can control and access the plate reader.

3. Print the template (Figure 3.2) to use as reference when loading the standards, controls, and samples as described in the next step. Templates contain rows, labeled with a marking pen, of strips of 8 wells that snap into the blank frame. (If the laboratory wishes to use a different template, provide a copy to the EPA HQ Laboratory Review Coordinator for approval prior to first use. (See Chapter 2 of the manual for contact information.)

4. Using the 100-µL pipette, add 50 µL, each, of the standards, controls, and samples to the appropriate wells in the plate. Place all six standards (0.00, 0.15, 0.40, 1.00, 2.0 and 5.0 µg/L), the kit control (0.75 µL), and negative control, in pairs, starting in the well in the upper left-hand corner of the kit as shown in Figure 3.2. Verify that the software displays the same template or make any necessary corrections.

	1	2	3	4	5	6	7	8	9	10	11	12
A	S0	S4	NC	U4	U8	U12	U16	U20	U24	U28	U32	U36
B	S0	S4	NC	U4	U8	U12	U16	U20	U24	U28	U32	U36
C	S1	S5	U1	U5	U9	U13	U17	U21	U25	U29	U33	U37
D	S1	S5	U1	U5	U9	U13	U17	U21	U25	U29	U33	U37
E	S2	KC	U2	U6	U10	U14	U18	U22	U26	U30	U34	U38
F	S2	KC	U2	U6	U10	U14	U18	U22	U26	U30	U34	U38
G	S3	KC	U3	U7	U11	U15	U19	U23	U27	U31	U35	U39
H	S3	NC	U3	U7	U11	U15	U19	U23	U27	U31	U35	U39

Key:
S0-S5 = Standards;
KC = Control supplied with Kit (i.e., Kit Control);
NC = Negative Control;
U = Unknown (sample collected by the field crew);

Figure 3.2 Microcystins: Template for samples

5. Add 50 µL of the pink antibody solution to each well using the multi-channel pipettor and a reagent reservoir. Use dedicated reagent reservoirs for each reagent to avoid contamination from one reagent to another.

6. Place the sealing Parafilm over the wells.

7. Manually mix the contents by moving the strip holder in a rapid circular motion on the benchtop for 30 seconds. Be careful not to spill the contents.

8. Place the plate in a dimly lit area (as defined in Section 3.3.1) for 90 minutes.

9. After 90 minutes, carefully remove the Parafilm.

10. Empty the contents of the plate into the sink, pat inverted plate dry on a stack of paper towels, and then wash the wells of the plate three times with 250 µL of washing solution using the multi-channel pipette. After adding the washing solution each time, empty the solution into the sink and use the paper towels as before.

11. Add 100 µL of enzyme conjugate solution to all wells using the multi-channel pipettor.

12. Cover the wells with Parafilm.

13. Manually mix the contents by moving the strip holder in a rapid circular motion on the benchtop for 30 seconds. Be careful not to spill the contents.

14. Place the strip holder in a dimly lit area for 30 minutes.

15. After 30 minutes, remove the Parafilm, decant, and rinse the wells three times again with 250 µL of washing solution as described in step 10.

16. Add 100 µL of color solution to the wells using the multi-channel pipette and reagent reservoir. This color solution will make the contents have a blue hue.

17. Cover the wells with Parafilm.

18. Manually mix the contents by moving the strip holder in a rapid circular motion on the benchtop for 30 seconds. Be careful not to spill the contents.

19. Place the plate in a dimly lit area for 20 minutes.

20. After 20 minutes, remove the Parafilm and add 50 µL of stopping solution to the wells in the same sequence as for the color solution. This will turn the contents a bright yellow color. After adding the stopping solution, read the plate within 15 minutes.

21. Within 15 minutes of adding the stopping solution, use the microplate ELISA photometer (plate reader) to determine the absorbance at 450 nm. The software (i.e., commercial ELISA evaluation program) calculates the absorbance and concentration values of the samples from the calibration curve and the average values for each pair. Use a 4-parameter standard curve fit to determine the concentrations.

22. Dispose of solution in plates in a lab sink. Rinse plates and sink with water to dilute the weak acid present.

23. Perform QC evaluations of the data as follows:
    a. If the following **failures** occur, then the laboratory must reanalyze all samples in the analytical run:
        i. Standard curve with a correlation coefficient, R, of less than 0.99
        ii. Standards S0-S5 must have decreasing absorbance values. First, calculate the average values for each standard. That is, if $\bar{A}_i$ is the absorbance average for S_i, then the absorbance averages must be:
            $$\bar{A}_0 > \bar{A}_1 > \bar{A}_2 > \bar{A}_3 > \bar{A}_4 > \bar{A}_5$$
        iii. The average absorbance of the standard S0 less than 0.8 (i.e., $\bar{A}_0 < 0.8$).
        iv. Two or more negative control sample results report detectable concentrations of microcystins (i.e., values ≥ 0.1 µg/L). If this occurs, then evaluate possible causes (e.g., cross-contamination between samples), and if appropriate, modify laboratory processes before the next analytical run.
        v. Results for control samples of outside the acceptable range of 0.75 +/- 0.185 µg/L. That is, results must be between 0.565 µg/L and 0.935 µg/L.

b. If either, or both, of the following situations occur, then the sample must be reanalyzed (maximum of two analyses,[4] consisting of the original analysis and, if necessary, one reanalysis):
    i. The concentration value registers as HIGH (exceeds the calibration range).[5] Dilute the sample for the reanalysis per Section 3.5.5.
    ii. The %CV > 15% between the duplicate absorbance values for a sample.

24. If the sample has a salinity of 3.5 ppt or greater, then convert the results by multiplying by 1.75. If the assay was non-detected, then the detection limit is 0.175 µg/L. The reporting limit is 0.263 µg/L. The calibration range is 0.263 µg/L to 8.75 µg/L.

25. Record the results, even if the data failed the quality control requirements in #23b, for each well in EPA's data template (see Table 3.2 for required elements). The required entries are for the following columns:
    a. **TYPE** indicates the sample type using one of the following codes: S0-S5 for standards; KC or NC for controls; and U for unknown sample.
    b. **CONC** contains the numeric concentration value. Two special cases:
        i. Non-detected concentrations: If the sample is non-detected, then provide the sample-specific detection limit which is 0.1 µg/L if the sample is undiluted with a salinity<3.5 ppt in the sample. See step 24 for reporting values for samples with salinity ≥3.5 ppt. See Section 3.5.5 for calculating the sample-specific detection limit for a diluted sample.
        ii. If the result shows that it is "HI," this indicates that the sample value is outside of the calibration range and must be diluted and re-run using another analytical run. Leave the CONC column blank and record 'HI' in the DATA FLAG column.
    c. **DATA FLAGS** have codes for the following special cases:
        i. **ND** if the sample was non-detected;
        ii. **J** if the value is detected but at a level below the reporting limit of 0.15 µg/L (for undiluted samples with salinity <3.5 ppt; see step 24 for samples with salinity ≥3.5 ppt);
        iii. **HI** if the concentration value registers as HIGH (exceeds the calibration range).
    d. **QUALITY FLAGS** have codes for the following special cases:
        i. **QCF** if there is a QC failure per step 23 above. The QCF code must be used for all failures to facilitate data analysis.
        ii. **Q** for any other quality issue (describe in **COMMENTS**)
    e. DILUTION FACTOR is only required if the sample was diluted.

[4] In its data analyses, EPA compares the microcystins data values to 10 µg/L, which is the World Health Organization threshold for moderate risk. If a sample is diluted once following the procedures in Section 3.5.5 and the concentration still registers as HIGH, the concentration is recorded as >50 µg/L which is greater than the WHO threshold. EPA does not require additional dilution to obtain a more precise value, but a laboratory may choose to increase the dilution of the sample and report the associated concentration value.
[5] A value of HIGH is not a QA/QC failure, but rather indicates a necessity to find the correct dilution to get it within calibration.

f. **DUP AVG** and **DUP CV** are required for duplicate samples and control samples (use all three values if the controls are used in triplicate).

Table 3.2 Microcystins: Required Data Elements

STAGE	FIELD	FORMAT	DESCRIPTION	
LOGIN	LAB ID	Character	Name or abbreviation for QC laboratory	
	DATE RECEIVED	MMDDYY	Date sample was received by lab	
	SITE ID	Character	NCCA site ID code as recorded on sample label or tracking form (blank if standard or control)	
	VISIT NUMBER	Numeric	sequential visits to site (1 or 2) (blank if standard or control)	
	SAMPLE ID	Numeric	6-digit Sample ID number as recorded on sample jar or tracking form (blank if standard or control)	
	DATE COLLECTED	MMDDYY	Date sample was collected (blank if standard or control)	
	CONDITION CODE	Character	Sample condition upon arrival at the laboratory (blank if standard or control)	
			Flag	Definition
			Blank or N	Not a sample (blank, standard, or control)
			OK	Sample is in good condition
			C	Sample container is cracked
			L	Sample or container is leaking
			ML	Sample label is missing
			W	Sample iswarm (>8°)
	CONDITION COMMENT	Character	Comments about the condition of the sample. If the condition code='W' then provide the temperature	
ANALYSIS	TECHNICIAN	Character	Name or initials of technician performing the procedure	
	ANALYSIS DATE	MMDDYY	Date when samples are inserted into the wells per Section 3.5.4	
	ANALYSIS TIME	24-hour time	Time when 1st sample is inserted into the wells per Section 3.5.4	
	KIT EXPIRE DATE	MMDDYY	Expiration date on kit box	
	KIT ID	Character	Kit identification code. If one does not exist, assign a unique code to each kit.	
	R2	Numeric	R^2 from curve fit to the average absorbance values for the standards. Value is between 0 and 1.	
	TYPE	Character	Type of solution being tested in the well	
			Code	Definition
			KC	Kit Control
			NC	Negative Control
			S0,S1, S2,S3, S4, S5	Standard

STAGE	FIELD	FORMAT	DESCRIPTION	
			U	Sample of unknown concentration
	LOCATION	Character	Location of well in the kit (e.g., B5 would be the fifth well from the left in the second row B)	
	SALINITY	Numeric	If the sample vial has the salinity marked on the vial, record the value in units of parts per thousand. Otherwise, leave blank.	
	CONC	Numeric	Concentration or sample-specific detection limit of contents of well in µg/L. Sample-specific detection limit should be 0.1 µg/L for a sample with salinity <3.5 ppt which hasn't been diluted. (Detection limit is 0.175 µg/L for samples with salinity ≥3.5 ppt)	
	ABSORBANCE	Numeric	Absorbance value	
	DILUTION FACTOR	Numeric	10, 100, etc for number of times the sample was diluted. If not diluted, leave blank or record 1	
	CV_ABSORB	Numeric	Calculated %CV of duplicate values of absorbance for a sample. Only calculated for TYPE=U, KC, or NC. Enter %CV. Value is between 0 and 100%.	
	AVG_ABSORB	Numeric	Calculated average of absorbance values for a sample. Only provided for TYPE=U, KC, NC, or SC. Average value of the original sample and its duplicate (or replicates for KC and NC).	
	AVG_CONC	Numeric	Calculated average of concentration values for a sample. Substitute for any value below the reporting limit.	
	DATA FLAG (if appropriate)	Character	Data qualifier codes associated with specific identifications of voucher samples. These codes provide more information than those used when reporting receipt of samples. A technician may use alternative or additional qualifiers if definitions are provided as part of the submitted data package (e.g., as a separate worksheet page of the data submission file).	
			Flag	Definition
			ND	Concentration below detection.
			HI	Result indicated a high concentration (i.e., outside calibration range)
			J	Concentration above detection but below reporting limit.
	QUAL_FLAG	QCF/Q	QCF	QC failure
			Q	Other quality concerns, not identified above
	COMMENTS	Character	Explanation for data flag(s) (if needed) or other comments.	

3.5.5 Dilutions (if needed)

Dilutions if needed are prepared as follows (using clean glass tubes):

1. 1:10 dilution
    a. Add 900 µL of distilled water to a clean vial. (Note: Dilutions may also be made using the kit's diluent rather than distilled water.)
    b. Pipette 100 µL from the sample into the vial. (To provide more accurate dilutions and less chance of contaminating the diluent, add the diluent to the vial before the sample.)
    c. Mix by vortexing.
    d. Multiply final concentration and Abraxis' detection limit by 10 to obtain the sample-specific detection limit.. For example, for a sample with salinity<3.5 ppt, Abraxis' detection limit is 0.1 µg/L and the sample-specific detection would be 1.0 µg/L for a 1:10 dilution.

2. 1:100 dilution
    a. Add 3.96 mL of distilled water to a clean, appropriately labeled glass vial. (Note: Dilutions may also be made using the kit's diluent rather than distilled water.)
    b. Vortex the sample to mix thoroughly, then pipette 40 µL from the sample and add to the water (or diluent) in the appropriate labeled vial. Vortex.
    c. Multiply the final concentration and Abraxis' detection limit by 100 to obtain the sample-specific detection limit. For example, for a sample with salinity<3.5 ppt, Abraxis' detection limit is 0.1 µg/L and the sample-specific detection would be 10 µg/L for a 1:100 dilution.

3. Other dilutions can be calculated in the same manner as #1 and #2 if needed.

3.6 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements.

3.6.1 Assistance Visits

Assistance visits are intended to familiarize EPA with actual procedures being implemented by different laboratories; and to ensure a clear and consistent understanding of procedures and activities by both EPA and the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter. EPA will develop, review and approve the checklist prior to conducting an assistance visit.

3.6.2 QC Samples

The External QC Coordinator will instruct the QC contractor to provide one or two identical sets of freshwater and/or seawater performance test samples to all participating laboratories. If the laboratory will assay both freshwater and seawater samples, then it will receive both sets (i.e.,

freshwater and seawater). Each set will contain five samples to test the expected range of concentrations in the NCCA samples.

For the contract laboratory, the QC contractor will provide the first set to be run with the first set of samples and a second set to be run at the midpoint of the assigned samples. If available, a third set will be run with the final batch of samples. Because most state laboratories will have relatively few samples that can be analyzed using a single kit, the QC contractor will send only one set to each state laboratory.

Each laboratory will run the QC samples following the same procedures used for the other samples. The External QC Coordinator will compare the results and assess patterns in the data (e.g., one laboratory being consistently higher or lower than all others). Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any deviations from the Method or unique laboratory practices that might account for differences between the laboratory and others. With this additional information, the External QC Coordinator will determine an appropriate course of action, including no action, flagging the data, or excluding some or all of the laboratory's data.

3.6.3 Summary of QA/QC Requirements

Table 3.3 provides a summary of the quality control requirements described in Sections 3.5 and 3.6.

Table 3.3 Microcystins: Sample analysis quality control activities and objectives

Quality Control Activity	Description and Requirements	Corrective Action
Kit – Shelf Life	Is within its expiration date listed on kit box.	If kit has expired, then discard or clearly label as expired and set aside for training activities.
Kit - Contents	All required contents must be present and in acceptable condition. This is important because Abraxis has calibrated the standards and reagents separately for each kit.	If any bottles are missing or damaged, discard the kit.
Calibration	All of the following must be met: Standard curve must have a correlation coefficient of ≥0.99; Average absorbance value, $\bar{A}_0$, for S0 must be ≥0.80; and Standards S0-S5 must have decreasing average absorbance values. That is, if $\bar{A}_i$ is the average of the absorbance values for S_i, then the absorbance average values must be: $\bar{A}_0 > \bar{A}_1 > \bar{A}_2 > \bar{A}_3 > \bar{A}_4 > \bar{A}_5$	If any requirement fails: Results from the analytical run are not reported. All samples in the analytical run are reanalyzed until calibration provides acceptable results. At its discretion, the lab may consult with EPA for guidance on persistent difficulties with calibration.
Kit Control	The average concentration value of the duplicates (or triplicate) must be within the range of 0.75 +/- 0.185 µg/L. That is, the	If either requirement fails: Results from the analytical run are not reported

Quality Control Activity	Description and Requirements	Corrective Action
	average must be between 0.565 µg/L and 0.935 µg/L.	The lab evaluates its processes, and if appropriate, modifies its processes to correct possible contamination or other problems. The lab reanalyzes all samples in the analytical run until the controls meet the requirements.
Negative Control	The values for the negative control replicates must meet the following requirements: All concentration values must be < 0.15 µg/L (i.e., the reporting limit; and one or more concentration results must be nondetectable (i.e., <0.10 µg/L)	
Sample Evaluations	All samples are run in duplicate. Each duplicate pair must have %CV≤15% between its absorbance values.	If %CV of the absorbances for the sample>15%, then: Record the results for both duplicates using different start dates and/or start times to distinguish between the runs.. Report the data for both duplicate results using Quality Control Failure flag "QCF"; and re-analyze the sample in a new analytical run. No samples are to be run more than twice. If the second run passes, then the data analyst will exclude the data from the first run (which will have been flagged with "QCF"). If both runs fail, the data analyst will determine if either value should be used in the analysis (e.g., it might be acceptable to use data if the CV is just slightly over 15%).
Results Within Calibration Range	All samples are run in duplicate. If both of the values are less than the upper calibration range (i.e., ≤ 5.0 µg/L for undiluted samples with salinity<3.5 ppt; ≤ 8.75 µg/L for undiluted samples with salinity ≥3.5 ppt), then the requirement is met.	If a result registers as "HIGH", then record the result with a data flag of "HI." If one or both duplicates register as 'HIGH,' then the sample must be diluted and re-run. No samples are to be run more than twice. The lab reports both the original and diluted sample results.
External Quality Control Sample	External QC Coordinator, supported by QC contractor, provides 1-2 sets of identical samples to all laboratories and compares results.	Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any deviations from the Method or unique laboratory practices that might account for differences between the laboratory and others. With this additional information, the External QC

Quality Control Activity	Description and Requirements	Corrective Action
		Coordinator will determine an appropriate course of action, including no action, flagging the data, or excluding some or all of the laboratory's data.

3.7 Sample and Record Retention

The laboratory shall retain:

1. The sample materials, including vials, for a minimum of 3 years from the date the EPA publishes the final report. During this time, the laboratory shall freeze the materials. The laboratory shall periodically check the sample materials for degradation.

2. Original records, including laboratory notebooks and the reference library, for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

3.8 References

Abraxis, "Microcystins-ADDA ELISA (Microtiter Plate)," Product 520011, R021412, Undated. Retrieved January 2014 from http://www.abraxiskits.com/uploads/products/docfiles/278_Microcystin%20PL%20ADDA%20users%20R120214.pdf.

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James, R., et al., "Environmental Technology Verification Report: Abraxis Microcystin Test Kits: ADDA ELISA Test Kit; DM ELISA Test Kit; Strip Test Kit," in Environmental Technology Verification System Center 2010. Retrieved March 2013 from http://nepis.epa.gov/Adobe/PDF/P100EL6B.pdf

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4.0 BENTHIC MACROINVERTEBRATES

This chapter describes the steps for identifying benthic macroinvertebrate organisms in samples collected in coastal waters and the Great Lakes during the 2015 National Coastal Condition Assessment (NCCA). Field crews preserve samples in the field with formalin and ship them to a central holding facility or directly to the laboratory. Because NCCA samples generally have fewer than 400 organisms, this procedure requires the laboratory to fully sort and identify all organisms in the sample. If, upon initial inspection, a sample appears likely to have more than 400 organisms, contact the EPA HQ Laboratory Review Coordinator (see contact information in Chapter 2) for processing instructions. (EPA may require use of the subsampling procedures such as those described in the Laboratory Operations Manual for the 2013-2014 National Rivers and Streams Assessment (NRSA)).[6]

In the following discussion, Sections 4.1, 4.2, and 4.3 summarize the procedure; health and safety concerns; and definitions and required resources. Section 4.4 provides the steps for acknowledging sample receipt. Section 4.5 provides the steps for preparing and picking organisms from the sample. Sections 4.6 – 4.8 provide the steps for the taxonomy identification; data entry; and sample and record retention. Sections 4.9 and 4.10 describe EPA's external review of laboratory operations and quality measures. Section 4.11 identifies references used in developing the procedure. Attachment 4.1 provides an example of a taxonomic bench sheet.

4.1 Summary of Method

The procedure describes the steps for picking and identifying organisms from sediment samples. This section provides a summary of the procedure and quality control measures.

The sorter evenly distributes each sample across a tray(s) and then picks all organisms from the sample. During the identification step, a taxonomist identifies all organisms to the target taxonomic levels for the survey and discards materials that do not meet the identification criteria. For each species or lowest identifiable taxonomic level, the taxonomist includes at least one representative organism in the laboratory's reference collection for NCCA 2015.

As part of the quality control measures, a second taxonomist will re-identify a subset (usually 10%) of the samples to quantify enumeration and taxonomic precision, or consistency, as percent difference in enumeration (PDE) and percent taxonomic disagreement (PTD), to help target corrective actions, and ultimately to help minimize problems during data analysis.

4.2 Health and Safety Warnings

In addition to the laboratory's requirements, persons using this procedure must abide by the following health and safety procedures:

6 USEPA, 2013, National Rivers and Streams Assessment 2013-14: Laboratory Operations Manual EPA 841-B-12-010.

1. Wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear / goggles).

2. When working with potential hazardous chemicals (e.g. Rose Bengal) or biological agents (benthic organisms and sediments), avoid inhalation, skin contact, eye contact, or ingestion. If skin contact occurs, remove clothing immediately and wash / rinse thoroughly. Wash the affected skin areas thoroughly with large amounts of soap and water.

4.3 Definitions and Required Resources (Laboratory, Personnel, and Equipment)

This section provides definitions and required resources for using this procedure. Section 4.3.1 defines the terms used throughout the procedure. Section 4.3.2 describes the expertise required for each laboratory using the procedure. Section 4.3.3 describes the roles and responsibilities of the personnel involved in the procedure. Section 4.3.4 identifies the equipment necessary to apply the procedure in preparing, sorting, and identifying benthic macroinvertebrate organisms in samples.

4.3.1 Definitions

The procedure uses the following throughout the document:

Dissecting microscope: Microscope configured to allow low magnification of three-dimensional objects that are larger or thicker than the compound microscope can accommodate.

Distinct taxa: Data analysts use the number of distinct (i.e., unique) taxa within a given sample to evaluate the richness associated with the sample location. The distinctness attribute is assessed sample by sample, and not across all samples. To facilitate the data analyses, the database includes an additional variable ("flag") that is used for the first identification of a particular taxon in a sample. Section 4.6 provides the steps used to identify which taxa are flagged.

Good quality digital photograph: Good quality means that other taxonomists can readily identify the taxon from one or multiple photographs and the library can readily locate the photographs. To ensure that the photographs meet these objectives, the image must be:

- Taken through the microscope at a high enough resolution so that the key diagnostic features are distinguishable and clear. Include all features that would be necessary for an experienced taxonomist to identify the specimen, this may require multiple photographs and at different magnifications.

- Positioned so that it includes:

  - Only one taxon in the photo. If necessary, the laboratory may edit (e.g., crop) the digital photograph and save the file with a new filename as specified below. Both the original and edited files must be included in the digital library.
  - A scale bar or measurements in an appropriate location to indicate the size of the specimen.
  - One specimen that lies flat on the surface instead of tilted (to the extent practicable).

- Saved using a format that preserves the image in the highest resolution possible.

- Saved with a filename that is consistent within the digital library and shall include the following elements in the order listed below:
  - NCCA2015
  - Laboratory name (or abbreviation)
  - Sample number
  - Taxa name
  - Magnification (if applicable, otherwise indicate no magnification as "1x")
  - Date (format YYYYMMDD) that the photograph was taken.
  - Appendage of "e" if the photograph was edited (e.g., cropped).

  For example, on September 8, 2015, laboratory ABC identified the specimen in sample 1234 to be a *Capitella capitata* and took a digital photograph at a resolution of 40x and then cropped the photograph to eliminate extraneous material. The filenames of the original and edited photographs would be: NCCA2_ABC_1234_ capitella capitata_40x_20150908.gif and NCCA2_ABC_1234_ capitella capitata_40x_20150908e.gif.

Elutriate: Circulate water over the sample in order to wash away the lighter or finer particles of the detritus.

Inorganic material: Material that is not capable of further decay (e.g., gravel, sand, silt)

Integrated Taxonomic Information System (ITIS): Database with standardized, reliable information on species nomenclature and their hierarchical taxonomic classification.

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management (IM) System: The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. The samples are collected during the field stage of NCCA.

Organic material: Material derived from living organisms that is capable of further decay (e.g., leaves, sticks, algae).

Percent sorting efficiency (PSE): Number of organisms recovered by sorter (A) compared to the combined (total) number of recoveries by the sorter (A) and independent sorter (B) for a sample (sorter B sorts through pickate and counts only organisms missed by Sorter A).

$$PSE = \frac{A}{A+B} \times 100 \quad (1)$$

Percent disagreement in enumeration (PDE): measure of taxonomic precision comparing the number of organisms, n_1, counted in a sample by the primary taxonomist with the number of organisms, n_2, counted by the internal or external QC taxonomist.

$$PDE = \frac{|n_1 - n_2|}{n_1 + n_2} \times 100 \quad (2)$$

Percent taxonomic disagreement (PTD): measure of taxonomic precision comparing the number of agreements (positive comparisons, $comp_{pos}$) of the primary taxonomist and internal or external QC taxonomists. In the following equation, N is the total number of organisms in the larger of the two counts.

$$PTD = \left[1 - \frac{comp_{pos}}{N}\right] \times 100 \quad (3)$$

Pickate: This is the remaining material left from the tray after the sorter has removed all benthic macroinvertebrates. This could include small stones, sticks or leaves, etc.

Primary laboratory: The laboratory that 1) sorts the sample; and 2) provides the first identification of benthic macroinvertebrates in the sample.

Secondary laboratory: The laboratory selected by the External QC Coordinator. It provides an independent identification of the benthic macroinvertebrates in the sample. The secondary laboratory must provide QC taxonomists who did not participate in the original identifications for the sample.

Target taxonomic levels: Target taxonomic levels for the NCCA is typically species (lowest practical level). NCCA excludes meiofauna (due to being smaller than 0.5 mm) from identifications. Additional exceptions include Oligochaeta (Class) and Chironomidae (Family) in samples from marine, polyhaline and mesohaline regions **ONLY**.

Taxonomic Bench Sheet: Form used by the laboratory to record information about the sample during the identification procedure.

Taxonomic Serial Number (TSN): stable and unique identifier that the Integrated Taxonomic Information System (ITIS), Encyclopedia of Life, and/or Catalogue of Life

couples with each scientific name to serve as the "common denominator" for accessing information. ITIS numbers are preferred, but when they are not available, secondary sources are acceptable.

a)

4.3.2 Laboratory

The procedure may be used by any laboratory that demonstrates competency in analytical work and quality procedures as documented by any one or more of the following::

1. Analytical work: To demonstrate its expertise, the laboratory shall provide EPA with one or more of the following:
    a. Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
    b. Memorandum describing experience with analyses that are the same or similar to the requirements of this method.
    c. Dated copy of relevant Accreditation or Certification (NELAC, ISO, state, etc.) for the laboratory and/or its experts who will perform and/or oversee the analyses. The accreditation must be for the entirety of analysis that the laboratory will be performing.
    d. Memorandum that describes the laboratory's participation in round robin studies and/or performance studies.
    e. Report of findings from an on-site technical assessment or audit.

2. Quality procedures.
    a. To demonstrate its expertise in quality assurance and quality control procedures, the laboratory shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).
    b. To demonstrate its ongoing commitment, the person in charge of quality issues for the laboratory shall sign the NCCA 2015 QAPP Certification Page.

3. Reporting standardized data. To demonstrate its expertise, the laboratory shall provide EPA with a memorandum that confirms that the laboratory has a computerized Laboratory Information Management System (LIMS) routinely used to track samples and record laboratory results. The memorandum also shall confirm that the laboratory will use LIMS to record and report results from the procedure.

4.3.3 Personnel

The procedure may be used by any person who has received training in processing and identification of benthic macroinvertebrates. For purposes of this procedure, EPA assumes that the following personnel are responsible for performing specific duties:

Internal Taxonomy QC Officer provides oversight of daily operations, sample processing, monitors QC activities at the laboratory to determine conformance, and conducts performance and systems audits of the procedures. The laboratory must retain documentation for the qualifications for the Internal Taxonomy QC Officer meeting the following requirements. The laboratory must provide, or otherwise make available, this documentation to EPA upon request. The Internal Taxonomy QC Officer is an experienced taxonomist who:

1. Demonstrated an initial enumeration and identification proficiency (as measured by PDE≤5% and PTD≤15%.
2. Maintains enumeration and identification proficiency in periodic QC checks (i.e., 1 in 10 samples with a minimum of one sample checked).

External QC Coordinator is an EPA staff person. Because the assigned duties are primarily administrative in nature, the External QC Coordinator is not required to have laboratory experience, although such experience would be preferable.

External QC Taxonomists, are selected by the External QC Coordinator (after consultation with EPA experts), and have demonstrated expertise and experience to be used as a quasi "gold standard" for taxonomic evaluations.

Taxonomists are trained, and have considerable experience, in identifying benthic macroinvertebrates, i.e., taxonomy. It is also important that the taxonomist maintains contact with other taxonomists through professional societies and other interactions, and keeps up with the pertinent literature, since systematics and species identifications change over time. EPA prefers, but does not require, that the freshwater taxonomists are certified by the Society of Freshwater Science (SFS). Each laboratory must submit the resume or *curriculum vitae* for the taxonomists who identify benthic macroinvertebrates for the NCCA samples to the EPA Project QC Officer.

Sorters are laboratory technicians who have basic training in laboratory procedures. An "experienced" sorter is one that has achieved ≥90% sorting efficiency in 5 consecutive samples.

4.3.4 Equipment/Materials

The procedure requires the following equipment and materials for sample preparation (subsampling), sorting, and taxonomic identifications.

4.3.4.1 Sample Preparation (Subsampling) and Sorting Equipment/Materials

- U.S. 35 sieve (500 µm)
- Round buckets
- Standardized, possibly, gridded screen (40 Mesh (380-µm openings, T304 stainless steel wire, 34GA (0.010"))
- 6-cm scoop
- White plastic or enamel pan (6" x 9") for sorting
- Teaspoon

- Permanent ink pen (e.g Pigma Micron® pen)
- Dropper
- Fine-tipped forceps (watchmaker type, straight and curved)
- Vials with caps or stoppers
- Sample labels for vials
- 70-80% ethanol
- Stereo zoom microscope (6-10X magnification)

4.3.4.2 Taxonomy Identification Equipment/Materials

- Stereo dissecting microscope with fiber optics light source (50-60X magnification)
- Compound microscope (10, 40, and 100X objectives, with phase-contrast capability)
- Digital camera with high resolution capability mounted on a microscope
- Petri dishes
- Microscope slides (1" x 3" flat, precleaned)
- Cover slips (appropriately sized)
- CMCP-10 (or other appropriate mounting medium)
- Permanent ink pen (e.g Pigma Micron® pen)
- Dropper
- Fine-tipped forceps (watchmaker type, straight and curved)
- Vials with caps or stoppers
- Sample labels for vials
- 70 - 80% non-denatured ethanol in plastic wash bottle
- Taxonomic Bench Sheet (Attachment 4.1 provides an example)
- Hand tally counter

4.4 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel should start the following login steps within 24 clock hours of receiving a delivery.

1. Record receipt of samples in the NARS IM system (within 24 clock hours) and the laboratory's Information Management System (LIMS). Assign the appropriate chronological bench number to each sample. Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Inspect each jar THE SAME DAY THEY ARE RECEIVED:
   a. Add 70-80% formalin to the jar, if necessary (i.e., to cover the contents completely).
   b. Verify that the site identification and sample number on the label also appear on the chain of custody form in the shipment.
   c. Notify the EPA HQ Laboratory Review Coordinator (see contact information in Chapter 2) if any jars were broken and/or there are discrepancies between the custody form and jars.

3. Store the sample containers at room temperature until sorting begins. If the sample will be stored for a long time before sorting, replace the formalin with ethanol for better preservation of the organisms.

4. Maintain the chain-of-custody form with the samples; it will be needed if the samples are transported to any other location (e.g., for taxonomic identification, external QC evaluation).

5. Verify that the login information includes the required data elements in Table 4.1. After completing all required elements, provide the information to the data entry personnel.

Table 4.4.1 Benthics Macroinvertebrates Login: Required Data Elements

FIELD	FORMAT	DESCRIPTION	
LAB NAME	Character	Name of lab	
LAB ID (optional)	Character	Lab sample id	
DATE RECEIVED	MMDDYY	Date sample was received by lab	
SITE ID	Character	NCCA site identification code as used on sample label	
VISIT NUMBER	Numeric	Sequential visits to site (1 or 2, if specified on label)	
SAMPLE ID	Numeric	Sample number as used on field sheet (on sample label)	
DATE COLLECTED	Date	Date sample was taken	
SALINITY	Numeric	Salinity: Value is provided on the sample label	
CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory.	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		NP	Not enough preservative used
		Q	Other quality concerns, not identified above (explain in COND_COMMENTS)
COND_COMMENTS	Character	Explanation for Q FLAG (if needed)	

4.5 Sample Preparation and Picking Organisms

This section describes the steps for the sorter in preparing the sample and picking organisms.

1. Remove the lid from the sample container and remove the internal sample label.

2. Carefully decant the formalin from the sample container by pouring the fluid through a sieve (U.S. 35) into a separate container. Inspect the mesh of the sieve for any organisms and return any organisms found to the sample container so they can be included in the sample sort process.

3. Remove sieved organisms from the sample container and place into a sorting tray.

4. Sort all samples under a minimum of 6x (maximum of 10x) dissecting microscope. Remove the macroinvertebrates from the detritus with forceps. In general, do not remove:
    - Empty snail or bivalve shells
    - Organisms of water surface-dwelling or strict water column[2] arthropod taxa, and meiofauna.
    - Incidentally-collected terrestrial taxa.
    - Fragments such as legs, antennae, gills, wings, or tails.

   For Oligochaeta, attempt to remove only whole organisms or fragments that include the head. In other words, do not remove fragments without the head.
    - In case of uncertainties, place the organism in the sort vial for the taxonomist to make the final determination.

5. Place picked organisms of the same type into a single set of jars and vials containing 70-80% ethanol.

6. This QC step is performed if: 1) the sorter (sorter A) has not reached 90% proficiency in 5 consecutive samples (referred to as the "proficiency QC check" below); or 2) this sample is the 1 in 10 sample QC check for experienced sorters (referred to as the "periodic QC check" below). For this step, a second sorter (sorter B):
    - Performs QC checks using the same power microscope as the sorter;
    - Extracts any missed organisms found in the pickate from Sorter A and places them into the sample vial, or other suitable sample vial;
    - Notes the number of organisms missed; and
    - Adds that number to the final count of the sample.
    - Calculates the PSE for the sample (see Section 4.3.1 for definition; equation 1). If the PSE is:
        - <90% and the sample is the:
            - Proficiency QC check, a second sorter must check the next 5 samples until the original sorter has PSE≥90% for 5 consecutive samples.
            - Periodic QC check, then a second sorter examines the original sorter's samples since the last QC check for missed organisms. The original sorter must again demonstrate proficiency by achieving a PSE≥90% in 5 consecutive samples.
        - ≥90% and the sample is the:
            - Proficiency QC check, the sample counts towards the 1 in 5 consecutive samples used to establish proficiency.
            - Periodic QC check, no corrective action is required.
    - Records the results from the QC step. The laboratory must record the results from all QC steps, even if they exceed the frequency required by this step. The laboratory must provide the sorter QC results to EPA upon request.

[2]Strict water column taxa are those that do not have at least one life stage that is benthic (i.e., bottom-dwelling).

7. Remove the remaining material left on the sorting pan (i.e. material such as sticks, organic debris) and place it in a separate container with preservative (70-80% ethanol). Label the container "Pickate," on both internal and external labels.

8. Label the vials and jars of sorted organisms and material using permanent ink (e.g., using a Pigma Micron® pen). Internal sample labels should be made of cotton rag paper or an acceptable substitute.

9. Retain the vials and materials for the time period specified in Section 4.8.

10. Thoroughly clean all sample preparation and sorting equipment and make sure all equipment is free of organisms prior to sorting the next sample.

4.6 Taxonomic Identification

The taxonomist performs the following steps in identifying the benthic macroinvertebrate organisms:

1. Upon receipt of a set of sample vials from the sorter:
    a. Compare all site identification codes and sample numbers on the form with those entered on the labels of samples, and resolve any discrepancies with the sorter.
    b. Determine if any vials are broken. For any broken vial, attempt to recover as much of the sample as possible. Describe the damage in the LAB_COMMENTS field in the database.
    c. Maintain the chain-of-custody form with the sample vials; it will be needed to return/store them.

2. Empty one sample vial at a time into a small Petri dish. Add 70-80% ethanol to keep the organisms covered. Remove the internal sample label and complete the top portion of a Taxonomic Bench Sheet (for an example, see Attachment 4.1), using the information from the label. Depending on the type of organisms, select the appropriate step:
    a. For all *Chironomidae* organisms, extract the organisms from the Petri dish.
        i. Prepare slide mounts using CMCP-10 (or CMC-9, CMC-10, or other media) and applying a coverslip. All organisms must be visible, which generally means a maximum of 10-20 organisms per slide. Label the slides with the same sample identification code or log-in number as the ethanol organisms.
        ii. If the laboratory prefers to use another method than slide mounting, the EPA External QC Coordinator will grant a waiver if the following applies:
            1) The request is for a laboratory located at a single location. For example, EPA would *not* consider the combined qualifications of a prime contract laboratory and its subcontract laboratories. Instead, for whichever laboratories met the requirements, EPA would evaluate and grant (or deny) a waiver for the prime contract laboratory separate from each of its subcontractor laboratories.

2) The request for a waiver must identify and describe a minimum of three studies. For each study, the external QC evaluation must demonstrate that the laboratory met or exceeded the NCCA QC requirements (i.e., PDE≤5% and PTD≤15%) for its *Chironomidae* organisms.
3) The laboratory agrees to mount the organisms on slides if it fails one of the periodic (NCCA) external QC evaluations, as follows:
    a. It must mount all *Chironomidae* organisms in samples processed since the previous external QC evaluation (i.e., for which it met the PDE and PTD requirements).
    b. It must continue to mount all *Chironomidae* organisms for the unprocessed samples.

b. For all other organisms, remove similar organisms to other dishes (keep these covered with 70-80% ethanol).

3. View the sample to ensure that all necessary diagnostic characters have been observed, according to the taxonomic key or other literature using:
    a. A stereo dissecting microscope for organisms in dishes.
    b. A compound microscope for slides of *Chironomidae* and *Oligochaeta* organisms

4. Identify organisms to the lowest practical taxonomic level (species is the target for all organisms with the exception of meiofauna, (due to being smaller than 0.5 mm). Additional exceptions include Oligochaeta (Class) and Chironomidae (Family) in samples from marine, polyhaline and mesohaline regions **ONLY**. If a laboratory or individual taxonomist is having trouble reaching species for a taxonomic group (not for an individual organism which might be damaged or otherwise difficult to identify), the lab must contact the NCCA project lead for guidance. Add any necessary data qualifiers (see list provided with Required Data Elements in Table 4.2).
    a. Enter the Taxonomic Serial Number (TSN) as it appears in the column "Unique Identifier" of the taxa list provided by EPA.
    b. Note whether the identification of a group of organisms is distinct (Distinct=Y/N) from other organisms in the same sample as follows:
        i. If the organisms can be identified to the target level, then Distinct="Y."
        ii. If an organism cannot be identified to the target level then assign values as follows:
            1) If at least some of the organisms in the sample can be identified to the target level, then:
                a. Distinct="Y" for organisms identified at the target level; and
                b. Distinct="N" for organisms that were identified at a higher taxonomic level (e.g., family) that may contain a target level taxa already identified in a given sample (e.g., genus).
                c. An example would be, if some organisms from a sample are identified to *Macoma*, but other organisms in the sample could only be identified to *Tellinidae* and/or

Veneroida, then *Macoma* would be distinct, but *Tellinidae* and/or *Veneroida* would not be Distinct.

2) If none of the organisms in the sample could be identified at the target level, then:
    a. Distinct=”Y” for organisms identified at the lowest taxonomic level (e.g., family); and
    b. Distinct=”N” for organisms identified at a higher level (e.g., order).
    c. For example, if a taxonomist can identify a number of *Veneroida* (Order) families, but a number of the organisms could not be taken past *Veneroida*, then the individual families would be distinct, but the order would not be distinct.

Record the identifications. For example, using the taxonomic bench sheet in Attachment 4.1, record the identification in the Column labeled "taxon." Enter the number of larvae, pupae, and adults, or total count (e.g. mollusks), if appropriate life history column does not apply, of each taxon under the appropriate columns.

iii. If the target taxonomic level cannot be achieved due to immature or damaged organisms this should be noted in the data file in the QA_FLAG field (e.g., QA_FLAG=IM). Table 4.2 provides other codes for the QA_FLAG field.

iv. If damaged organisms can be identified, they are counted ONLY if the:
    1) Fragment includes the head, and, in the case of arthropods, the thorax;
    2) Oligochaetes have a sufficient number of segments in the head;
    3) Mollusk shell (bivalve or gastropod) is occupied by a organism;
    4) Organism is the sole representative of a taxon in the sample.

v. If a unique taxon is determined for which the appropriate taxonomic level is not available in the literature and there are other taxa in that taxonomic level:
    1) Provide good quality digital photographs of the organism to outside experts for identification; and
    2) Include the tentative identification in the database with a data qualifier code of QA_FLAG='UN' so that these organisms can be distinguished from other organisms in the data analysis.
    3) When the outside expert identifies the organism, update the database with the correct identification.

5. Compare taxa names from the taxa list provided by EPA to the names used for the identifications. Check the non-matches for the following common problems and correct them.
    a. Abbreviations
    b. Extra information identifiers (e.g., sp., spp., , nr., cf., genus 1, w/ hair chaetae)
    c. Extra character (e.g., "?", "Acentrella ?turbida", blank space)
    d. The word "probably" or "prob" (e.g., "Microcylloepus prob. similis")
    e. Double names (e.g., Callibaetis callibaetis)
    f. Common misspellings
    g. Tribes/subfamilies/subgenus sometimes may not appear
    h. Species with incorrect genus (Hydatopsyche betteni)
    i. Split level taxonomy (e.g., Cricotopus/Orthocladius)
    Invalid name (e.g., taxonomic change, synonym; Sphaeriidae vs. Pisiidae)

6. Complete the identification by entering the totals for each developmental stage and the total number of each taxon in the cells at the bottom of the sheet. Cross-check to be sure the totals were summed correctly.

7. Provide the data to the Internal Taxonomic Officer for another review to confirm that the identifications use the same nomenclature as the taxa list provided by EPA and the laboratory's reference collection.

8. Make two copies of the bench sheet or computer file used to record the identifications. They are distributed as follows: 1) the project file; and 2) EPA's External QC Coordinator.

9. Prepare a list of primary and secondary technical literature used in completing the identifications. Provide complete citations in bibliographic format, including authors' names, date of publication, title of document, name of journal or publisher, volume and page numbers, or ISBN number, as appropriate. These citations will be kept on file with the Internal Taxonomic QC Officer, who will periodically review the reference collection to ensure that it is complete.

10. Verify that the reference collection contains at least one organism that represents each genus (or lowest taxonomic level) identified from all sample. For any missing references, choose an appropriate organism(s) from the sample to represent a taxon name in the master taxa list:
    a. Place the physical specimen in the reference library.
    b. Place two labels in the sample container to identify: organisms placed in the reference collection, and those in the non-reference organisms.
    c. Obtain a good quality representative digital photographs of the specimen (see instructions in Section 4.3.1).

11. If the Internal Taxonomy QC Officer selects the sample for a QC check, the Internal Taxonomy QC Officer re-counts and re-identifies the organisms in the sample following the same steps above for the original taxonomist. One in 10 of the taxonomist's samples must be checked. The Internal Taxonomy QC Officer records the independent verifications on a bench sheet or computer file. The Internal Taxonomy QC Officer will also supply a list of taxa that were found to be problematic during their QC sorting check, which can be submitted in an Excel or Word document format. (If the Internal Taxonomy QC Officer performs the QC check more frequently, then all QC data must be submitted.)

12. Carefully return the rest of the organisms to the original sample vial, fill with 70-80% ethanol, and cap tightly.

13. Re-package the samples and slide-mounted organisms carefully, and sign and date the chain-of-custody form. Return or store the samples according to laboratory protocols and requirements in Section 4.8.

14. Verify that all required data elements in Table 4.2 have been recorded by the taxonomist and Internal Taxonomy QC Officer. If the results were recorded on paper, provide the Taxonomic Bench Sheet to the data entry personnel.

Table 4.2 Benthic Macroinvertebrates Taxonomic Identification: Required Data Elements

FIELD	FORMAT	DESCRIPTION
LAB NAME	Character	Name of lab

FIELD	FORMAT	DESCRIPTION	
LAB ID (optional)	Character	Lab sample id	
DATE RECEIVED	Date	Date sample was received by lab	
SITE ID	Character	NCCA site identification code as used on sample label	
VISIT NUMBER	Numeric	Sequential visits to site (1 or 2, if specified on label)	
SAMPLE ID	Numeric	Sample number as used on field sheet (on sample label)	
DATE COLLECTED	Date	Date sample was taken	
DATE TAXON	Date	Date that the taxonomist started identifying organisms in the sample	
ANALYST NAME	Character	Name of taxonomist or Internal Taxonomy QC Officer (if record provides results of QC check)	
QC VERIFICATION	Character	Y if the record provides the results from the QC check	
FAMILY	Character	Taxonomic family	
SUBFAMILY	Character	Taxonomic subfamily	
TRIBE	Character	Taxonomic tribe	
GENUS GROUP	Character	Taxonomic genus group (e.g., *thienemannimyia*)	
GENUS	Character	Taxonomic genus	
SPECIES	Character	Taxonomic species	
TSN	Numeric	Taxonomic Serial Number as defined by "UniqueIdentifier" in taxa list provided by EPA. If taxon is not in this list, provide citation for reference used to identify organism in CITATION field	
LAB TIN (OPTIONAL)	Character	Lab taxa ID number	
TAXANAME	Character	Unique taxon name in the taxa list provided by EPA	
ABUNDANCE LARVAE	Numeric	Number of individual larvae or immature bugs	
ABUNDANCE PUPAE	Numeric	Number of individual pupae	
ABUNDANCE ADULT	Numeric	Number of individual adults	
ABUNDANCE TOTAL	Numeric	Total number of individuals	
DISTINCT	Character	Distinct taxa in sample (y/n) (See description in Section 4.6)	
CITATION	Character	Citation for reference used to identify organism, if taxon not present in taxa list provided by EPA database	
QA FLAG (if appropriate)	Character	QA/QC flag (lab may use its own flags, if defined in QA_COMMENTS field or provided to NARS IM team)	
		Flag	Definition
		DD	Damaged Organism, poor condition or fragments
		IM	Immature
		IN	Indeterminate (explain in QA_COMMENTS field)
		NP	Not enough preservative used
		NT	Not able to meet target level for identification (may be used with other codes, or explain in QA_COMMENTS field)
		S	Sample shipping problem (explain in QA_COMMENTS field)

FIELD	FORMAT	DESCRIPTION	
		UN	Unknown. Identification is tentative. Organism has been sent to expert taxonomist for definitive identification.
		Q	Other quality concerns, not identified above
QA_COMMENTS	Character	Explanation for QA FLAG (if needed)	
LAB COMMENTS	Character	General laboratory analysis comments	

4.7 Data Entry

Tables 4.1 and 4.2 identify the required data elements that the sorting and taxonomic laboratories must provide to EPA, preferably in EPA's data template, available separately from EPA. In addition, the laboratory must provide the resume or *curriculum vitae* for each taxonomist who identifies benthic macroinvertebrates for the NCCA samples. The resume or *cv* for each taxonomist is submitted once to EPA's External QC Coordinator.

4.8 Sample and Record Retention

The laboratory shall retain:

1. The sample materials, including vials, slides, and sorting residuals, for a minimum of 3 years from the date the EPA publishes the final report. During this time, the laboratory shall store the materials in a cool location away from sunlight. The laboratory shall periodically check the sample materials for degradation and refill jars and vials with 70-80% ethanol if necessary.

2. Original records, including laboratory notebooks and the reference library, for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

4.9 External Taxonomic Quality Control

EPA requires that all NCCA laboratories ("primary laboratories") participate in the External Taxonomic Quality Control Evaluation. Each taxonomist must participate in the QC evaluation, even if the taxonomist is under subcontract with, or consulting for, another firm.

In contrast to the internal QC evaluation in Section 4.6 that verify adherence to the procedures and ensures in-laboratory consistency between taxonomists, the purpose of the external QC evaluation is to ensure consistency between laboratories and taxonomists. To achieve this objective, EPA compares the primary laboratory results to those from a second laboratory, considered a quasi "gold standard" for taxonomic evaluations.

The External QC Coordinator, who is an EPA staff member, is responsible for selecting and managing the "QC contractor." To eliminate the appearance of any inherent bias, the QC contractor must be dedicated to QA/QC functions, and thus, must not be a primary laboratory or a field sampling contractor for NCCA. The QC contractor is responsible for complying with instructions from the External QC Coordinator; obtaining and managing the secondary laboratory; coordinating and paying for shipments of the QC samples between locations; comparing sample identifications by different laboratories; facilitating reconciliation teleconferences; and preparing brief summary reports.

The External QC Coordinator will arrange for the QC contractor to conduct a minimum of two QC evaluations. To the extent practicable, the External QC Coordinator and QC contractor will schedule batch evaluations evenly throughout the project period.

Each QC evaluation consists of the following steps:

1. In consultation with the QC contractor, the External QC Coordinator determines an appropriate time to conduct the evaluation based upon the total number of samples assigned to the laboratory, the delivery schedule, processing schedule, and the following constraints:
    a. Availability of samples from other laboratories. For example, if three state laboratories are each processing less than 30 samples, the External QC Coordinator might combine their samples into one batch for the QC evaluation.
    b. If a primary laboratory is responsible for processing 100 samples or more for the NCCA, the External QC Coordinator will split their samples into several batches (e.g., each 50 to 100 samples) so that EPA can evaluate and correct performance on an ongoing basis.

2. The External QC Coordinator provides the QC contractor with a list of laboratories and processed samples. Sample identification includes the site identification code, sample number, and taxonomist who performed the identifications.

3. The QC contractor randomly selects 10% of the samples from each NCCA laboratory, subject to the following constraints:
    a. If the primary laboratory received fewer than 30 samples, then the QC contractor randomly selects three samples for the evaluation.
    b. For each taxonomist identified on the list, the QC contractor ensures that the selection includes one or more of his/her samples.
    c. The External QC Coordinator may elect to provide an initial evaluation of the national laboratory by selecting a small batch from the samples that the laboratory completed in the first 2-3 months.

4. The QC contractor provides a list of the QC samples, and instructions, to the External QC Coordinator and each primary laboratory participating in the evaluation. Although the External QC Coordinator and QC contractor may tailor the instructions for the participating taxonomists' preferences, the instructions are likely to specify the following:

a. Pack and ship the QC samples to the central holding facility designated by the QC contractor. Instructions are likely to require that the:
    i. Shipments contain chain-of-custody documentation for all slides and containers.
    ii. Containers (e.g., slides, vials) include the site identification code and sample number.
    iii. Containers cannot be marked in any way that might identify the taxonomic classification for any organism.
    iv. The number of taxa in a vial or container should be based on practical considerations (e.g., size of animals and amount of ethanol needed for preservation, amount of ethanol allowed in a single shipment to meet DOT shipping requirements).
b. Track the QC samples using forms provided by the QC contractor.
c. Email a spreadsheet with the data for the QC samples to the External QC Coordinator. (EPA requires that all labs use its spreadsheet template for recording the taxonomic data.)

5. The QC contractor reviews the condition of the QC samples (e.g., verifies that the containers do not identify taxon for any organism) and ships the samples to the secondary laboratory along with instructions and the EPA template for reporting data.

6. Within 24 hours of receipt, the secondary laboratory:
    a. Notifies the QC contractor that it has received the samples;
    b. Faxes or emails any additional receipt records, including discrepancies, within 24 hours; and
    c. Completes any other instructions from the QC contractor.

7. The secondary laboratory:
    a. Re-identifies and re-counts following the procedures in the Method, except does not:
        i. Develop a reference library.
        ii. Photograph organisms unless the taxa are identified for reconciliation discussion.
        iii. Perform any internal QC checks.
    b. Records the required data elements in Section 4.7.
    c. Enters the data using EPA's spreadsheet template for the taxonomic data.
    d. Emails the completed spreadsheet to the QC contractor.

8. The QC contractor compares the original taxonomic results (i.e., data) generated by the primary laboratory to the taxonomic results generated by the secondary laboratory for each sample. As part of this evaluation, the QC contractor calculates PDE and PTD using the equations in Section 4.3.1 and compares their values to the QC requirements in the Section 4.10.

9. If any samples exceed the PDE or PTD limits in Section 4.10, the QC contractor consults with the External QC Coordinator to determine if reconciliation calls are necessary to

resolve differences. The External QC Coordinator may decide that a reconciliation call is unnecessary if there appears to be an obvious explanation for differences, few samples are affected, or other reasons.

10. The QC contractor schedules and facilitates reconciliation teleconferences with EPA and the laboratories.
    a. In preparation for the teleconferences:
        i. The QC contractor instructs the secondary laboratory to photograph representative specimens for each taxon identified for discussion.
        ii. The QC contractor provides the participants with a spreadsheet that includes:
            1. List of samples and taxon identifications for discussion;
            2. Relevant data from the primary and secondary laboratories; and
            3. PDE and PTD values.
        iii. The primary and secondary laboratories provide participants with the relevant reference (or citation) and photograph for each taxonomic identification for the discussion.
        iv. The QC contractor emails a meeting announcement for a convenient time for all participants. The email identifies instructions for accessing the External QC Coordinator's toll-free teleconference line.
    b. Within a week after the teleconference, the QC contractor sends an email to the External QC Coordinator and other teleconference participants that summarizes:
        i. Agreements to use common nomenclature for discrepancies;
        ii. Commitments to reevaluate identifications by reexamining samples;
        iii. Application of changes that are appropriate for all samples, not just the QC samples (e.g., common nomenclature)
        iv. Items that will not be resolved for some reason (e.g., sample degraded during shipment).

11. After completing the reconciliation calls, the participants complete the following steps:
    a. Secondary laboratory:
        i. Reexamines samples as deemed necessary during the reconciliation call
        ii. Updates its database with changes to:
            1. QC samples per reexamination and other items in the QC contractor email; and
            2. Non-QC samples as appropriate (e.g., nomenclature changes apply to all samples, not just QC samples).
        iii. Provides database to QC contractor.
    b. QC contractor confirms that the secondary laboratory (i.e., its subcontractor) completed its assignments before allowing the secondary laboratory to move to the next step.
    c. Secondary laboratory stores its original records, including laboratory notebooks and the reference library, for a minimum of 10 years from the date that EPA publishes the final report.
    d. Secondary laboratory and QC contractor follow steps 4 and 5 above to return the samples to the primary laboratory.

e. After receiving the samples (and tracking per step 4), the primary laboratory:
    i. Reexamines samples as deemed necessary during the reconciliation call;
    ii. Updates its database with changes to:
        1. QC samples per reexamination and other items in the QC contractor email; and
        2. Non-QC samples as appropriate (e.g., nomenclature changes apply to all samples, not just QC samples)
    iii. Provides the revised database to the External QC Coordinator (not the QC contractor). It also confirms that it has completed all relevant items identified in the QC contractor's email summary of the teleconferences (from Step 10.b).
f. QC contractor provides EPA with a report or memorandum that:
    i. Identifies the participating laboratories, with the following information about each laboratory:
        1. Laboratory name
        2. Address
        3. Contact person (name, telephone, and email)
    ii. Quantifies the taxonomic precision (PDE and PTD) as they were prior to the reconciliation call;
    iii. Assesses data acceptability;
    iv. Highlights taxonomic problem areas;
    v. Identifies any discrepancies for which the External QC Coordinator determined that a reconciliation teleconference was not necessary;
    vi. Identifies primary and secondary laboratory commitments to change its identifications or provide additional review of any organisms; and
    vii. Provides recommendations for improving precision for other samples not included in the QC evaluation.

12. After review, the External QC Coordinator:
    a. Submits the report, and draft technical direction with next steps for the laboratory, to the EPA staff managing or coordinating with the primary laboratory.
    b. Determines if significant differences within the batch of QC samples warrant re-identification of samples by the primary laboratory and a second QC evaluation by the secondary laboratory. If deemed necessary, EPA will instruct the primary laboratory to include the samples for review with the next batch of QC samples.

As an additional verification on the generation of the data, EPA may conduct assistance visits at the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter. The objective of the visit would be to:

- Confirm the laboratory is properly implementing the steps in the method.
- Assist with questions from laboratory personnel.
- Suggest corrections if any errors are made.

4.10 Quality Assurance/Quality Control (QA/QC)

Equation 4.1 Percent sorting efficiency (PSE)
Number of organisms found by the sorter (A) compared to the combined (total) number of found by the sorter (A) and the number recovered by the QC Officer (B) from Sorter A's pickate for a sample. PSE should be ≥90%.

$$PSE = \frac{A}{A+B} \times 100$$

Equation 4.2 Percent disagreement in enumeration (PDE)
Measure of taxonomic precision comparing the number of organisms, n_1, counted in a sample by the primary taxonomist with the number of organisms, n_2, counted by the internal or external QC taxonomist. PDE should be ≤5%.

$$PDE = \frac{|n_1 - n_2|}{n_1 + n_2} \times 100$$

Equation 4.3 Percent taxonomic disagreement (PTD)
Measure of taxonomic precision comparing the number of agreements (positive comparisons, $comp_{pos}$) of the primary taxonomist and internal or external QC taxonomists. In the following equation, N is the total number of organisms in the larger of the two counts. PTD should be ≤15%.

$$PTD = \left[1 - \left(\frac{comp_{pos}}{N}\right)\right] \times 100$$

Table 4.3 Benthic Macroinvertebrates: Measurement Data Quality Objectives

Variable or Measurement	Precision	Accuracy
Sort and Pick	90% [a]	90% [a]
Identification	85% [b]	95% [c]

NA = not applicable; [a] As measured by PSE; [b] As measured by (100%-PTD); [c] As measured by (100%-PDE)

Table 4.4 Benthic Macroinvertebrates: Laboratory quality control

Check or Sample Description	Frequency	Acceptance Criteria	Corrective Action
SAMPLE PROCESSING AND SORTING			
Sample pickate examined by another sorter	10% of all samples (minimum of 1)	PSE ≥ 90%	If < 90%, examine all residuals of samples by that sorter and retrain sorter

Check or Sample Description	Frequency	Acceptance Criteria	Corrective Action
	completed per sorter		
IDENTIFICATION			
Duplicate identification by Internal Taxonomy QC Officer	1 in 10 samples per taxonomist,	PTD ≤15%	If PTD >15%, reidentify all samples completed by that taxonomist since last meeting the acceptance criteria, focusing on taxa of concern
Independent identification by outside, expert, taxonomist	All uncertain taxa	Uncertain identifications to be confirmed by expert in particular taxa	Record both tentative and independent IDs
External QC	10% of all samples completed per laboratory	PDE ≤ 5% PTD ≤ 15%	If PDE > 5%, implement recommended corrective actions. If PTD > 15%, implement recommended corrective actions.
Use of widely/commonly accepted taxonomic references by all NCCA labs	For all identifications	All keys and references used by each lab must be on bibliography prepared by one or more additional NCCA labs or in The taxa list provided by EPA. This requirement demonstrates the general acceptance of the references by the scientific community.	If a lab proposes to use other references, the lab must obtain prior permission from External QC Officer before submitting the data with the identifications based upon the references.
Prepare reference collection	Each new taxon per laboratory	Complete reference collection to be maintained by each individual laboratory	Internal Taxonomy QC Officer periodically reviews data and reference collection to ensure reference collection is complete and identifications are accurate
DATA VALIDATION			
Taxonomic "reasonableness" checks	All data sheets	Taxa known to occur for coastal waters or Great Lakes.	Second or third identification by expert in that taxon

4.11 References

Epler, J.H. 2001. Identification manual for the larval chironomidae (Diptera) of North and South Carolina. A guide to the taxonomy of the midges of the southeastern United States, including Florida. Special Publication SJ2001-SP13. North Carolina Department of Environment and Natural Resources, Raleigh, NC, and St. Johns River Water Management District, Palatka, FL. 526 pp.

Merritt, R.W., K.W. Cummins, and M.B. Berg (editors). 2008. An Introduction to the Aquatic Insects of North America, 4rd edition. Kendall/Hunt Publishing Company, Dubuque, Iowa.
Stribling, J.B., S.R. Moulton, and G.T. Lester. 2003. "Determining the quality of taxonomic data." Journal of the North American Benthological Society 22(4):621-631.

USEPA. 2012. National Rivers and Streams Assessment 2013-2014: Laboratory Operations Manual. EPA-841-B-12-010. U.S. Environmental Protection Agency, Office of Water, Washington, DC.

Attachment 4.1: Benthic Macroinvertebrates: Taxonomy Bench Sheet (example)

Laboratory Information		Sample Information	
Project ID		Sample ID	
Station Name		Site ID	
Station Location		Date Collected	
Station Number		Field Crew ID	

Taxonomist Name_____
Date 1st Organism Identified in Sample:_____ **QC Check?** Y / N

TSN (Use # in UniqueIdentifier from taxa list provided by EPA)	Taxon	Distinct (Y/N)	Counts of Organisms in the Taxon:				Cumulative Number of Organisms in Sample	Data Qualifier (Codes in Table 4.2)
			Total (any stage)	Larvae	Pupae	Adults		

Comments:

5.0 WHOLE BODY FISH PROCESSING AND CONTAMINANT ANALYSIS

This chapter describes fish processing and analysis requirements for whole body fish samples. The purpose is to determine concentrations of contaminants in fish samples collected in the 2015 NCCA and related studies. The laboratory shall perform analysis to determine the lipid content, concentrations of metals, mercury, pesticides, and PCBs found in fish within coastal waters and Great Lakes. EPA also may require the national contract laboratory to analyze the samples for PAHs; however, EPA will not require the State laboratories to analyze for them.

At each sampling site, the Field Operations Manual (FOM) instructs the crews to collect five fish of the same species (or 10 sea urchins of any species) and similar size for each sample. The crew, or EPA's batch laboratory, then ships the fish specimens on dry ice to the laboratory.

In the following discussion, Sections 5.1, 5.2, and 5.3 summarize the procedure; health and safety concerns; and definitions and required resources. Section 5.4 provides the steps for acknowledging sample receipt. Section 5.5 provides the steps for creating whole fish composites. Sections 5.6 – 5.7 provide the minimum requirements that the laboratory must meet in performing the contaminant analyses and the required data elements. Section 5.8 describes EPA's external review of laboratory operations and other quality measures. Section 5.9 identifies references used in developing the procedure.

5.1 Summary of the Procedure

This chapter describes the fish processing and contaminant determination of whole fish samples collected for EPA's 2015 National Coastal Condition Assessment (NCCA). To ensure consistent and uncontaminated fish preparation activities across all samples, it is important that all NCCA participating laboratories adhere to the fish preparation procedures described in Section 5.5. The procedure is an adaption of instructions developed for fish tissue preparation for the National Rivers and Streams Assessment. As described in Section 5.6 the laboratory may choose to use any method that meets EPA's specifications for contamination measurements unless contractually bound to use specific methods (note, those methods must still meet EPA's specifications for contamination measurements)..

5.2 Health and Safety Warnings

The laboratory must require its staff to abide by appropriate health and safety precautions. In addition to the laboratory's usual requirements such as a Chemical Hygiene Plan, the laboratory must adhere to the following health and safety procedures:

1. Laboratory facilities must properly store and dispose of solutions of weak acid.

2. Laboratory personnel must wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear, gloves).

3. When working with potential hazardous chemicals (e.g., weak acid), laboratory personnel must avoid inhalation, skin contact, eye contact, or ingestion. Laboratory personnel must avoid contacting skin and mucous membranes with acid. If skin contact occurs, remove clothing immediately. Wash and rinse the affected skin areas thoroughly with large amounts of water.

4. When operating grinding equipment, the laboratory personnel must exercise caution.

5.3 Definitions and Required Resources (Personnel, Laboratories, and Equipment)

This section provides definitions and required resources for using the procedure.

5.3.1 Definitions

The procedure uses the following terms:

Detection Limit is the minimum concentration at which the analyte can be *detected* with confidence. In other words, the outcome can be reported with confidence that it is greater than zero (i.e., present in the sample). Also see "Sample-Specific Detection Limit."

Duplicates are defined as two aliquots of the same sample which are analyzed separately using identical procedures. The results are used to evaluate the precision of the laboratory analyses.

Fish Composite: Each composite consists of all parts of the fish including the head, skin, internal organs, muscle, and bones. For sea urchins, it includes only the gonad tissue because it is essentially the only tissue present. Unless otherwise specified, references to "fish" include "sea urchins." With the exception of sea urchins, NCCA does not provide support for analyses of any other invertebrates such as crustacean (e.g., lobster, crabs).

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management System (NARS IM): The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. Freshwater and coastal samples will be collected during the field stage of NCCA.

Non-routine sample: A non-routine sample is any sample that does not meet the definition of a routine sample. See Section 5.5.1 for more information.

Percent Recovery: Recovery is measured by comparing the concentrations of a sample split into two parts; and one part is spiked with a known concentration value. C_s is the

concentration measured in the spiked part; *C* is the concentration measured in the unspiked part; and *s* is the known concentration amount for the spike. The following equation is used to calculate the percent recovery:

$$\%Rs = \frac{C_s - C}{s} \times 100$$

Relative Standard Deviation (RSD): The precision at each concentration is reported in terms of the RSD. To calculate the RSD, first calculate the standard deviation, *S*, as follows:

$$S = \left[\frac{1}{n-1}\sum_{k=1}^{n}(C_s - \bar{C})^2\right]^{1/2}$$

where *n* is the number of replicate samples, *C*, is the concentration measure for the kth sample, and $\bar{C}$ is the average concentration of the replicate samples. Then, RSD is calculated as:

$$RSD = \left|\frac{S}{\bar{C}}\right| \times 100$$

Reporting Limit: A reporting limit is the point at which the measured value of the analyte can be reported with confidence.

Routine sample: A routine composite sample consists of individual adult fish of a single species that meet EPA's length requirement (Length of smallest fish in the composite must be at least 75% of the length of the longest fish),, and sufficient number of fish to meet target mass of 300 grams. See Section 5.5.1 for more information.

Sample-Specific Detection Limit: Most samples will have a sample-specific detection equal to the method's detection limit. For diluted samples, the sample-specific detection limit will be the product of the method's detection limit and the dilution factor. Typical values for the dilution factors will be 10 or 100.

Spiked Sample: See Percent Recovery definition for purpose of spiked samples.

TOCOR: Task Order Contracting Officer's Representative is EPA's contact person for laboratories under contract to EPA.

5.3.2 General Requirements for Laboratories

Competency: To demonstrate its competency, the laboratory shall provide analyte and matrix specific information to EPA. EPA will accept one or more of the following as a demonstration of competency:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.

- Documentation detailing the competency of the organization, including professional certifications for fish-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.

Also, the lab must provide a demonstration of competency with fish samples in achieving the method detection limits, accuracy, and precision targets.

Quality assurance and quality control requirements.
To demonstrate its expertise in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).

To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

5.3.3 Personnel

The procedure refers to the following personnel:

Laboratory Technician: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual.

External QC Coordinator is an EPA staff person who is responsible for selecting and managing the "**QC contractor**." To eliminate the appearance of any inherent bias, the QC contractor must be dedicated to QA/QC functions, and thus, must not be a primary laboratory or a field sampling contractor for NCCA. The QC contractor is responsible for complying with instructions from the External QC Coordinator; coordinating and paying for shipments of the performance samples to participating laboratories; comparing immunoassay results from the laboratories; and preparing brief summary reports.

5.3.4 Equipment/Materials

The procedures require the following equipment and information:

- Scale
- Powder-free nitrile gloves
- Tape measure
- 5% nitric acid
- Deionized water (DI water)
- Grinding equipment
- Glass containers
- Jars

5.4 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery. The laboratory must inspect the samples promptly on receipt. As samples arrive, the laboratory must:

1. Log the samples into the National Aquatic Resource Survey Information Management system (NARS-IM) within 24 clock hours. Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Check that each shipping container has arrived undamaged. Check the temperature of one of the samples in the cooler using a thermometer that reads to at least -20 ºC (i.e., the expected temperature of frozen samples), or an infra-red (IR) temperature "gun" and record the reading. Record the condition and temperature of the sample in the database using the codes in Table 5.1.

3. Compare the information on the label on each individual fish specimen to the sample tracking form for each composite and verify that each specimen was included in the shipment and is properly wrapped and labeled. The crew labels each fish specimen using the sample identification code and appends a specimen identification code. For example, if the sample number is "NCCA15-1111," then the crew might label specimen "A" as "NCCA15-1111.A." Record the number of fish in each sample.

4. Weigh each sample (i.e., all fish specimens collectively), record the weight in the database, and confirm that the sample meets the weight requirements of 140 grams (g) for a routine sample. If the sample weight is less than the required minimum, contact EPA for instructions, which are likely to involve preparing fewer aliquots for possibly fewer types of analyses than originally intended (e.g., perhaps EPA might eliminate the pesticides analysis for the sample).

5. Verify that all required data elements, per Table 5.1, have been recorded. If any elements are missing, then enter them into the database.

6. Transfer the samples to the freezer for long-term storage. Except during processing and analysis stages, the samples must be stored frozen to less than or equal -20 °C.

7. Notify the EPA immediately about any problems involving sample integrity, conformity, or inconsistencies as soon as possible following sample receipt and inspection.

Table 5.1 Whole Body Fish Login: Required Data Elements

Variable	Type	Description
SITE_ID	Character	Site identification code
SAMPLE	Character	Sample number
DATE_COLLECT	Date	Date that the field crew collected the sample

Variable	Type	Description		
	ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory (fish should be frozen).	
	NUMBER_FISH	Numeric	Number of fish in the sample	
	SAMPLE_WT	Numeric	Total weight of sample (all fish)	
	CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
			Flag	Definition
			OK	Sample is in good condition
			C	Sample wrapping is cracked
			L	Sample or container is leaking
			ML	Sample label is missing
			NF	Sample is not at proper temperature
			Q	Other quality concerns, not identified above
	COND_COMMENT	Character	Explanation for Q FLAG (if needed)	

5.5 Whole Fish Preparation and Homogenization Procedures

This section describes the whole fish preparation and homogenization procedures. As described in Section 5.5.1, if a laboratory determines that a sample is non-routine, the laboratory contacts the EPA HQ NCCA Laboratory Review Coordinator (Chapter 2 provides contact information) for additional instructions before continuing with the compositing and homogenization procedures in Section 5.5.2. Section 5.5.3 describes rigorous equipment cleaning and rinsate collection steps used before the compositing and homogenization steps in Section 5.5.4.

5.5.1 Sample Classification: Routine or Non-Routine

Each sample is either a "routine" composite sample, or a "non-routine" composite sample, based on the following definitions:

- *Routine sample* – A routine composite sample consists of individual adult fish of a single species that meet EPA's length and other requirements. For example, the species must be one of the target species identified in Appendix B of this LOM. The laboratory homogenizes the fish to prepare one composite sample.
- *Non-routine sample* – A non-routine sample is any sample that does not meet the definition of a routine sample. When field crews collect non-routine samples, depending on the circumstances, EPA will provide instructions for processing, or possibly destroying, the non-routine samples. These instructions also may include discarding some of the fish in the composite sample based on size before proceeding with homogenizing. For non-routine composites, the laboratory homogenizes only the designated specimens, i.e., those that EPA identifies by specimen number.

Note: Non-routine samples do not include samples from an incorrect sampling location, an unnecessary duplicate sample, or inappropriate fish species. EPA does not plan on using these "invalid" samples, so it is imperative that the sample preparation laboratory not process any sample without specific instructions from EPA. Therefore, laboratories shall retain such samples in frozen storage until EPA determines the appropriate course of action, which may include processing the sample. If the status of any composite sample in the instructions is not clear, the laboratory must contact EPA and wait for clarification.

5.5.2 Fish Examination and Preparation

This section describes the steps for fish examination and preparation.

1. Put on powder-free nitrile gloves (if not already gloved) before unpacking individual fish specimens. For sea urchins, wear thick rubber gloves to provide protection from the urchin spines. As samples are unpacked and unwrapped, inspect each fish carefully for any damage (e.g., tears in the skin or punctures in the gut). Document any damage in comments per Table 5.2.

2. The field crews measured the total length of each fish specimen in the field and recorded those lengths on the sample tracking form. Because of the importance of length measurements, EPA requires laboratories to perform a second series of measurements of the length for each fish. Because it may be difficult to reproduce the field measurements of fish length when the specimens are still partially frozen, begin processing the specimens in the following steps:
    a. Lay them out in order by specimen number (e.g., the portion of the sample ID after the decimal point)
    b. Allow them to partially thaw to the point that each specimen can be laid relatively flat.
    c. Using the length data on the sample tracking form (or the relative length order data in the fish sample processing instructions spreadsheet), confirm that the specimen ID for the longest specimen recorded on the tracking form is the same as the specimen ID on the label of the longest specimen. Repeat this relative length comparison for each of the other specimen IDs to ensure that the length orders based on the recorded lengths in the sample tracking form are consistent with the specimen IDs on the individual fish labels. This check is important for confirming that the field crews attached the correct label to each fish in the composite sample.
    d. Record the required data elements per Table 5.2 for the length of each species.

8. Weigh each fish to the nearest gram (wet weight) prior to any sample processing. In the database, record the required weight data elements per Table 5.2 for each specimen.

9. Identify and record the species of each fish specimen. Confirm that the species is one of the target species listed in Appendix B of this LOM.

10. Determine if the sample is routine or non-routine (per classification definitions in Section 5.5.1) and record its classification and any applicable fish code from Table 5.3. Return

any non-routine sample to the freezer and contact the EPA HQ NCCA Laboratory Review Coordinator for processing instructions (see Chapter 2 for contact information).

11. Verify that all required data elements, per Tables 5.2 and 5.3, have been recorded. If any elements are missing, then enter them into the database.

12. Rinse each fish with deionized water and remove any adhering slime as a precautionary measure to treat for possible contamination from sample handling in the field. Use HDPE wash bottles for rinsing fish and for cleaning homogenization equipment and utensils. Do **NOT** use Teflon® wash bottles for these procedures.

13. Return to freezer for storage until ready to homogenize the sample. If the laboratory intends to proceed directly to homogenization, then allow the sample to partially thaw while cleaning the equipment as described in the next section.

Table 5.2 Whole Body Fish: Data Elements for Each Fish Specimen

Variable	Type	Description
SITE_ID	Character	Site identification code
SAMPLE	Character	Sample number
SPECIMEN_ID	Character	Identification code assigned to a single fish
SPECIES	Character	Species of fish
FISH_WT	Numeric	Weight of fish
WT_UNIT	Character	Units of fish weight (kg, lb)
FISH_LEN	Numeric	Length of fish
LEN_UNIT	Character	Units of fish length (cm, in)
COMMENT	Character	Comment about condition of fish or other observations

Table 5.3 Whole Body Fish: Data Elements from Examination of Each Sample

Variable	Type	Description		
SITE_ID	Character	Site identification code		
SAMPLE	Character	Sample number		
SAMPLE_CLASS	Character	Sample classification: Routine or Non-routine		
FISH CODE	Character	Codes describing any deviations from the FOM criteria for fish collection for each sample		
		Flag	Definition	
		SP	Not all specimens are of the same species	
		LE	Not all specimens lengths are within 75% of longest fish	
		NS	Specimen number is fewer than minimum of 5 or greater than 20 maximum	

Variable	Type	Description		
			WT	Mass does not meet minimum of 140 grams *
			LL	Longest fish exceeds 400 mm maximum length
			LS	Shortest fish below 100 mm minimum length
			Q	Other quality concerns, not identified above

* Field crews are required to collect a minimum of 300 grams, but the minimum required for laboratory analyses is 140 grams.

5.5.3 Equipment Cleaning and Rinsate Collection

This section describes the rigorous cleaning required to protect against cross-contamination of samples. To verify that the cleaning procedures are effective, EPA requires the collection of rinsate samples as described below.

1. Before processing any sample, thoroughly clean all of the homogenization equipment. Disassemble the homogenization equipment (i.e., blender, grinder, or other device) and thoroughly **clean all surfaces and parts** that contact the sample. Similarly, **clean all knives, cutting boards, and other utensils used**. The cleaning steps are as follows:
    a. Wash with a detergent solution (phosphate- and scent-free) and warm tap water
    b. Rinse three times with warm tap water
    c. Rinse three times with deionized (DI) water
    d. Rinse with acetone
    e. Rinse three times with DI water
    f. Rinse with (not soak in) 5% nitric acid
    g. Rinse three times with DI water
    h. Allow the components to air dry
    i. Reassemble the homogenization equipment

2. Once per batch (i.e., once per maximum of 20 samples), collect rinsate samples for use in assessing any equipment contamination. To minimize the number of project samples that might be affected by cross contamination, collect the normal rinsate samples on the first day that samples in a batch of 20 are processed. Ideally (not required), the laboratory will vary the point at which the rinsates are collected on that first day over the course of the project (e.g., between the 1st and 2nd samples for one batch, the 2nd and 3rd samples for another batch, etc.). Prior to reassembling the homogenization equipment, use the following steps to prepare enough rinsate samples for the relevant QA/QC activities:
    a. Prepare each **hexane rinsate sample** by pouring a 100-mL portion of pesticide-grade hexane over all parts of homogenization equipment, including the cutting boards and knives, and collect it in a clean glass container. Place an additional 100-mL aliquot of clean hexane in a similar glass container for use as a solvent blank. Allow the solvent to evaporate from the equipment. Per QA/QC requirements, the laboratory will analyze the rinsate and solvent blank for the

Polychlorinated biphenyls (PCBs), pesticides, and Polycyclic Aromatic hydrocarbons (PAHs) selected for NCCA analysis.

b. Once the hexane has evaporated, prepare **each DI water rinsate** using 250 mL of DI water. Collect the DI water rinsate in a clean glass or HDPE container. Place a second aliquot of DI water in a separate similar clean container for use as a blank. Acidify these two samples to pH < 2 with nitric acid. Per QA/QC requirements, the laboratory will analyze the rinsate and blank samples for metals and mercury.
c. Store the rinsates and blanks at a cold, not freezing, temperature (<6 ºC).

5.5.4 Compositing and Homogenization Procedure

This section describes the steps for a "batch" homogenization method that uses the entire homogenized volume of all fish specimens to prepare the composite. In contrast to an "individual" method that would combine equal weights of tissue from each specimen, the batch homogenization method uses the complete specimens regardless of each individual specimen's proportion to one another. The steps are as follows:

1. Change gloves *between* samples. The technician may use the same gloves in handling all fish *within* a given sample.

2. Partially thaw samples for ease of grinding during homogenization.

3. For sea urchins, prepare the sea urchin for compositing by cracking open the shell of each sea urchin in the sample. From all of the sea urchins in the sample, extract and composite only the gonad tissue. (The gonad tissue is essentially the only tissue present in sea urchins.)

4. Process each sample using a size-appropriate homogenization apparatus (e.g., automatic grinder or high-speed blender). If difficulties arise with the samples sticking to equipment, try the following:
    a. Chill the grinder briefly with a few small pieces or pellets of dry ice.
    b. Add pellets of dry ice to the specimens as they enter the grinder.

5. Mix the specimens thoroughly until completely homogenized as evidenced by a final composite sample of soupy composition with uniform color and texture. Visible chunks or pieces of skin, bone, or tissue (e.g., liver tissue has red bits) will hinder extraction and digestion and, therefore, are NOT acceptable.

6. Grind the sample a second time, using the same grinding equipment. It is not necessary to clean the grinding equipment between grinding cycles of the same sample. This second grinding should proceed more quickly. The final sample must have a soupy composition with uniform color and texture. If there are obvious differences in color or texture, grind the entire sample a third time.

7. Prepare the sample aliquots for each type of analysis (e.g., mercury, PCBs) and place any remaining sample materials in a separate jar. Table 5.4 provides target mass weights needed for each type of analysis. When filling jars, leave sufficient space, at least 20%,

at the top of each jar to allow for expansion of the tissue as it freezes. *Jars filled beyond 80% capacity may break when freezing.* Wipe off the outside of the jars to remove any residue or moisture. Label each container and place inside one heavy-weight food-grade self-sealing plastic freezer bag to avoid sample loss due to breakage. Freeze the tissue aliquots at -20 ºC, and maintain samples in the freezer until analysis.

8. For one sample in every batch (same batch as specified for the rinsate samples collected in Section 5.5.3), the laboratory conducts triplicate analyses of the lipid content to confirm that the grinding has resulted in an homogeneous sample. As with the collection of rinsate samples, the laboratory performs the homogeneity testing on the first day on which samples in a batch of 20 are processed. However, the sample chosen for homogeneity testing must be one that yields enough tissue mass to support the added mass needed for triplicate lipid aliquots (15 to 30 g).

    a. The laboratory selects one sample processed on the first day of every batch that will provide well over 140 g of total tissue mass.

    b. From that sample, place three 5- to 10-g aliquots in clean glass or plastic containers of suitable size and label as appropriate.

    c. Calculate the mean lipid content (in percent), the standard deviation (SD), and the relative standard deviation (RSD) as follows:

$$\text{mean } \%\,\text{lipids} = \frac{\sum_{i=1}^{3} (\%\,\text{lipids})_i}{3}$$

$$\text{SD} = \sqrt{\frac{\sum_{i=1}^{3} (\%\,\text{lipids}_i - \text{mean lipids})^2}{2}}$$

$$\text{RSD} = \frac{\text{SD}}{\text{mean}}$$

    d. If the RSD of the triplicate results is:
        - Less than or equal to the QC criterion, then the homogenization effort is judged to be sufficient for all samples in that QC batch.
        - Otherwise, corrective action consists of regrinding all of the aliquots from each composite sample in the affected batch until meeting the QC criterion. This may entail retrieving all sample aliquots (see Table 5.4) from the freezer, allowing them to partially thaw, homogenizing them again, determining new lipids results, and performing a new homogenization QC determination. New sample containers are required for any rehomogenized samples. Also, follow the steps in Section 5.5.3 for cleaning the equipment between each composite sample in rehomogenizing the samples.

e. For this sample analyzed in triplicate, record the lipid content measured in the first analysis.

9. Before homogenizing the next sample, clean the **grinding equipment and all other sample preparation equipment** using the procedures described in Section 5.5.3.

Table 5.4. Whole Body Fish: Initial Aliquot Requirements

Analysis	Target Mass	Sample Jar Requirements
Mercury	5 - 10 g	50-mL HDPE straight-sided jar with **foil-lined lid**, or conical HDPE tube with snap top
Metals other than mercury	5 - 10 g	50-mL HDPE straight-sided jar with **foil-lined lid**, or conical HDPE tube with snap top
PCBs	30 - 35 g	125-mL straight-sided amber or clear glass jar with PTFE-lined lid
Pesticides	30 - 35 g	125-mL straight-sided amber or clear glass jar with PTFE-lined lid
PAHs (only by EPA request)	30 – 35 g	125-mL straight-sided amber or clear glass jar with PTFE-lined lid
Lipids	10 - 15 g	Laboratory's choice, as this aliquot will be used in-house to determine the lipid content of the sample
Maximum*	140 g	

*In the event that insufficient fish tissue mass exists to prepare the required number of aliquots, contact EPA for instructions.

5.6 Contaminant Analysis: Requirements

The laboratory shall perform analysis of the homogenized composites to determine the lipid content, concentrations of metals, mercury, pesticides, and PCBs. EPA also may require the national contract laboratory to analyze the samples for PAHs; however, EPA will not require the State laboratories to analyze for them. With the exception of sea urchins, NCCA does not provide support for analyses of any other invertebrates such as crustaceans (e.g., lobster, crabs).

After preparing the fish composites as described in Section 5.5, laboratories may choose to use any analysis method, including those in Table 5.5, that measures contaminants to the levels of the method detection limits identified in Table 5.6. In addition, the method must meet the target precision of 30% and the target accuracy as follows:

- Metals: 20%
- Organics (PCBs, pesticides, and PAHs): 35%

The laboratory must store the fish samples frozen at a maximum of -20° C and complete the analyses within one year.[7]

Table 5.5 Whole Body Fish: Analytical Methods

Analysis	Extraction	Methods that Meet the QA/QC Requirements (any method that meets the QA/QC requirements is acceptable)
Metals (except Mercury)	Any method using microwave assisted digestion[8]	EPA Method 6020A[9]
Mercury		EPA Method 245[10]
PCBs, Pesticides, PAHs	EPA Method 3540C[11]	EPA Method 8270[12]
Percent Lipids	Any method using hexane	EPA Method 9071B[13]

Table 5.6 Whole Body Fish: Lipids and Required Contaminants

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
LIPID	% Wet Weight	% LIPID			
METAL	µg/wet g (mg/L)	Aluminum	7429-90-5		10.0
		Arsenic	7440-38-2		2.0
		Cadmium	7440-43-9		0.2
		Chromium	7440-47-3		0.1
		Copper	7440-50-8		5.0
		Iron	7439-89-6		50.0
		Lead	7439-92-1		0.1

[7] NCCA allows for a 1-year holding time because of the sheer volume of sample collected in a short amount of time. Generally, EPA recommends different holding times, see for example Appendix J "Recommended procedures for preparing whole fish composite homogenate samples" in *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 (Fish Sampling and Analysis)*, 3rd Edition, 2000. EPA #823-B-00-007. Retrieved from http://water.epa.gov/scitech/swguidance/fishshellfish/techguidance/risk/upload/2009_04_23_fish_advice_volume1_v1cover.pdf.

[8] For example, see Method 3150A "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils," retrieved from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf.

[9] For example, Method 6020A "Inductively Coupled Plasma-Mass Spectrometry" retrieved from http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/6020a.pdf.

[10] For example, Method 245.7 "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, Revision 2.0" (EPA-821-R-05-001, February 2005), retrieved from http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_07_10_methods_method_245_7.pdf.

[11] For example, see Method 3540C "Soxhlet Extraction" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3540c.pdf.

[12] For example, Method 8270D "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) retrieved from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/8270d.

[13] Method 9171B "n-Hexane Extractable Material (HEM) for Sludge, Sediment, And Solid Samples," retrieved from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9071b.pdf.

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
		Mercury	7439-97-6		0.01
		Nickel	7440-02-0		0.5
		Selenium	7782-49-2		1.0
		Silver	7440-22-4		0.3
		Tin	7440-31-5		0.05
		Vanadium	7440-62-2		1.0
		Zinc	7440-66-6		50.0
PCB	ng/wet g (µg/L)	2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	2051-24-3	209	2.0
		2,4'-Dichlorobiphenyl	34883-43-7	8	2.0
		2,2',3,4',5,5',6-Heptachlorobiphenyl	35065-29-3	180	2.0
		2,2',3,3'4,4',5,6-Octachlorobiphenyl	52663-78-2	195	2.0
		2,2',3,4 ,5,5',6-Heptachlorobiphenyl	52663-68-0	187	2.0
		2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3	128	2.0
		2,2',3,3'4,4',5-Heptachlorobiphenyl	35065-30-6	170	2.0
		2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138	2.0
		2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153	2.0
		2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206	2.0
		2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	105	2.0
		2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101	2.0
		2,3',4,4',5-Pentachlorobiphenyl	31508-00-6	118	2.0
		2,3,3',4,6'-Pentachlorobiphenyl	38380-03-9	110	2.0
		3,3',4,4',5-Pentachlorobiphenyl	57465-28-8	126	2.0
		2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44	2.0
		3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	77	2.0
		2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52	2.0
		2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66	2.0
		2,2',5-Trichlorobiphenyl	37680-65-2	18	2.0
		2,4,4'-Trichlorobiphenyl	7012-37-5	28	2.0
PEST	ng/wet g (µg/L)	2,4'-DDD	53-19-0		2.0
		2,4'-DDE	3424-82-6		2.0
		2,4'-DDT	789-02-6		2.0
		4,4'-DDD	72-54-8		2.0
		4,4'-DDE	72-55-9		2.0
		4,4'-DDT	50-29-3		2.0
		Aldrin	309-00-2		2.0
		Alpha-BHC	319-84-6		2.0
		Beta-BHC	319-85-7		2.0
		Delta-BHC	319-86-8		2.0
		Alpha-Chlordane	5103-71-9		2.0
		Gamma-Chlordane	5566-34-7		2.0
		Dieldrin	60-57-1		2.0

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
		Endosulfan I	959-98-8		2.0
		Endosulfan II	33213-65-9		2.0
		Endosulfan Sulfate	1031-07-8		2.0
		Endrin	72-20-8		2.0
		Endrin Aldehyde	7421-93-4		2.0
		Endrin Ketone	53494-70-5		2.0
		Heptachlor	76-44-8		2.0
		Heptachlor Epoxide	1024-57-3		2.0
		Hexachlorobenzene	118-74-1		2.0
		Lindane	58-89-9		2.0
		Mirex	2385-85-5		2.0
		Cis-Nonachlor	5103-73-1		2.0
		Oxychlordane	26880-48-8		2.0
		Trans-Nonachlor	39765-80-5		2.0
PAHs*		Acenaphthene	83-32-9		2.0
		Acenaphthylene	208-96-8		2.0
		Anthracene	120-12-7		2.0
		Benz(a)anthracene	200-280-6		2.0
		Benzo(b)fluoranthene	205-99-2		2.0
		Benzo(k)fluoranthene	207-08-9		2.0
		Benzo(g,h,i)perylene	191-24-27-2		2.0
		Benzo(a)pyrene	50-32-8		2.0
		Benzo(e)pyrene	192-97-2		2.0
		Biphenyl	92-54-4		2.0
		Chrysene	218-01-9		2.0
		Dibenz(a,h)anthracene	53-70-3		2.0
		Dibenzothiophene	132-65-0		2.0
		2,6-Dimethylnaphthalene	581-42-0		2.0
		Fluoranthene	205-99-2		2.0
		Fluorene	86-73-7		2.0
		Indeno(1,2,3-c,d)pyrene	193-39-5		2.0
		1-Methylnaphthalene	90-12-0		2.0
		2-Methylnaphthalene	91-57-6		2.0
		1-Methylphenanthrene	832-69-9		2.0
		Naphthalene	91-20-3		2.0
		Perylene	198-55-0		2.0
		Phenanthrene	85-01-8		2.0
		Pyrene	129-00-0		2.0
		2,3,5-Trimethylnaphthalene	2245-38-7		2.0

* EPA also may require the national contract laboratory to analyze the samples for PAHs; however, EPA will not require the State laboratories to analyze for them.

5.7 Data Entry

Tables 5.1 (Section 5.4), 5.2 (Section 5.5), 5.3 (Section 5.5), and 5.7 (below) identify the required data elements that laboratories must provide to EPA, preferably in EPA's data template, available separately from EPA.

Table 5.7 Whole Body Fish: Data Elements for Each Sample

Variable	Type	Description		
SITE_ID	Character	Site identification code or type of QC sample (e.g., LAB BLANK)		
SAMPLE	Character	Sample number, LCS, QCCS, Blank, Matrix Spike, or Rinsate		
REPEAT	Numeric	Duplicate or Triplicate (otherwise blank)		
DATE_COLLECT	Date	Date that the field crew collected the sample		
	ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory (fish should be frozen).	
	NUMBER_FISH	Numeric	Number of fish in the sample	
	SAMPLE_WT	Numeric	Total weight of sample (all fish)	
	SAMPLE_CLASS	Character	Sample classification: Routine or Non-routine	
	CONDITION CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
			Flag	Definition
			OK	Sample is in good condition
			C	Sample wrapping is cracked
			L	Sample or wrapping is leaking
			ML	Sample label is missing
			NF	Sample is not at proper temperature
	COND_COMMENT	Character	Explanation for Q FLAG (if needed)	
	FISH CODE	Character	Codes describing any deviations from the criteria for fish collection for each sample	
			Flag	Definition
			SP	Not all specimens are of the same species
			LE	Not all specimens lengths are within 75% of longest fish
			NS	Specimen number is fewer than minimum of 5 or greater than 20 maximum
			WT	Mass does not meet minimum of 500 grams

Variable	Type	Description		
			LL	Longest fish exceeds 400 mm maximum length
			LS	Shortest fish below 100 mm minimum length
			Q	Other quality concerns, not identified above
PARAMETER	Character	Analyte name		
CAS_NO	Character	CAS Registry number corresponding to the analyte		
LABNAME	Character	Laboratory name (abbreviation)		
METHOD	Character	Laboratory method used		
ANALYST	Character	Last name or initials of person who performed the analysis		
REVIEWER	Character	Last name or initials of the person who provided a separate independent review of the data		
INSTRUMENT	Character	Identification of instrument used for the analysis – provide enough information to identify the particular instrument in the laboratory		
DATE PREPARED	Date	Date that the sample homogenization started		
DATE ANALYSIS	Date	Date that the sample analysis started		
QC_BATCH_LOT	Character	Unique laboratory quality control lot numbers assigned to the batch of samples. The lot number must associate each batch of field samples to the appropriate rinsates, laboratory control sample, matrix spike, laboratory duplicate, and method blank samples.		
HOLDING TIME	Y/N	Analysis performed within holding time		
MATRIX	Character	Fish		
MDL	Numeric	Lab method detection limit (based upon lab's historical data)		
LRL	Numeric	Lab reporting limit (based upon lab's historical data)		
DILUTION	Numeric	Dilution of sample (blank or 1 if no dilution)		
RECOVERY	Numeric	Only for appropriate QC samples		
RESULT	Numeric	Concentration value		
REASON	Character	Reason for qualification in RESULT_QUAL (usually blank)		
RESULT_QUAL	Character	Data qualifier (usually blank)		
UNIT	Character	Unit of measurement for RESULT, MDL, and RL		
QC_CODE	Character	Apply laboratory defined QC codes and describe in the comments field. Provide set of laboratory's code as part of the case narrative		
COMMENT	Character	Explain situation that created QC code, or any unusual aspects of the analysis		

5.8 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements.

5.8.1 Assistance Visits

Assistance visits are intended to familiarize EPA with actual procedures being implemented by different laboratories; and to ensure a clear and consistent understanding of procedures and activities by both EPA and the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter.

5.8.2 QC Samples

Once or twice during the performance period, the External QC Coordinator will provide one or two identical sets of QC samples to all participating laboratories. Each set will contain up to five QC samples. As determined by the External QC Coordinator, the QC samples may be synthetic; aliquots of additional samples collected at NCCA sites; or reference samples obtained from an organization such as the National Institute of Standards. Each laboratory will run the QC samples following the same procedures used for the other samples. The External QC Coordinator will compare the results to the expected value and determine consistency between laboratories (e.g., determine if one laboratory is consistently higher or lower than all others). Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any unique laboratory practices that might account for differences between the laboratory and others. The contractor shall analyze the external QC samples using the same procedures as those for the field samples.

5.8.3 Summary of QA/QC Requirements

QC protocols are an integral part of all analytical procedures to ensure that the results are reliable and the analytical stage of the measurement system is maintained in a state of statistical control. The laboratory must conduct QC analyses for each batch of samples. Each batch shall consist of no more than 20 samples. Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate measures such as laboratory control sample, matrix spike, laboratory duplicate, and method blank samples. Also, each laboratory QC samples (i.e., preparation and instrument blanks, laboratory control sample (LCS), spike/duplicate, etc.) must be give a unique sample identification. Table 5.8 provides a summary of the quality control requirements.

Table 5.8 Whole Body Fish: Quality control activities

Quality Control Activity	Description and Requirements	Corrective Action
Demonstrate competency for analyzing fish samples with the required methods	Demonstration of competency with fish samples in achieving the method detection limits. accuracy, and precision targets	EPA will not approve any laboratory for NCCA sample processing if the laboratory cannot demonstrate competency. In other words, EPA will select another laboratory that can demonstrate competency for its NCCA samples.

Quality Control Activity	Description and Requirements	Corrective Action
Check condition of sample when it arrives.	Sample issues, such as punctures or rips in wrapping; missing label; temperature; adherence to holding time requirements; sufficient volume for test. All samples should arrive at the laboratory in a frozen state.	Assign appropriate condition code identified in Table 5.1.
Store sample appropriately. While stored at the laboratory, the sample must be kept at a maximum temperature of -20° C.	Check the temperature of the freezer per laboratory's standard operating procedures.	Record temperature of sample upon arrival at the laboratory. If at any other time, samples are warmer than required, note temperature and duration in comment field.
Determine if all fish meet the criteria	Evaluate if the sample contains fish of the same species and are similar in size (within 75%), and provides enough material to run the analysis	Contact the EPA HQ NCCA Laboratory Review Coordinator* for a decision on fish selection and/or chemical analysis.
Analyze sample within holding time	The test must be completed within the holding time (i.e., 28 days for mercury; 6 months for other metals; and 1 year for all others). If the original test fails, then the retest also must be conducted within the holding time.	Perform test, but note reason for performing test outside holding time. EPA expects that the laboratory will exercise every effort to perform tests before the holding time expires.
Perform once at the start of each batch to evaluate the labeled compound recovery (LCR) in a Laboratory Control Sample (LCS). This tests the performance of the equipment.	Control limits for recovery cannot exceed 100±20%.	First, prepare and analyze one additional LCS. If the second blank meets the requirement, then no further action is required. If the second LCS fails, then determine and correct the problem before proceeding with any sample analyses.
Perform once at the start of each batch to evaluate the entire extraction and analysis process using a Method Blank	Control limits cannot exceed the laboratory reporting level (LRL).	First, prepare and analyze one additional blank. If the second blank meets the requirement, then no further action is required. If the second blank fails, then determine and correct the problem (e.g., homogenization, reagent contamination, instrument calibration, or contamination introduced during filtration) before proceeding with any sample analyses. Reestablish statistical

Quality Control Activity	Description and Requirements	Corrective Action
		control by analyzing three blank samples. Report values of all blanks analyzed.
Check calibration immediately before and immediately after the sample batch is run (abbreviated as QCCS for quality control check sample)	Results must be ±10% of each other or as specified in method criteria	If calibration fails before analysis, recalibrate and reanalyze QCCS until it passes. If check fails after all samples in the batch have been analyzed, verify the QCCS reading. If the QCCS reading fails a second time, then reanalyze all samples in the batch and report both sets of results. For the first run, include a data qualifier that indicates that the QCCS reading taken immediately following the first run failed. For the second run, include a data qualifier that indicates that it is the second set and whether the QCCS reading immediately following that second run passed. No sample is to be analyzed more than twice.
Evaluate rinsates for first sample in each batch. This evaluation is a surrogate for assessing cross-contamination.	Results must be below the LRL.	If first rinsate is above LRL, analyze rinsate from a second sample. If second rinsate sample also has results above the LRL, then assign a data qualifier to all samples in the batch for the parameters with results above the LRL in the rinsates. Also, improve procedures for cleaning all surfaces, knives, and homogenization equipment between samples.
Compare lipids in triplicate for the first sample in each batch. This evaluation is a surrogate for assessing homogenization.	Substitute the LRL for any value below the LRL before calculating the RSD. If the RSD of the triplicate results is ≤20%, then the homogenization effort is judged to be sufficient for all samples in the batch.	If the RSD could not be achieved, then regrind all samples in the batch one or more times as described in Section 5.5
Compare results of one laboratory duplicate sample or matrix spike duplicate sample for each batch	Results must be within the target precision goal in Table 5.8.1 (30% for all analytes).	If both results are below LRL, then conclude that the test has passed. Otherwise, prepare and analyze a split from different sample in the batch. If the second result is within the target precision goal (see Table 5.8.1) of the original sample, then

Quality Control Activity	Description and Requirements	Corrective Action
		report the data and findings for both QC samples. However, if the two results differ by more than the target precision goal, review precision of QCCS measurements for batch; check preparation of split sample; etc. and report evaluation and findings in the case narrative. Consult with the EPA HQ NCCA Laboratory Review Coordinator* to determine if reanalysis of the entire batch (at the laboratory's expense) is necessary. If no reanalysis is necessary, report and quantify all samples in batch. If reanalysis is necessary, then report all QC sample and the 2nd analysis of the batch. If the second set also is unacceptable, then assign a data code to each sample in the batch.
Compare results of one matrix spike sample per batch to evaluate performance in matrix	Evaluate performance after the first 3 batches. Ideally, control limits for recovery will not exceed the target accuracy goal (Table 5.8.1), but this may not be realistic for all parameters with this matrix.	If both results are below LRL, then conclude that the test has passed for the batch. Otherwise, if any results are not within the target accuracy goal for the 3 batches, within 2 working days, contact the EPA HQ NCCA Laboratory Review Coordinator* to discuss method performance and potential improvements. Continue to perform the test for every batch. Report the results from the original analysis, the matrix spike, matrix spike duplicate, and %recovery.
Maintain the required MDL identified in the Section 5.6	Evaluate for each sample	If MDL could not be achieved, then provide dilution factor or QC code and explanation in the comment field.
Use consistent units for QC samples and field samples	Verify that all units are provided in wet weight units and consistently within each indicator type as follows: Metals in µg/g or ppm. PCBs, pesticides, and PAHs in ng/g or µg/L.	If dry units are reported for any sample (QC or field), reanalyze the sample and report only the reanalysis results. If it is not possible to provide the results in wet units, then assign a QC code and describe the reason for dry

Quality Control Activity	Description and Requirements	Corrective Action
		units in the comments field of the database.
Maintain completeness	Completeness objective is 95% for all parameters.	Contact EPA HQ NCCA Laboratory Review Coordinator* immediately if issues affect laboratory's ability to meet completeness objective.

*Chapter 2 provides contact information for the EPA HQ NCCA Laboratory Review Coordinator. Laboratories under contract to EPA must contact the Task Order's Contracting Officer's Representative (TOCOR) instead of the Laboratory Review Coordinator.

5.9 Sample and Record Retention

The laboratory shall retain:

1. The sample materials, including vials, for a minimum of 3 years from the date the EPA publishes the final report. During this time, the laboratory shall freeze the materials. The laboratory shall periodically check the sample materials for degradation.

2. Original records, including laboratory notebooks and the reference library, for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

5.10 References

All references are from U.S. Environmental Protection Agency:

Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 (Fish Sampling and Analysis), 3rd Edition, 2000. Appendix J "Recommended procedures for preparing whole fish composite homogenate samples". EPA #823-B-00-007. Retrieved from http://water.epa.gov/scitech/swguidance/fishshellfish/techguidance/risk/upload/2009_04_23_fish_advice_volume1_v1cover.pdf.

Method 245.7 "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, Revision 2.0" (EPA-821-R-05-001, February 2005), retrieved from http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_07_10_methods_method_245_7.pdf.

Method 3150A "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils," retrieved from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf.

Method 6020A "Inductively Coupled Plasma-Mass Spectrometry" retrieved from http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/6020a.pdf.
Method 8270D "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) retrieved from Method 8270D "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry.

Method 9171B "n-Hexane Extractable Material (HEM) for Sludge, Sediment, And Solid Samples," retrieved from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9071b.pdf.

6.0 SEDIMENT CONTAMINANT, GRAIN SIZE, AND TOC ANALYSES

This chapter describes the analysis requirements for sediment samples. The purpose is to determine concentrations of contaminants, grain size, and total organic carbon (TOC) in sediment samples collected in the 2015 NCCA and related studies. The laboratory shall perform analysis to determine the moisture content, concentrations of metals, mercury, pesticides, and PCBs found in sediments in coastal waters and Great Lakes.

At each sampling site, the Field Operations Manual (FOM) instructs the crews to collect sediment samples. The field crew then ships the samples on wet ice to either its own state laboratory or EPA's batching laboratory. Once the samples arrive, the laboratory will freeze the samples for the contaminant analyses and refrigerate the grain size and TOC samples.

In the following discussion, Sections 6.1, 6.2, and 6.3 summarize the procedure; health and safety concerns; and definitions and required resources. Section 6.4 provides the steps for acknowledging sample receipt. Sections 6.5 – 6.6 provide the minimum requirements that the laboratory must meet in performing the contaminant analyses and the required data elements. Section 6.7 describes EPA's external review of laboratory operations and other quality measures. Section 6.8 identifies references used in developing the procedure.

6.1 Summary of the Procedure

This chapter describes the contaminant, grain size, and TOC determination of sediment samples collected for EPA's 2015 National Coastal Condition Assessment (NCCA). As described in Section 6.5, unless otherwise contractually bound by other requirements, the laboratory may choose to use any method that meets EPA's specifications for contamination measurements.

6.2 Health and Safety Warnings

The laboratory must require its staff to abide by appropriate health and safety precautions. In addition to the laboratory's usual requirements such as a Chemical Hygiene Plan, the laboratory must adhere to the following health and safety procedures:

1. Laboratory facilities must properly store and dispose of solutions of weak acid.

2. Laboratory personnel must wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear, gloves).

3. When working with potential hazardous chemicals (e.g., weak acid), laboratory personnel must avoid inhalation, skin contact, eye contact, or ingestion. Laboratory personnel must avoid contacting skin and mucous membranes with acid. If skin contact occurs, remove clothing immediately. Wash and rinse the affected skin areas thoroughly with large amounts of water.

6.3 Definitions and Required Resources (Personnel, Laboratories, and Equipment)

This section provides definitions and required resources for using the procedure.

6.3.1 Definitions

The procedure uses the following terms:

Detection Limit is the minimum concentration at which the analyte can be *detected* with confidence. In other words, the outcome can be reported with confidence that it is greater than zero (i.e., present in the sample). Also see "Sample-Specific Detection Limit."

Duplicates are defined as two aliquots of the same sample which are analyzed separately using identical procedures. The results are used to evaluate the precision of the laboratory analyses.

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management System (NARS IM): The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. Freshwater and coastal samples will be collected during the field stage of NCCA.

Percent Recovery: Recovery is measured by comparing the concentrations of a sample split into two parts; and one part is spiked with a known concentration value. C_s is the concentration measured in the spiked part; C is the concentration measured in the unspiked part; and s is the known concentration amount for the spike. The following equation is used to calculate the percent recovery:

$$\%Rs = \frac{C_s - C}{s} \times 100$$

Relative Percent Difference (RPD): Relative percent difference compares the matrix spike (S) and the matrix spike duplicate (D) using the following equation:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Reporting Limit: A reporting limit is the point at which the measured value of the analyte can be reported with confidence.

Sample-Specific Detection Limit: Most samples will have a sample-specific detection equal to the method's detection limit. For diluted samples, the sample-specific detection limit will be the product of the method's detection limit and the dilution factor. Typical values for the dilution factors will be 10 or 100.

Spiked Sample: See Percent Recovery definition for purpose of spiked samples.

TOC: Total Organic Carbon

TOCOR: Task Order Contracting Officer's Representative is EPA's contact person for laboratories under contract to EPA.

6.3.2 General Requirements for Laboratories

Competency. To demonstrate its competency, the laboratory shall provide analyte and matrix specific information to EPA. EPA will accept one or more of the following as a demonstration of competency:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
- Documentation detailing the competency of the organization, including professional certifications for water-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.
- Demonstration of competency with sediment samples in achieving the method detection limits, accuracy, and precision targets.

Quality assurance and quality control requirements.
To demonstrate its competency in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).

To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

6.3.3 Personnel

The procedure refers to the following personnel:

Laboratory Technician: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual.

External QC Coordinator is an EPA staff person who is responsible for selecting and managing the "**QC contractor**." To eliminate the appearance of any inherent bias, the QC contractor must be dedicated to QA/QC functions, and thus, must not be a primary laboratory or a field sampling contractor for NCCA. The QC contractor is responsible for complying with instructions from the External QC Coordinator; coordinating and paying for shipments of the performance samples to participating laboratories; comparing immunoassay results from the laboratories; and preparing brief summary reports.

6.3.4 Equipment/Materials

The analytical methods, selected by the laboratory, specify the required equipment.

6.4 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery. The laboratory must inspect the samples promptly on receipt. As samples arrive, the laboratory must:

1. Log the samples into the National Aquatic Resource Survey Information Management system (NARS-IM) within 24 clock hours. Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Check that each shipping container has arrived undamaged. Check the temperature of one of the samples in the cooler using a thermometer that reads from 21 ºC (i.e., room temperature) down to -20 ºC or lower (i.e., the expected temperature of frozen samples), or an infra-red (IR) temperature "gun" and record the reading. Field crews ship sediment samples on wet ice; the batch laboratory freezes the sample and ships with dry ice. Record the condition and temperature of the sample in the database using the codes in Table 6.1.

3. Verify that all required data elements, per Table 6.1, have been recorded. If any elements are missing, then enter them into the database.

4. Transfer the samples to the freezer for long-term storage. Except during processing and analysis stages, the samples must be stored frozen to less than or equal -20 °C.

5. Notify the EPA immediately about any problems involving sample integrity, conformity, or inconsistencies as soon as possible following sample receipt and inspection.

Table 6.1 Sediment Chemistry, Grain Size, and TOC Login: Required Data Elements

Variable	Type	Description	
SITE_ID	Character	Site identification code	
SAMPLE	Character	Sample number	
DATE_COLLECT	Date	Date that the field crew collected the sample	
	ANALYSIS_TYPE	Character	Contaminant, TOC, or GRAIN SIZE
	ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory
	CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control

Variable	Type	Description		
			Flag	Definition
			OK	Sample is in good condition
			C	Sample container is cracked
			L	Sample or container is leaking
			ML	Sample label is missing
			Q	Other quality concerns, not identified above
	COND_COMMENT	Character	Explanation for Q FLAG (if needed)	

6.5 Laboratory Analysis: Requirements

The laboratory shall perform analysis of the sediment samples to determine the moisture content, grain size, and concentrations of TOC, metals, mercury, pesticides, PAHs, and PCBs.

Table 6.2 identifies the storage requirements. Laboratories may choose to use any analysis method, including those in Table 6.2, which measures the parameters to the levels of the method detection limits identified in Table 6.3. In addition, the contaminant analysis method must meet the precision and accuracy targets of 30% and 20%, respectively. For each batch of contaminant samples, precision is assessed using the relative percent difference (RPD) between the matrix spike (MS) and the matrix spike duplicate (MSD); and accuracy by the average percent recovery (%Rs) between the matrix spike and matrix spike duplicate. Section 6.3.1 provides the equations used to calculate the RPD and %Rs. The precision and accuracy targets for each batch of TOC are both 10% and determined by the RPD of one sample and its duplicate (for precision) and the analysis of Certified Reference Material (CRM; for accuracy). The grain size target precision is 10% as determined using a Laboratory Control Sample (LCS) (accuracy is not applicable).

Table 6.2 Sediment Chemistry, Grain Size, and TOC: Analytical Methods

Storage Requirements	Type	Methods that Meet the QA/QC Requirements (any method that meets the QA/QC requirements is acceptable)
Freeze samples with maximum of -20° C	Metals (except Mercury)	Extraction: EPA Method 3051A Analysis: EPA Method 6020A[14]

14 For example, see:

- Method 3051A "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, And Oils" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf; and
- Method 6020A "Inductively Coupled Plasma-Mass Spectrometry" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/6020a.pdf.

	Mercury	EPA Method 245.7[15]
	PCBs, Pesticides, PAHs	Extraction: EPA Method 3540C Analysis: EPA Method 8270D[16]
	TOC	Lloyd Kahn Method[17]
Refrigerate at 4° C (do not freeze)	Grain Size	Any method that reports the determination as %silt and meets QA/QC requirements

Table 6.3 Sediment Chemistry, Grain Size, and TOC: Required Parameters

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
	% sand and % silt/clay	Grain Size	not applicable		0.05%
	mg/kg	Total Organic Carbon (TOC)	not applicable		0.01%
METAL	dry weight µg/g (ppm)	Aluminum	7429-90-5		1500
		Antimony	7440-36-0		0.2
		Arsenic	7440-38-2		1.5
		Cadmium	7440-43-9		0.05
		Chromium	7440-47-3		5.0
		Copper	7440-50-8		5.0
		Iron	7439-89-6		500
		Lead	7439-92-1		1.0
		Manganese	7439-96-5		1.0
		Mercury	7439-97-6		0.01
		Nickel	7440-02-0		1.0
		Selenium	7782-49-2		0.1
		Silver	7440-22-4		0.3
		Tin	7440-31-5		0.1
		Vanadium	7440-62-2		1.0
		Zinc	7440-66-6		2.0
PCB	dry weight ng/g (ppb)	2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl	2051-24-3	209	1.0
		2,4'-Dichlorobiphenyl	34883-43-7	8	1.0
		2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	170	1.0

15 For example, see Method 245.7 "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, Revision 2.0" (EPA-821-R-05-001, February 2005), retrieved June 27, 2014 from http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_07_10_methods_method_245_7.pdf.

16 For example, see:

- Method 3540C "Soxhlet Extraction" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3540c.pdf; and
- Method 8270D "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/8270d.pdf.

17 For example, the "Lloyd Kahn Method" developed by Lloyd Kahn at EPA Region II and retrieved from www.nj.gov/dep/srp/guidance/rs/lloydkahn.pdf.

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
		2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	187	1.0
		2,2',3,4',5,5',6-Heptachlorobiphenyl	35065-29-3	180	1.0
		2,2',3,3',4,4'-Hexachlorobiphenyl	38380-07-3	128	1.0
		2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	138	1.0
		2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	153	1.0
		2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	206	1.0
		2,2',3,3',4,4',5,6-Octachlorobiphenyl	52663-78-2	195	1.0
		2,3,3',4,4'-Pentachlorobiphenyl	32598-14-4	105	1.0
		2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	101	1.0
		2,3',4,4',5-Pentachlorobiphenyl	31508-00-6	118	1.0
		2,3,3',4,6'-Pentachlorobiphenyl	38380-03-9	110	1.0
		3,3',4,4',5-Pentachlorobiphenyl	57465-28-8	126	1.0
		2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	44	1.0
		3,3',4,4'-Tetrachlorobiphenyl	32598-13-3	77	1.0
		2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	52	1.0
		2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	66	1.0
		2,2',5-Trichlorobiphenyl	37680-65-2	18	1.0
		2,4,4'-Trichlorobiphenyl	7012-37-5	28	1.0
PEST	dry weight ng/g (ppb)	2,4'-DDD	53-19-0		1.0
		2,4'-DDE	3424-82-6		1.0
		2,4'-DDT	789-02-6		1.0
		4,4'-DDD	72-54-8		1.0
		4,4'-DDE	72-55-9		1.0
		4,4'-DDT	50-29-3		1.0
		Aldrin	309-00-2		1.0
		Alpha-BHC	319-84-6		1.0
		Beta-BHC	319-85-7		1.0
		Delta-BHC	319-86-8		1.0
		Alpha-Chlordane	5103-71-9		1.0
		Gamma-Chlordane	5566-34-7		1.0
		Dieldrin	60-57-1		1.0
		Endosulfan I	959-98-8		1.0
		Endosulfan II	33213-65-9		1.0
		Endosulfan Sulfate	1031-07-8		1.0
		Endrin	72-20-8		1.0
		Endrin Aldehyde	7421-93-4		1.0
		Endrin Ketone	53494-70-5		1.0
		Heptachlor	76-44-8		1.0
		Heptachlor Epoxide	1024-57-3		1.0
		Hexachlorobenzene	118-74-1		1.0
		Lindane	58-89-9		1.0
		Mirex	2385-85-5		1.0
		Cis-Nonachlor	5103-73-1		1.0

Type	UNITS	Parameter	CAS Number	PCB Number (where applicable)	MDL Target
		Oxychlordane	26880-48-8		1.0
		Trans-Nonachlor	39765-80-5		1.0
PAHs	dry weight ng/g (ppb)	Acenaphthene	83-32-9		10
		Acenaphthylene	208-96-8		10
		Anthracene	120-12-7		10
		Benz(a)anthracene	200-280-6		10
		Benzo(b)fluoranthene	205-99-2		10
		Benzo(k)fluoranthene	207-08-9		10
		Benzo(g,h,i)perylene	191-24-27-2		10
		Benzo(a)pyrene	50-32-8		10
		Benzo(e)pyrene	192-9		10
		Biphenyl	92-54-4		10
		Chrysene	218-01-9		10
		Dibenz(a,h)anthracene	53-70-3		10
		Dibenzothiophene	132-65-0		10
		2,6-Dimethylnaphthalene	581-42-0		10
		Fluoranthene	205-99-2		10
		Fluorene	86-73-7		10
		Indeno(1,2,3-c,d)pyrene	193-39-5		10
		1-Methylnaphthalene	90-12-0		10
		2-Methylnaphthalene	91-57-6		10
		1-Methylphenanthrene	832-69-9		10
		Naphthalene	91-20-3		10
		Perylene	198-55-0		10
		Phenanthrene	85-01-8		10
		Pyrene	129-00-0		10
		2,3,5-Trimethylnaphthalene	2245-38-7		10

6.6 Data Entry

Table 6.4 identifies the required data elements that laboratories must provide to EPA, preferably in EPA's data template, available separately from EPA. If the laboratory applies its own QC codes, the data transmittal should define the codes.

Table 6.4 Sediment Chemistry, Grain Size, and TOC: Data Elements for Each Sample

Variable	Type	Description
SITE_ID	Character	Site identification code or type of QC sample (e.g., LAB BLANK)
SAMPLE	Character	Sample number, LCS, QCCS, Blank, Matrix Spike, or CRM
ANALYSIS_TYPE	Character	Contaminant, TOC, or GRAIN SIZE
REPEAT	Numeric	Duplicate
DATE_COLLECT	Date	Date that the field crew collected the sample

Variable	Type	Description	
ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory	
CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		VT	Volume not sufficient for testing
		VR	Volume not sufficient for a retest, if required
		Q	Other quality concerns, not identified above
COND_COMMENT	Character	Explanation for Q FLAG (if needed)	
PARAMETER	Character	Analyte name	
CAS_NO	Character	CAS Registry number	
LABNAME	Character	Laboratory name (abbreviation)	
METHOD	Character	Laboratory method used	
ANALYST	Character	Last name or initials of person who performed the analysis	
REVIEWER	Character	Last name or initials of the person who provided a separate independent review of the data	
INSTRUMENT	Character	Identification of instrument used for the analysis – provide enough information to identify the particular instrument in the laboratory	
DATE PROCESSED	Date	Date that the analysis started	
QC_BATCH_LOT	Character	Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate laboratory control sample, matrix spike, laboratory duplicate, method blank, and CRM samples.	
HOLDING TIME	Y/N	Analysis performed within holding time	
MATRIX	Character	Sediment (Water also is a permissible value if the laboratory analyzes a very liquid sediment sample as water)	
MDL	Numeric	Lab method detection limit (based upon lab's historical data)	
LRL	Numeric	Lab reporting limit (based upon lab's historical data)	
MOISTURE	Numeric	Moisture in the sample (value used by lab to convert wet units to dry)	
MOIST_UNIT	Character	Unit used to report moisture (% or mg/kg)	
DILUTION	Numeric	Dilution of sample (blank or 1 if no dilution)	
RECOVERY	Numeric	Only for appropriate QC samples	
RESULT	Numeric	Concentration value	
REASON	Character	Reason for qualification in RESULT_QUAL (usually blank)	
RESULT_QUAL	Character	Data qualifier (usually blank)	
UNIT	Character	Unit of measurement for RESULT, MDL, and RL	
QC_CODE	Character	Apply laboratory defined QC codes and describe in the comments field. Provide set of laboratory's code as part of the case narrative	

Variable	Type	Description
COMMENT	Character	Explain situation that created QC code, or any unusual aspects of the analysis

6.7 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements.

6.7.1 Assistance Visits

Assistance visits are intended to familiarize EPA with actual procedures being implemented by different laboratories; and to ensure a clear and consistent understanding of procedures and activities by both EPA and the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter.

6.7.2 QC Samples

Once or twice during the performance period, the External QC Coordinator will provide one or two identical sets of QC samples to all participating laboratories. Each set will contain up to five QC samples. As determined by the External QC Coordinator, the QC samples may be synthetic; aliquots of additional samples collected at NCCA sites; or reference samples obtained from an organization such as the National Institute of Standards. Each laboratory will run the QC samples following the same procedures used for the other samples. The External QC Coordinator will compare the results to the expected value and determine consistency between laboratories (e.g., determine if one laboratory is consistently higher or lower than all others). Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any unique laboratory practices that might account for differences between the laboratory and others. The contractor shall analyze the external QC samples using the same procedures as those for the field samples.

6.7.3 Summary of QA/QC Requirements

QC protocols are an integral part of all analytical procedures to ensure that the results are reliable and the analytical stage of the measurement system is maintained in a state of statistical control. The laboratory must conduct QC analyses for each batch of samples. Each batch shall consist of no more than 20 samples. Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate measures such as laboratory control sample, matrix spike, laboratory duplicate, and method blank samples. Also, each laboratory QC samples (i.e., preparation and instrument blanks, laboratory control sample (LCS), spike/duplicate, etc.) must be given a unique sample identification. Table 6.5 provides a summary of the quality control requirements.

Table 6.5 Sediment Chemistry, Grain Size, and TOC: Quality control activities for samples

Activity	Evaluation	Corrective Action
Demonstrate competency for analyzing sediment samples to meet the performance measures	Demonstration of competency with sediment samples in achieving the method detection limits, accuracy, and precision targets.	EPA will not approve any laboratory for NCCA sample processing if the laboratory cannot demonstrate competency. In other words, EPA will select another laboratory that can demonstrate competency for its NCCA samples.
Check condition of sample when it arrives.	Sample issues such as cracked container; missing label; sufficient volume for test.	Assign appropriate condition code identified in Table 6.4.
Store sample appropriately. While stored at the laboratory, the sample must be kept at a temperature ≤-20° C except jars for grain analyses are refrigerated at 4°C.	Check the temperature of the refrigerator/freezer and refrigerator per laboratory's standard operating procedures.	Record temperature of sample upon arrival at the laboratory. If at any other time, samples are warmer than required, note temperature and duration in comment field. Data analyst will consider temperature deviations in evaluating the data. He/she will flag the deviations and determine whether the data appear to be affected and/or the data should be excluded from the analyses.
Analyze sample within holding time	The test must be completed within the holding time of 1 year. If the original test fails, then the retest also must be conducted within the holding time.	Perform test, but note reason for performing test outside holding time. EPA expects that the laboratory will exercise every effort to perform tests before the holding time expires.
Perform once at the start of each batch to evaluate the labeled compound recovery (LCR) in a Laboratory Control Sample (LCS). This tests the performance of the equipment.	Control limits for recovery cannot exceed 100±20%.	First, prepare and analyze one additional LCS. If the second blank meets the requirement, then no further action is required. If the second LCS fails, then determine and correct the problem before proceeding with any sample analyses.
Perform once at the start of each batch to evaluate the entire extraction and analysis process using a Method Blank	Control limits cannot exceed the laboratory reporting level (LRL).	First, prepare and analyze one additional blank. If the second blank meets the requirement, then no further action is required. If the second blank fails, then determine and correct the problem (e.g., contamination, instrument calibration) before proceeding with any sample analyses. Reestablish statistical

Activity	Evaluation	Corrective Action
		control by analyzing three blank samples. Report values of all blanks analyzed.
Check calibration immediately before and immediately after the sample batch (abbreviated as QCCS for quality control check sample)	Results must be ±10% of each other or as specified in method criteria	If calibration fails before analysis, recalibrate and reanalyze QCCS until it passes. If check fails after all samples the batch have been analyzed, verify the QCCS reading. If the QCCS reading fails a second time, then reanalyze all samples in the batch and report only the set of results associated with the acceptable QCCS reading. Also report all QCCS readings for the batch.
Compare results of one laboratory duplicate sample (for TOC) or matrix spike duplicate sample (for contaminants) for each batch (not required for grain size)	Results must be within the target precision goal in Section 6.5.	If both results are below LRL, then conclude that the test has passed. Otherwise, prepare and analyze a split from different sample in the batch. If the second result is within the target precision goal (see Section 6.5) of the original sample, then report the data and findings for both QC samples. However, if the two results differ by more than the target precision goal, review precision of QCCS measurements for batch; check preparation of split sample; etc. and report evaluation and findings in the case narrative. Consult with the EPA HQ NCCA Laboratory Review Coordinator to determine if reanalysis of the entire batch (at the laboratory's expense) is necessary. If no reanalysis is necessary, report and quantify all samples in batch. If reanalysis is necessary, then report all QC sample and the 2nd analysis of the batch. If the second set also is unacceptable, then assign a data code to each sample in the batch.
Compare results of one matrix spike sample per batch to evaluate performance in matrix	Evaluate performance after the first 3 batches; and then every subsequent batch. Ideally, control limits for recovery will not	If both the original and duplicate results are below LRL, then conclude that the test has passed for the batch. Otherwise, if any

Activity	Evaluation	Corrective Action
(not required for TOC and grain size)	exceed the target accuracy goal, but this may not be realistic for all parameters with this matrix.	results are not within the target accuracy goal for the first 3 batches, within 2 working days, contact the EPA HQ NCCA Laboratory Review Coordinator to discuss method performance and potential improvements. After achieving acceptable results or EPA's permission to continue, perform the test for every subsequent batch. For each batch, report the results from the original analysis and its duplicate and their RPD for TOC; the matrix spike, matrix spike duplicate, RPD and %recovery for contaminants.
Compare results of TOC Certified Reference Material once per each batch	Value must be within 10% of the certified value.	If value is outside the acceptable range, analyze a second CRM. If the second CRM also is measured outside the acceptable range, then determine and correct the problem (e.g., contamination, instrument calibration) before reanalyzing all samples in the batch.
Maintain the required MDL identified in Section 6.5	Evaluate for each sample	If MDL could not be achieved, then provide dilution factor or QC code and explanation in the comment field.
Participate in External Quality Control	Evaluate QC samples provided by the External QC Coordinator	Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any deviations from the Method or unique laboratory practices that might account for differences between the laboratory and others. With this additional information, the External QC Coordinator will determine an appropriate course of action, including no action, flagging the data, or excluding some or all of the laboratory's data.
Maintain completeness	Completeness objective is 95% for all parameters.	Contact EPA HQ NCCA Laboratory Review Coordinator immediately if issues affect

Activity	Evaluation	Corrective Action
		laboratory's ability to meet completeness objective.

*Chapter 2 provides contact information for the EPA HQ NCCA Laboratory Review Coordinator. Laboratories under contract to EPA must contact the Task Order's Contracting Officer's Representative (TOCOR) instead of the Laboratory Review Coordinator.

6.8 Sample and Record Retention

The laboratory shall retain:

1. The sample materials, including vials, for a minimum of 3 years from the date the EPA publishes the final report. During this time, the laboratory shall freeze the materials used in the contaminant analyses and refrigerate those used for the grain size and TOC. The laboratory shall periodically check the sample materials for degradation.

2. Original records, including laboratory notebooks and the reference library, for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

6.9 References

All references are from U.S. Environmental Protection Agency:

Method 245.7 "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, Revision 2.0" (EPA-821-R-05-001, February 2005), retrieved June 27, 2014 from http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_07_10_methods_method_245_7.pdf.

Method 3051a "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, And Oils" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf.

Method 3150A "Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils," retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf.

Method 3540C Method 3540C "Soxhlet Extraction" retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3540c.pdf.

Method 6020A “Inductively Coupled Plasma-Mass Spectrometry” retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/6020A.pdf.

Method 8270D “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/8270D.pdf.

Method 9171B “n-Hexane Extractable Material (HEM) for Sludge, Sediment, And Solid Samples,” retrieved June 27, 2014 from http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/9071b.pdf.

7.0 WATER CHEMISTRY AND CHLOROPHYLL *A*

This chapter describes the analysis requirements for water quality samples. The purpose is to determine concentrations of water quality parameters and chlorophyll *a* in water quality samples collected in the 2015 NCCA and related studies. The laboratory shall perform analysis to determine levels of ammonia (NH_3), nitrate (NO_3), nitrate-nitrite (NO_3-NO_2), total nitrogen (TN), total phosphorous (TP) and ortho-phosphate (PO_4) (also called soluble reactive phosphorus (SRP), pH, conductivity and chlorophyll *a* found in coastal waters and Great Lakes. In addition, the laboratory shall measure chloride (Cl) and sulfate (SO_4) levels in Great Lakes samples.

In the following discussion, Sections 7.1, 7.2, and 7.3 summarize the procedure; health and safety concerns; and definitions and required resources. Section 7.4 provides the steps for acknowledging sample receipt. Sections 7.5 – 7.6 provide the minimum requirements that the laboratory must meet in performing the analyses and the required data elements. Section 7.7 describes EPA's external review of laboratory operations and other quality measures. Section 7.8 identifies references used in developing the procedure.

7.1 Summary of the Procedure

This chapter describes the analysis of ammonia, nitrate-nitrite, total nitrogen, total phosphorous and ortho-phosphate, nitrate, pH, conductivity and chlorophyll *a,* and chloride samples collected for EPA's 2015 National Coastal Condition Assessment (NCCA). As described in Section 7.5, unless otherwise contractually bound by other requirements, the laboratory may choose to use any method that meets EPA's specifications for contamination measurements.

7.2 Health and Safety Warnings

The laboratory must require its staff to abide by appropriate health and safety precautions. In addition to the laboratory's usual requirements such as a Chemical Hygiene Plan, the laboratory must adhere to the following health and safety procedures:

1. Laboratory facilities must properly store and dispose of solutions of weak acid.

2. Laboratory personnel must wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear, gloves).

3. When working with potential hazardous chemicals (e.g., weak acid), laboratory personnel must avoid inhalation, skin contact, eye contact, or ingestion. Laboratory personnel must avoid contacting skin and mucous membranes with acid. If skin contact occurs, remove clothing immediately. Wash and rinse the affected skin areas thoroughly with large amounts of water.

7.3 Definitions and Required Resources (Personnel, Laboratories, and Equipment)

This section provides definitions and required resources for using the procedure.

7.3.1 Definitions

The procedure uses the following terms:

Cl: Chloride

Detection Limit is the minimum concentration at which the analyte can be *detected* with confidence. In other words, the outcome can be reported with confidence that it is greater than zero (i.e., present in the sample) Also see "Sample-Specific Detection Limit."

Duplicates are defined as two aliquots of the same sample which are analyzed separately using identical procedures. The results are used to evaluate the precision of the laboratory analyses.

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management System (NARS IM): The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. Freshwater and coastal samples will be collected during the field stage of NCCA.

NH_3: Ammonia

NO_3: Nitrate

NO_3-NO_2: Nitrate-nitrite

Percent Recovery: Recovery is measured by comparing the concentrations of a sample split into two parts; and one part is spiked with a known concentration value. C_s is the concentration measured in the spiked part; C is the concentration measured in the unspiked part; and s is the known concentration amount for the spike. The following equation is used to calculate the percent recovery:

$$\%Rs = \frac{C_s - C}{s} \times 100$$

Relative Standard Deviation (RSD): The precision at each concentration is reported in terms of the RSD. To calculate the RSD, first calculate the standard deviation, S, as follows:

$$S = \left[\frac{1}{n-1}\sum_{k=1}^{n}(C_s - \bar{C})^2\right]^{1/2}$$

where n is the number of replicate samples, C, is the concentration measure for the k[th] sample, and $\bar{C}$ is the average concentration of the replicate samples. Then, RSD is calculated as:

$$RSD = \left|\frac{S}{\bar{C}}\right| \times 100$$

Reporting Limit: A reporting limit is the point at which the measured value of the analyte can be reported with confidence.

Sample-Specific Detection Limit: Most samples will have a sample-specific detection equal to the method's detection limit. For diluted samples, the sample-specific detection limit will be the product of the method's detection limit and the dilution factor. Typical values for the dilution factors will be 10 or 100.

SO_4: Sulfate.

Spiked Sample: See Percent Recovery definition for purpose of spiked samples.

SRP: Soluble Reactive Phosphorus (also called orthophosphate)

TN: Total nitrogen

TP: Total phosphorous

7.3.2 General Requirements for Laboratories

Expertise. To demonstrate its competency/expertise, the laboratory shall provide EPA with performance data demonstrating their proficiencies in analyzing water quality samples. In addition, the laboratory must provide one or more of the following:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
- Documentation detailing the expertise of the organization, including professional certifications for water-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.

Quality assurance and quality control requirements.
To demonstrate its expertise in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), Laboratory Quality Assurance Manuals, QAPPs, and applicable Standard Operating Procedures (SOPs).

To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

7.3.3 Personnel

The procedure refers to the following personnel:

> **Laboratory Technician**: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual.

7.3.4 Equipment/Materials

The analytical method, selected by the laboratory, identifies the necessary equipment.

7.4 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery. For each sampled site, the lab will receive the following samples on wet ice:

- One 250 ml amber bottle labeled 'CHEM' for water chemistry analyses
- A filter in a 50 ml tube for chlorophyll *a* labeled 'CHLA'

Additionally, as a separate batch shipment the lab will receive 250 ml bottles labeled 'NUTS' for dissolved nutrients analyses (either from the crews or from an EPA batching laboratory). Crews and the batch lab will maintain these samples frozen but will ship overnight on wet ice.

The laboratory technician must inspect the samples promptly on receipt and:

1. Log the samples into the National Aquatic Resource Survey Information Management system (NARS-IM) within 24 clock hours. Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Check that each shipping container has arrived undamaged. Check the temperature of one of the samples in the cooler using a thermometer that reads to at least -20 ºC (i.e., the expected temperature of frozen samples), or an infra-red (IR) temperature "gun" and record the reading. Temperature of the wet ice shipments should be 4 ºC or at less. .Record the condition and temperature of the sample in the database using the codes in Table 7.1.

3. Verify that all required data elements, per Table 7.1, have been recorded in the NARS IM database. If any data elements are missing, then enter them into the database.

4. Transfer the samples for storage as follows:

a. Water chemistry aliquots are prepared following the requirements in Section 7.5 and then are stored in a refrigerator at 4° C.
b. Chlorophyll-*a* filters to the freezer for no more than 30 days before analysis. Except during processing and analysis stages, the filter must be stored frozen to less than or equal -20 °C ± 2°.
c. Dissolved nutrient samples are prepared following the requirements in Section 7.5 and then are stored in a refrigerator at 4° C.

5. Notify the EPA immediately about any problems involving sample integrity, conformity, or inconsistencies as soon as possible following sample receipt and inspection.

Table 7.1 Water Chemistry Login: Required Data Elements

Variable	Type	Description	
SITE_ID	Character	Site identification code	
SAMPLE	Character	Sample number	
DATE_COLLECT	Date	Date that the field crew collected the sample	
ANALYSIS_TYPE	Character	Water Chemistry or Chlorophyll α or Nutrients	
ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory (CHEM, CHLA and NUTS sample will be on wet ice);	
CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		NF	Sample is not at proper temperature
		Q	Other quality concerns, not identified above
COND_COMMENT	Character	Explanation for Q FLAG (if needed)	

7.5 Preparation of Water Chemistry Aliquots

Figure 7.1 presents the sample preparation processing steps for the water chemistry indicators, including filtering and acidifying.
For the dissolved nutrient (NUTS) sample, the laboratory technician:

1. Thaws the frozen sample.
2. Splits the sample into two aliquots as shown in figure 7.1.
3. Adds ultra-pure acid (H_2SO_4, depending on the analytes, see Table 7.2) to one of the two aliquots. Caps the bottle tightly and inverts the bottle several times to mix.
4. Stores all aliquots in a refrigerator at 4°C.

For the unfiltered, water chemistry (CHEM) sample, the laboratory technician

1. Thaws the frozen sample.
2. Splits the sample into two aliquots as shown in figure 7.1.
3. Adds ultra-pure acid (H_2SO_4,) to one aliquot of the unfiltered, CHEM sample. Caps the bottle tightly and inverts the bottle several times to mix.
4. Stores all aliquots in a refrigerator at 4°C.

If the dissolved nutrient sample is compromised in some way, the laboratory technician will filter a new sample from the water chem (CHEM) sample as follows:

1. Uses 0.4µm pore size polycarbonate filters for all filtration.
2. Rinses vacuum filter funnel units thoroughly with reverse-osmosis (RO) or de-ionized (DI) water (ASTM Type II reagent water) five times before each use and in between samples. After placing a filter in the funnel unit, run approximately 100 mL of RO or DI water through the filter, with vacuum pressure, to rinse the filter. Discard the rinse water.
3. Places the appropriate sample bottle under the funnel unit and filter sample directly into the bottle. If a new filter is needed, remove the sample bottle, and rinse the new filter with 100 mL of RO or DI water before continuing.
4. After all filtered and unfiltered aliquots are collected, adds ultra-pure acid (H_2SO_4, depending on the analyte, see Table 7.2) to the sample in the aliquot container. Cap tightly and invert the bottle several times to mix.
5. Stores all aliquots in a refrigerator at 4°C.

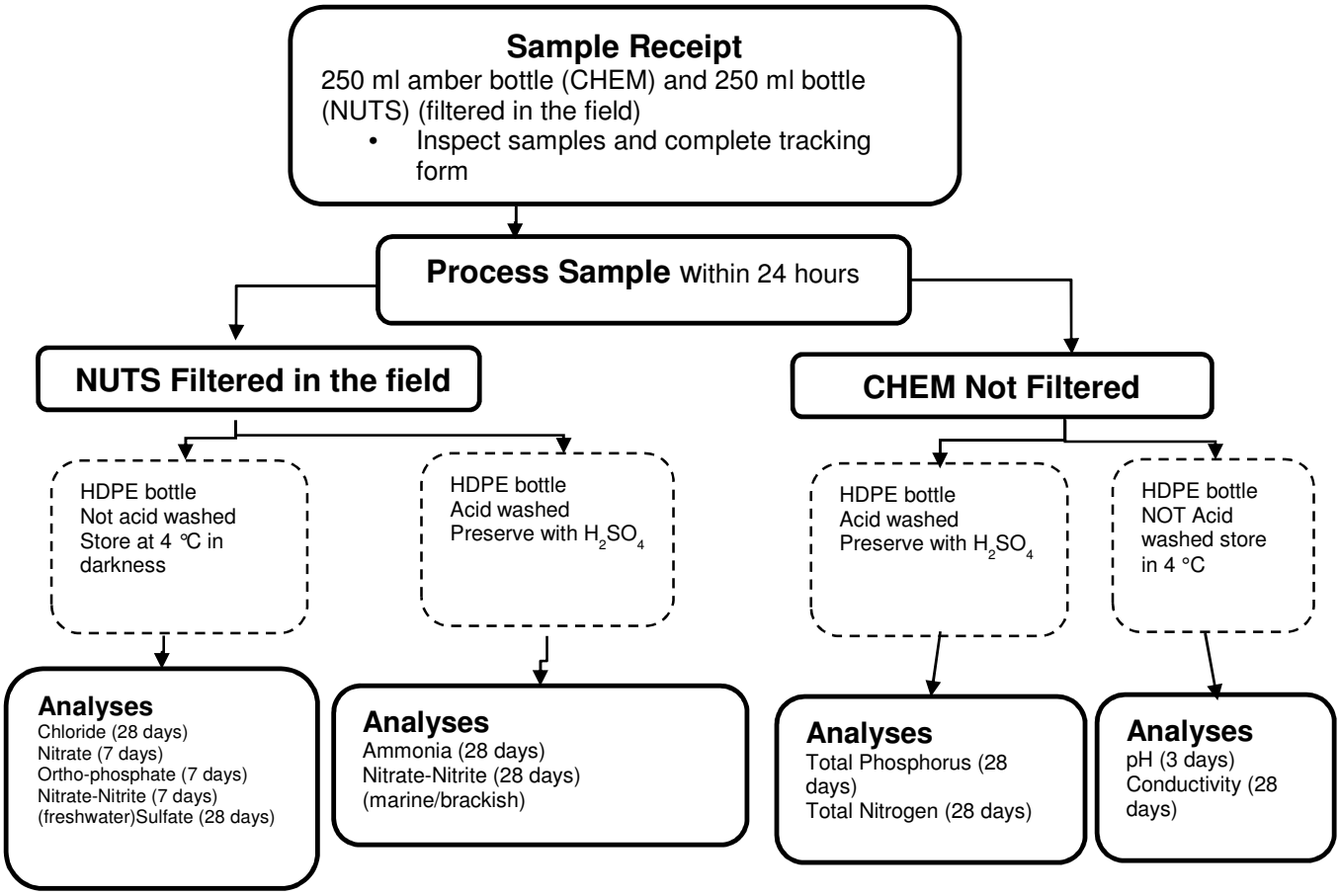

Figure 7.1 Water Chemistry and Dissolved Nutrient Samples: Receipt and Holding Times

Table 7.2 Water chemistry: acid preservatives added for various indicators

	Preservatives
	H_2SO_4 Used for:
Indicators	
	NH_4
	Total N
	Total P
	NO_2-NO_3

7.6 Water Chemistry and Chlorophyll *a* Analysis: Requirements

The laboratory shall perform analysis of the samples to determine the ammonia (NH_3), chloride and sulfate (Great Lakes only), nitrate-nitrite (NO_3-NO_2), total nitrogen (TN), total phosphorous (TP) and ortho-phosphate, nitrate (NO_3), and chlorophyll *a*. As an alternative to specifying laboratory methods for sample analysis, NCCA uses a performance-based approach that defines a set of laboratory method performance requirements for data quality as shown in Table 7.3. Method performance requirements for this project identify the reporting limit, precision, and accuracy objectives for each parameter. NCCA is designating the reporting limit as the lowest

value that the laboratory needs to quantify (as opposed to just detecting the parameter in the sample), and is the value of the lowest non-zero calibration standard that the laboratory must use. EPA has set the value to double the long-term method detection limit (LT-MDL), following guidance presented in Oblinger, Childress et al. (USGS, 1999)18.

NCCA expresses precision and accuracy objectives in both absolute and relative terms following Hunt and Wilson (1986). The transition value is the value at which performance objectives for precision and accuracy switch from absolute (≤ transition value) to relative (> transition value). For pH, the objectives are established for samples with lower, midrange and higher pH levels.

For duplicate samples, NCCA estimates the precision as the pooled standard deviation (calculated as the root-mean square) of all samples at the lower concentration range, and as the pooled percent relative standard deviation of all samples at the higher concentration range. For standard samples (of known concentration), precision is estimated as the standard deviation of repeated measurements across batches at the lower concentration range, and as percent relative standard deviation of repeated measurements across batches at the higher concentration range. Accuracy is estimated as the difference between the mean measured value and the target value of a performance evaluation and/or internal reference samples at the lower concentration range measured across sample batches, and as the percent difference at the higher concentration range.

Table 7.4 summarizes the analytical methods used at the NCCA central laboratory (EPA ORD-Corvallis). Other participating laboratories may use alternative analytical methods for each target analyte as long as they can satisfactorily demonstrate the alternative method is able to achieve the performance requirements as listed in Table 7.3. Appendix A identifies the information that the laboratory should provide to the NCCA Laboratory Review Coordinator to use in determining whether the laboratories meet the necessary requirements.

18 If a laboratory has questions related to meeting the -LT-MDL, they may contact the NCCA Laboratory Review Coordinator to discuss concerns.

Table 7.3 Water Chemistry and Chlorophyll-*a*: Laboratory Method Performance Requirements

Parameter	Units	Potential Range of Samples[1]	Method Detection Limit Objective[2]	Transition Value[3]	Precision Objective[4]	Accuracy Objective[5]
Ammonia (NH_3)	mg N/L	0 to 17	0.01 marine (0.7 µeq/L) 0.02 freshwater	0.10	± 0.01 or ±10%	± 0.01 or ±10%
Chloride (Cl)	mg Cl/L	0 to 5,000	0.20 (6 µeq/L)	1	± 0.10 or ±10%	± 0.10 or ±10%
Conductivity	µS/cm at 25˚C	1-66,000	1.0	20	±2 or ±10%	±2 or ± 5%
Nitrate-Nitrite (NO_3-NO_2)	mg N/L	0 to 360 (as nitrate)	0.01 marine 0.02 freshwater	0.10	± 0.01 or ±10%	± 0.01 or ±10%
pH (Laboratory)	Std Units	3.5-10	N/A	5.75, 8.25	≤5.75 or ≥ 8.25 = ±0.07; 5.75-8.25 = ±0.15	≤5.75 or ≥ 8.25 =±0.15; 5.75-8.25 = ±0.05
Total Nitrogen (TN)	mg N/L	0.1 to 90	0.01	0.10	± 0.01 or ±10%	± 0.01 or ±10%
Total Phosphorous (TP) and ortho-Phosphate	mg P/L	0 to 22 (as TP)	0.002	0.02	± 0.002 or ±10%	± 0.002 or ±10%
Nitrate (NO_3)	mg N/L	0. to 360	0.01 marine (10.1 µeq/L) 0.03 freshwater	0.1	± 0.01 or ±5%	± 0.01 or ±5%
Sulfate (SO_4)	mg/L	0 to 5000	0.5 freshwater (10.4 ueq/L)	2.5	±0.25 or ±10%	±0.25 or ±10%
Chlorophyll-*a*	µg/L in extract	0.7 to 11,000	1.5	15	± 1.5 or ±10%	± 1.5 or ±10%

[1] Estimated from samples analyzed at the EPA Western Ecological Division-Corvallis laboratory between 1999 and 2005

[2] The method detection limit is determined as a one-sided 99% confidence interval from repeated measurements of a low-level standard across several calibration curves.

[3] Value for which absolute (lower concentrations) vs. relative (higher concentrations) objectives for precision and accuracy are used.

[4] For duplicate samples, precision is estimated as the pooled standard deviation (calculated as the root-mean square) of all samples at the lower concentration range, and as the pooled percent relative standard deviation of all samples at the higher concentration range. For standard samples, precision is estimated as the standard deviation of repeated measurements across batches at the lower concentration range, and as

percent relative standard deviation of repeated measurements across batches at the higher concentration range.

For pH precision, the looser criteria applies to mid-range samples. For NCCA, that is less of a concern than the ability to measure more acidic or basic samples accurately and precisely.

[5] Accuracy is estimated as the difference between the measured (across batches) and target values of performance evaluation and/or internal reference samples at the lower concentration range, and as the percent difference at the higher concentration range.

Table 7.4 Water Chemistry and Chlorophyll-*a*: Analytical Methods Used by Central Laboratory, EPA ORD-Corvallis)

Analyte	Summary of Method[19]	References[20]	WRS SOP[21]
Nitrate+Nitrite, as N	Ion Chromatography (freshwater samples) OR FIA automated colorimetric (cadmium reduction for brackish samples)	EPA 300.6; SW-846 9056A; APHA 4110B EPA 353.2 APHA 4500-NO_3-N-E Lachat 10-107-04-1-C	WRS 36A.0 (April 2011 WRS 40A.5 (May 2011)
Ammonia, as N	FIA automated colorimetric (salicylate, dichloroisocyanurate)	Lachat 10-107-06-3-D	WRS 30A.4 (April 2011)
Total nitrogen (TN)	Persulfate Digestion; FIA Automated Colorimetric Analysis (Cadmium Reduction, sulfanilamide)	EPA353.2 (modified) APHA 4500-N-C (modified) ASTM WK31786 U.S. EPA (1987) Lachat 10-107-04-1-C (modified)	WRS 34A.5 (April 2011)
Total phosphorus (TP) and ortho-Phosphate	Persulfate Digestion; Automated Colorimetric Analysis (molybdate, ascorbic acid)	APHA 4500-P-E USGS I-4650-03 U.S. EPA (1987) Lachat 115-01-1-B (modified)	WRS 34A.5 (April 2011)
Nitrate, chloride, sulfate	Ion Chromatography (Great Lakes samples only)	EPA 300.6; SW-846 9056A; APHA 4110B	WRS 40A.5 (May 2011)
Chlorophyll-a (Chl-a)	Extraction 90% acetone analysis by fluorometry	EPA 445.0 , EPA 446.0	WRS 71A.3 (April 2011)
pH (lab)	Automated, using ManSci PC-Titrate w/ Titra-Sip autotitrator and Ross combination pH electrode. Initial pH determination for ANC titration	EPA 150.6 (modified)	WRS 16A.0 (April 2011)

[19] FIA=Flow injection analysis. AAS=Atomic Absorption Spectrometry

[20] U.S. EPA, 1987. *Handbook of Methods for Acid Deposition Studies: Laboratory Analyses for Surface Water Chemistry.* EPA/600/4-87/026. U.S. Environmental Protection Agency, Office of Research and Development, Washington D.C. APHA= American Public Health Association (*Standard Methods*). ASTM=American Society of Testing and Materials.

[21] WRS= Willamette Research Station. References are to laboratory SOP being used at central laboratory. Available upon request from the EPA HQ Laboratory Review Coordinator.

Analyte	Summary of Method[19]	References[20]	WRS SOP[21]
Specific conductance @ 25°C	Electrolytic, Man-Tech TitraSip automated analysis OR manual analysis, electrolytic	EPA 120.6	WRS 16A.0 (April 2011) WRS 11A.4 (April 2011)

7.7 Data Entry

Table 7.5 identifies the required data elements that laboratories must provide to EPA, preferably in EPA's data template, available separately from EPA.

Table 7.5 Water Chemistry and Chlorophyll-*a*: Data Elements for Each Sample

Variable	Type	Description	
SITE_ID	Character	Site identification code or type of QC sample (e.g., LAB BLANK)	
SAMPLE	Character	Sample number, LCS, QCCS, Blank, Matrix Spike, or CRM	
ANALYSIS_TYPE	Character	Contaminant	
REPEAT	Numeric	Duplicate	
DATE_COLLECT	Date	Date that the field crew collected the sample	
ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory	
CONDITION_CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		NF	Sample is not at proper temperature
		Q	Other quality concerns, not identified above
COND_COMMENT	Character	Explanation for Q FLAG (if needed)	
PARAMETER	Character	Analyte name	
CAS_NO	Character	CAS Registry number	
LABNAME	Character	Laboratory name (abbreviation)	
METHOD	Character	Laboratory method used	
ANALYST	Character	Last name or initials of person who performed the analysis	
REVIEWER	Character	Last name or initials of the person who provided a separate independent review of the data	
INSTRUMENT	Character	Identification of instrument used for the analysis – provide enough information to identify the particular instrument in the laboratory	
DATE PROCESSED	Date	Date that the analysis started	
QC_BATCH_LOT	Character	Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate laboratory control sample, matrix spike, laboratory duplicate, method blank, and CRM samples.	
HOLDING TIME	Y/N	Analysis performed within holding time	

Variable	Type	Description
MATRIX	Character	Water
MDL	Numeric	Lab method detection limit (based upon lab's historical data)
LRL	Numeric	Lab reporting limit (based upon lab's historical data)
DILUTION	Numeric	Dilution of sample (blank or 1 if no dilution)
RESULT	Numeric	Concentration value
REASON	Character	Reason for qualification in RESULT_QUAL (usually blank)
RESULT_QUAL	Character	Data qualifier (usually blank)
UNIT	Character	Unit of measurement for RESULT, MDL, and LRL
QC_CODE	Character	Apply laboratory defined QC codes and describe in the comments field. Provide set of laboratory's code as part of the case narrative
COMMENT	Character	Explain situation that created QC code, or any unusual aspects of the analysis

7.8 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements. QC protocols are an integral part of all analytical procedures to ensure that the results are reliable and the analytical stage of the measurement system is maintained in a state of statistical control. The laboratory must conduct QC analyses for each batch of samples. Each batch shall consist of no more than 20 samples. Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate measures such as laboratory control sample, matrix spike, laboratory duplicate, and method blank samples. Also, each laboratory QC samples (i.e., preparation and instrument blanks, laboratory control sample (LCS), spike/duplicate, etc.) must be give a unique sample identification. Table 7.5 provides a summary of the quality control requirements.

Table 7.5 Water Chemistry and Chlorophyll-*a*: Quality control activities for water quality samples

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
Demonstrate competency for analyzing water samples to meet the performance measures	All	Demonstration of past experience with water samples in achieving the method detection limits	Once	See Appendix A	EPA will not approve any laboratory for NCCA sample processing if the laboratory cannot demonstrate competency. In other words, EPA will select another laboratory that can

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
					demonstrate competency for its NCCA samples.
Check condition of sample when it arrives.	All	Sample issues such as cracked container; missing label; temperature; adherence to holding time requirements; sufficient volume for test.	Once	No sample issues or determination that sample can still be analyzed	Lab determines if the sample can be analyzed or has been too severely compromised (e.g., contamination). Assign appropriate condition code identified in Table 7.1.
Store sample appropriately.	All	Check the temperature of the refrigerator per laboratory's standard operating procedures.	Record temperature of sample upon arrival at the laboratory. Check temperature of the refrigerator/freezer where samples are stored at least daily if using a continuous temperature logger and twice daily (once at beginning of the day and once at the end) not using a continuous logger.	While stored at the laboratory, the sample must be kept at a maximum temperature of 4° C (for aliquots except chlorophyll *a*) and -20° C for the chlorophyll *a* sample.	If at any time samples are warmer than required, note temperature and duration (either from the continuous temperature log or from the last manual reading) in comment field. Lab will still perform test. EPA expects that the laboratory will exercise every effort to maintain samples at the correct temperature.

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
Analyze sample within holding time	All			The test must be completed within the holding time specified in the analytical method.	Perform test in all cases, but note reason for performing test outside holding time. EPA expects that the laboratory will exercise every effort to perform tests before the holding time expires.
Analyze Laboratory/ Reagent Blank	All		Once per day prior to sample analysis	Control limits ≤ MDL	Prepare and analyze new blank. Determine and correct problem (e.g., reagent contamination, instrument calibration, or contamination introduced during filtration) before proceeding with any sample analyses. Reestablish statistical control by analyzing three blank samples.
Analyze Filtration Blank	All dissolved analytes	ASTM Type II reagent water processed through filtration unit	Prepare once per week and archive Prepare filter blank for each box of 100 filters, and examine the results before any other filters are used from that box.	Measured concentrations <MDL	Measure archived samples if review of other laboratory blank information suggest source of contamination is sample processing.

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
Determine LT-MDL Limit for Quality Control Check Sample (QCCS)	All	Prepared so concentration is four to six times the LT-MDL objective	Once per day	Target LT-MDL value (which is calculated as a 99% confidence interval)	Confirm achieved LRL by repeated analysis of LT-MDL QCCS. Evaluate affected samples for possible re-analysis.
Analyze Calibration QCCS	All		Before and after sample analyses	±10% or method criteria	Repeat QCCS analysis. Recalibrate and analyze QCCS. Reanalyze all routine samples (including PE and field replicate samples) analyzed since the last acceptable QCCS measurement.
Analyze Laboratory Duplicate Sample	All		One per batch	Control limits < precision objective	If results are below LRL: Prepare and analyze split from different sample (volume permitting). Review precision of QCCS measurements for batch. Check preparation of split sample. Qualify all samples in batch for possible reanalysis.

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
Analyze Standard Reference Material (SRM)	When available for a particular indicator		One analysis in a minimum of five separate batches	Manufacturers certified range	Analyze standard in next batch to confirm suspected inaccuracy. Evaluate calibration and QCCS solutions and standards for contamination and preparation error. Correct before any further analyses of routine samples are conducted. Reestablish control by three successive reference standard measurements that are acceptable. Qualify all sample batches analyzed since the last acceptable reference standard measurement for possible reanalysis.
Analyze Matrix Spike Samples	Only prepared when samples with potential for matrix interferences are encountered		One per batch	Control limits for recovery cannot exceed 100±20%	Select two additional samples and prepare fortified subsamples. Reanalyze all suspected samples in batch by the method of

QC Sample Type and Description	Indicators	Description	Frequency	Acceptance Criteria	Corrective Action
					standard additions. Prepare three subsamples (unfortified, fortified with solution approximately equal to the endogenous concentration, and fortified with solution approximately twice the endogenous concentration).
Use consistent units for QC samples and field samples	All	Verify that all units are provided consistently within each indicator.	Data reporting	For each indicator, all field and QC samples are reported with the same measurement units	If it is not possible to provide the results in consistent units, then assign a QC code and describe the reason for different units in the comments field of the database.
Maintain completeness	All	Determine completeness	Data reporting	Completeness objective is 95% for all indicators (useable with or without flags).	Contact EPA HQ NCCA Laboratory Review Coordinator* immediately if issues affect laboratory's ability to meet completeness objective.

*Chapter 2 provides contact information for the EPA HQ NCCA Laboratory Review Coordinator. Laboratories under contract to EPA must contact the Task Order's Contracting Officer's Representative (TOCOR) instead of the Laboratory Review Coordinator.

7.9 Sample and Record Retention

The laboratory shall retain:

1. The sample materials for a minimum of 1 year after collection. During this time, the laboratory shall store the materials cold (e.g., 4 ° C) and in darkness. The lab shall retain the sample materials from the 1 year point until the EPA publishes the final report at ambient temperatures.
2. Original records, including laboratory notebooks for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

7.10 References

Hunt, D.T.E. and A.L. Wilson. 1986. ***The Chemical Analysis of Water: General Principles and Techniques***. 2nd ed. Royal Society of Chemistry, London, England.

USEPA, 1987. ***Handbook of Methods for Acid Deposition Studies: Laboratory Analyses for Surface Water Chemistry.*** EPA/600/4-87/026. U.S. Environmental Protection Agency, Office of Research and Development, Washington D.C.

USEPA. 1997. ***Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices – 2nd Edition***. EPA No. 600-R-97-072. U.S. Environmental Protection Agency, Office of Research and Development, Washington, DC, retrieved June 30, 1997 from http://www.epa.gov/microbes/documents/marinmet.pdf.

USEPA. September 1997. Method 353.4 "Determination of Nitrate and Nitrite in Estuarine and Coastal Waters by Gas Segmented Continuous Flow Colorimetric Analysis, Revision 2.0", retrieved June 30, 2014 from http://www.epa.gov/microbes/documents/m353_4.pdf.

USGS. 1999. "New reporting procedures based on long-term method detection levels and some considerations for interpretations of water-quality data provided by the U.S. Geological Survey National Water Quality Laboratory." Open-File Report: 99-193 by Childress, Oblinger, *et al.*, retrieved June 30, 2014 from http://pubs.usgs.gov/of/1999/0193/report.pdf.

Youden, W.J. 1969. Ranking laboratories by round-robin tests. In *Precision Measurement and Calibration*. H.H. Ku, ed. NBS Special Publication 300, Vol. 1. U.S. GPO Washington, D.C.

8.0 SEDIMENT TOXICITY TESTING

This chapter describes the analysis requirements for sediment toxicity testing. The purpose is to assess the toxicity of sediment samples collected in the 2015 NCCA and related studies.

At each sampling site, the Field Operations Manual (FOM) instructs the crews to collect sediment samples. The field crew then ships the samples on wet ice to the laboratory. If EPA uses a batching laboratory, it will refrigerate the samples, before shipping on wet ice to the analysis laboratory.

In the following discussion, Sections 8.1, 8.2, and 8.3 summarize the procedure; health and safety concerns; and definitions and required resources. Section 8.4 provides the steps for acknowledging sample receipt. Sections 8.5 – 8.6 provide the minimum requirements that the laboratory must meet in performing the analyses and the required data elements. Section 8.7 describes EPA's external review of laboratory operations and other quality measures. Section 8.8 identifies references used in developing the procedure.

8.1 Summary of the Procedure

This chapter describes toxicity testing of sediment samples collected for EPA's 2015 National Coastal Condition Assessment (NCCA). As described in Section 8.5, unless otherwise contractually bound by other requirements, the laboratory may choose to use any method that meets EPA's specifications.

8.2 Health and Safety Warnings

The laboratory must require its staff to abide by appropriate health and safety precautions. In addition to the laboratory's usual requirements such as a Chemical Hygiene Plan, the laboratory must adhere to the following health and safety procedures:

1. Laboratory facilities must properly store and dispose of solutions of weak acid.

2. Laboratory personnel must wear proper personal protection clothing and equipment (e.g. lab coat, protective eyewear, gloves).

3. When working with potential hazardous chemicals (e.g., weak acid), laboratory personnel must avoid inhalation, skin contact, eye contact, or ingestion. Laboratory personnel must avoid contacting skin and mucous membranes with acid. If skin contact occurs, remove clothing immediately. Wash and rinse the affected skin areas thoroughly with large amounts of water.

8.3 Definitions and Required Resources (Personnel, Laboratories, and Equipment)

This section provides definitions and required resources for using the procedure.

8.3.1 Definitions

The procedure uses the following terms:

Replicates are defined as two or more aliquots of the same sample which are analyzed separately using identical procedures. The results are used to evaluate the precision of the laboratory analyses.

NARS: National Aquatic Resource Surveys. The National Coastal Condition Assessment (NCCA) is part of the NARS program.

NARS Information Management System (NARS IM): The IM system established to support all surveys, including NCCA, in the NARS program. The IM system is used to track the samples from field collection to the laboratory.

NCCA: National Coastal Condition Assessment. Freshwater and coastal samples will be collected during the field stage of NCCA.

%CONT_SURV: Average percentage of organisms that survived in the replicate test chambers over the percent survival in control.

%REP_SURV: Percentage of organisms that survived in the test chamber for each set of replicates.

8.3.2 General Requirements for Laboratories

Expertise. To demonstrate its expertise, the laboratory shall provide EPA with performance data demonstrating their proficiencies in analyzing water quality samples. In addition, the laboratory must provide one or more of the following:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
- Documentation detailing the expertise of the organization, including professional certifications for water-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.

Quality assurance and quality control requirements.
To demonstrate its expertise in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).

To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

Preparation for the work

To demonstrate its preparation for the work, the laboratory shall provide documentation that it has complied with the following control analyses prior to the start of any work.

1. The laboratory shall ensure that the water source for the overlying water has been demonstrated to support survival, growth, and reproduction of the test organisms. The laboratory shall provide information on how the laboratory maintains the quality of the water used for the tests.
2. The laboratory shall ensure that the clean sediment is appropriate for the control tests. The laboratory shall provide information about the sediment chemistry analysis and explanation of how the control sediment was selected
3. The laboratory shall ensure that the organisms are healthy for the tests. The laboratory shall provide the source of the organisms; historic information about the culturing; and procedures for evaluating the condition and age of the organism and water quality upon arrival. If the laboratory intends to purchase the organisms (i.e., instead of in-house culturing), identify the commercial source; its shipping arrangements (e.g., test organisms are shipped in well-oxygenated water in insulated containers to maintain temperature during shipment); and evaluation upon arrival at the laboratory (e.g., measuring temperature and dissolved oxygen of the water in the shipping containers to determine if the organisms might have been subjected to low dissolved oxygen or temperature fluctuations).
4. The laboratory shall complete a "non-toxicant" test of each new chamber before using the chamber for NCCA samples. A "new" chamber is one that the laboratory has not previously used for any sediment toxicity testing for any client (e.g., replacement glassware). Ideally, although EPA is not requiring it, the laboratory will test freshwater and marine samples in wholly separate chambers.

   Test requirements: The test chambers contain control sediment (sometimes called the negative control) and clean overlying water for the amphipod species to be tested. Survival of the test organisms will demonstrate whether facilities, water, control sediment, and handling techniques are adequate to achieve acceptable species-specific control survival. For the test to be acceptable, survival at 10 days must equal or exceed the survival requirements in QA/QC specifications in Section 8.7.

8.3.3 Personnel

The procedure refers to the following personnel:

Laboratory Technician: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual.

External QC Coordinator is an EPA staff person who is responsible for selecting and managing the "**QC contractor**." To eliminate the appearance of any inherent bias, the QC contractor must be dedicated to QA/QC functions, and thus, must not be a primary laboratory or a field sampling contractor for NCCA. The QC contractor is responsible for complying with instructions from the External QC Coordinator; coordinating and paying

for shipments of the performance samples to participating laboratories; comparing results from the laboratories; and preparing brief summary reports.

8.3.4 Equipment/Materials

The analytical method, selected by the laboratory, identifies the necessary equipment.

8.4 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery. The laboratory must inspect the samples promptly on receipt. As samples arrive, the laboratory must:

1. Log the samples into the National Aquatic Resource Survey Information Management system (NARS-IM) within 24 clock hours. Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Check that each shipping container has arrived undamaged. Check the temperature of one of the samples in the cooler using a thermometer that measures temperatures between 0 ºC (refrigerated samples are typically 4 °C) and 30 °C (ambient room temperature is typically less than 26 °C), or an infra-red (IR) temperature "gun" and record the reading. Field crews and the batching laboratory will ship sediment samples on wet ice. Record the condition and temperature of the sample in the database using the codes in Table 8.1.

3. Verify that all required data elements, per Table 8.1, have been recorded. If any elements are missing, then enter them into the database.

4. Transfer the samples to the refrigerator until ready for toxicity testing. Except during processing and analysis stages, the samples must be stored at 4°C.

5. Notify the EPA immediately about any problems involving sample integrity, conformity, or inconsistencies as soon as possible following sample receipt and inspection.

Table 8.1 Sediment Toxicity Login: Required Data Elements

FIELD	FORMAT	DESCRIPTION
LAB ID	Character	Name or abbreviation for laboratory
TYPE	Character	Control or NCCA Sample
DATE RECEIVED	MMDDYY	Date sample was received by lab; leave blank for control
SITE ID	Character	NCCA site id as used on sample label; leave blank for control
VISIT NUMBER	Numeric	Sequential visits to site (1 (or blank) or 2); leave blank for control
SAMPLE ID	Numeric	Sample id as used on field sheet (on sample label); leave blank for control
DATE COLLECTED	MMDDYY	Date sample was collected; leave blank for control

FIELD	FORMAT	DESCRIPTION	
ARRIVAL_TEMP	Numeric	Temperature of sample upon arrival at the laboratory (it should arrive on wet ice).	
CONDITION CODE	Character	Condition codes describing the condition of the sample upon arrival at the laboratory; leave blank for control	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		NF	Sample is not at proper temperature
		VT	Volume not sufficient for testing (VT)
		VR	Volume not sufficient for a retest, if required
		HT	Received outside holding time
		Q	Other quality concerns, not identified above
COND_COMMENT	Character	Explanation for Q FLAG (if needed)	

8.5 Toxicity Testing: Requirements

The laboratory shall perform toxicity testing of sediment samples. Laboratories may choose to use any analysis method using the required organisms of *Hyalella azteca* (freshwater) or *Leptocheirus plumulosus* (marine). The laboratory's method must meet the quality requirements in Section 8.7, including mean survival of the control's treatments must remain greater than or equal to 80% and 90%, respectively. It is essential that the contractor require that all of its laboratory technicians use the same procedures and meet the required quality elements. At a minimum, the laboratory must:

1. Perform the procedures using the 10-day tests. Possible methods include those described in the following documents:
   a. Marine: Test Method 100.4 in EPA 600/R-94/025[22] or ASTM E1367-03[23]
   b. Freshwater: Test Method 100.1 in EPA 600/R-99/064[24] or ASTM E1706[25]

2. Test the following number of replicates for each sample and control:
   a. Marine: 5 replicates with 20 organisms per replicate
   b. Freshwater: 4 replicates with 10 organisms per replicate

[22] Chapter 11 in *Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods*, June 1994, retrieved from http://water.epa.gov/polwaste/sediments/cs/upload/marinemethod.pdf.
[23] American Society for Testing and Materials (ASTM). 2008. E1367-03 "Standard Guide for Conducting 10-Day Static Sediment Toxicity Tests With Marine and Estuarine Amphipods." *Annual Book of Standards, Water and Environmental Technology*, Vol. 11.05, West Conshohocken, PA.
[24] Section 11 in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates*, Second Edition, March 2000, retrieved from http://water.epa.gov/polwaste/sediments/cs/upload/freshmanual.pdf.
[25] ASTM 2009 E1706. "Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates."

3. Test no more than 10 samples and one control within each batch.

4. Use the following organisms for the tests:
    a. Marine: *Leptocheirus plumulosus*
    b. Freshwater: *Hyalella azteca*

5. Select organisms for each batch of tests that are:
    a. From the same culture;
    b. Cultured at the same temperature as will be used for the tests;
    c. (optional) EPA would prefer but does not require that the organisms are cultured in the same water as that used for testing.

6. Use a water source (for the overlying water) demonstrated to support survival, growth, and reproduction of the test organisms.
    a. For marine sediments, 175 mL of sediment and 800 mL of overlying seawater
    b. For freshwater sediments, 100mL of sediment and 175mL of overlying freshwater

7. Use clean sediment for control tests.

8. Implement the following for exposure/feeding
    a. For marine sediments, exposure is static (i.e., water is not renewed), and the animals are not fed over the 10 d exposure period
    b. For freshwater, exposure is renewed (i.e., 2 volumes a day) and the animals are fed over the 10 day exposure period

9. Follow the following procedure for homogenization/sieving: Water above the sediment is not discarded, but is mixed back into the sediment during homogenization. Sediments should be sieved for marine samples (following the 10 day method) and the sieve size should be noted. For freshwater samples, they should not be sieved to remove indigenous organisms unless there is a good reason to believe indigenous organisms may influence the response of the test organism. For freshwater samples, large indigenous organisms and large debris can be removed using forceps and if sediments must be sieved, the samples should be analyzed before and after sieving (e.g., pore-water metals, DOC, and AVS) to document the influence of sieving on sediment chemistry (note sieve size).

Additional details are provided in the summary tables 8.2 and 8.3.

Table 8.2 Test Conditions for Conducting 10-d Sediment Toxicity Tests for marine sediments

Parameter	Conditions
1. Test type:	Whole sediment toxicity test, static
2. Temperature:	25℃ for L. plumulosus
3. Salinity	20‰
4. Light quality:	Wide-spectrum fluorescent lights
5. Illuminance:	500 – 1000 lux
6. Photoperiod:	24L:0D
7. Test chamber:	1 L glass beaker or jar with ~10 cm I.D.

8. Sediment volume:	175 mL (2 cm)
9. Overlying water volume:	800 mL
10. Renewal of overlying water:	None
11. Size and life stage of amphipods:	L. plumulosus: 2-4 mm (no mature males or females)
12. Number of organisms per chamber:	20 per test chamber
13. Number of replicate chambers/treatment:	5 (required)
14. Feeding:	None
15. Aeration:	Water in each test chamber should be aerated overnight before start of test and throughout the test aeration at rate that maintains ≥90% saturation of dissolved oxygen concentration
16. Overlying water:	Clean sea water, natural or reconstituted water
17. Overlying water quality:	Temperature daily; pH, ammonia, salinity, and DO at test start and end.
18. Test duration:	10 d
19. Endpoints:	Survival
20. Test acceptability:	Minimum mean control survival of 90%

Table 8.3 Test Conditions for Conducting 10-d Sediment Toxicity Tests for freshwater sediments

Parameter	Conditions
1. Test type:	Whole-sediment toxicity test with renewal of overlying water
2. Temperature:	23°± 1°C
3. Light quality:	Wide-spectrum fluorescent lights
4. Illuminance:	100 to 1000 lux
5. Photoperiod:	16L:8D
6. Test chamber:	300 mL high-form beaker
7. Sediment volume	100 mL
8. Overlying water volume:	175 mL
9. Renewal of overlying water:	2 volume additions/d; continuous or intermittent (*e.g.*, 1 volume addition every 12 h)
10. Age of organisms:	7- to 14-d old at the start of the test (1- to 2-d range in age)
11. Number of organisms/ chamber:	10
12. Replicate chambers/treatment:	4 required
13. Feeding:	YCT food, fed 1.0 mL daily (1800 mg/L stock) to each test chamber.
14. Aeration:	None unless DO in overlying water drops below 2.5 mg/L
15. Test duration:	10 d
16. Endpoint:	Survival
17. Test acceptability:	Min. mean control survival of 80%.

8.6 Data Entry

Tables 8.3 and 8.4 identify the required data elements describing the test conditions and outcomes for the replicates and batches. Laboratories must provide the data elements to EPA, preferably in EPA's data template, available separately from EPA.

Table 8.3 Sediment Toxicity Replicates: Laboratory method performance requirements

FIELD	FORMAT	DESCRIPTION
LAB ID	Character	Name or abbreviation for laboratory
TYPE	Character	Control or NCCA Sample
SAMPLE ID	Numeric	Sample id as used on field sheet (on sample label); leave blank for control
RETEST	Y or blank	Y for yes if the sample is being retested; blank if original test or control
CHAMBER ID	Character	Identification code for test chamber
BATCH ID	Character	Identification code for batch
REPLICATE	Numeric	Replicate number: 1-5 for marine; 1-4 for freshwater
TEST TYPE	Character	Marine or Freshwater
ORGANISM	Character	Leptocheirus plumulosus (marine) or Hyalella azteca (freshwater)
NO_SURVIVED	Numeric	Number of organisms that survived out of 20 (marine) and 10 (freshwater)
%REP_SURV	Numeric	Percentage of organisms that survived in the test chamber for the replicate
REP_COMMENT	Character	Any comments about the test procedures or any abnormalities
%CONT_SURV	Numeric	Optional Field: Average percentage of organisms that survived in the replicate test chambers over the percent survival in control.

Table 8.4 Laboratory method performance requirements for sediment toxicity batches

FIELD	FORMAT	DESCRIPTION
BATCH ID	Character	Identification code for batch
BATCH_SAMPLES	Numeric	Number of NCCA samples in the batch (integer≤10) excluding the control
TEST TYPE	Character	Marine or Freshwater
ORGANISM	Character	Leptocheirus plumulosus (marine) or Hyalella azteca (freshwater)
CONTROL	Character	Source of control sediment
START_DATE	MMDDYY	Date that the laboratory starts the test procedure for the batch
END_DATE	MMDDYY	Date that the laboratory ends the test procedure for the batch
%SURV	Numeric	%Survival for the sample (or control) calculated using the %REP_SURV
BATCH_PASS	P/F	Indicate if the batch passed (P) or failed (F) the QA/QC requirements (e.g., control achieved required survival rates)
QC_CODE	Character	Laboratory assigned code for QC issues with the sample
QC_DESCRIPTION	Character	Description of conditions associated with the QC_CODE
SURV_COMMENT	Character	Any comments about the test procedures or any abnormalities

8.7 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements.

8.7.1 Assistance Visits

Assistance visits are intended to familiarize EPA with actual procedures being implemented by different laboratories; and to ensure a clear and consistent understanding of procedures and activities by both EPA and the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter.

8.7.2 QC Samples

Once or twice during the performance period, the External QC Coordinator will provide one or two identical sets of QC samples to all participating laboratories. Each set will contain up to five QC samples. As determined by the External QC Coordinator, the QC samples may be synthetic; aliquots of additional samples collected at NCCA sites; or reference samples obtained from an organization such as the National Institute of Standards. Each laboratory will run the QC samples following the same procedures used for the other samples. The External QC Coordinator will compare the results to the expected value and determine consistency between laboratories (e.g., determine if one laboratory is consistently higher or lower than all others). Based upon the evaluation, the External QC Coordinator may request additional information from one or more laboratories about any unique laboratory practices that might account for differences between the laboratory and others. The contractor shall analyze the external QC samples using the same procedures as those for the field samples.

8.7.3 Summary of QA/QC Requirements

QC protocols are an integral part of all analytical procedures to ensure that the results are reliable and the analytical stage of the measurement system is maintained in a state of statistical control. The laboratory must conduct QC analyses for each batch of samples. Each batch shall consist of no more than 10 samples. Unique laboratory quality control lot numbers must be assigned to each batch of samples. The lot number must associate each batch of field samples to the appropriate measures such as laboratory control samples. Table 8.5 provides a summary of the quality control requirements.

Table 8.5 Quality control activities for sediment toxicity samples

Activity	Evaluation	Corrective Action
Laboratory demonstrates competency for conducting sediment toxicity analyses	EPA will review SOPs, lab certifications, past performance results, etc. as part of the lab verification process.	EPA will not approve any laboratory for NCCA sample processing if the laboratory cannot demonstrate competency. In other words, EPA will select another laboratory that can

Activity	Evaluation	Corrective Action
		demonstrate competency for its NCCA samples.
Check condition of sample when it arrives.	Sample issues, such as cracked or leaking container; missing label; temperature; adherence to holding time requirements; insufficient volume for test.	Assign appropriate condition code identified in Table 8.1.
Sample storage	All samples: 4 °C at arrival at the laboratory (temperature recorded at arrival) and while stored at the laboratory.	Record temperature upon arrival at the laboratory. Check temperature of the refrigerator where samples are stored at least daily if using a continuous temperature logger and twice daily (beginning and end of day) if the lab does not have a continuous logger. If refrigerator is warmer than required, note temperature and duration (either from the continuous temperature log or from the last manual reading) in comment field. Lab will still perform test. EPA expects that the laboratory will exercise every effort to maintain samples at the correct temperature.
Holding Time	The test must be completed within 8 weeks after sample collection. If the original test fails, then the retest also must be conducted within the 8 weeks after sample collection.	Perform test, but note reason for performing test outside holding time. EPA expects that the laboratory will exercise every effort to perform tests before the holding time expires.
Check that the organisms are healthy before starting the test	Unhealthy organisms may appear to be discolored, or otherwise stressed (for example, greater than 20 percent mortality for the 48 hours before the start of a test).	Don't start test using unhealthy organisms.
Maintain conditions as required in Section 8.3.	Check conditions (e.g., temperature, DO) each test day. Record conditions in bench sheet or in laboratory database.	Note any deviations in comments field (Table 8.1). In extreme cases, conduct a new toxicity test for all samples

Activity	Evaluation	Corrective Action
		affected by the adverse conditions.
Control survival rates	For a test of a batch of samples to be considered valid, the control's mean survival in *Hyalella* and *Leptocheirus* treatments must remain ≥80% and ≥90%, respectively.	Data template includes a field to record if a test passed or failed the control requirements. If a test fails, retest all samples in the batch. Report both the original and retest results. If both tests fail, submit data to EPA for further consideration. Include comments in the data template noting any particular factors that may have caused the test to fail twice.

*Chapter 2 provides contact information for the EPA HQ NCCA Laboratory Review Coordinator. Laboratories under contract to EPA must contact the Task Order's Contracting Officer's Representative (TOCOR) instead of the Laboratory Review Coordinator.

8.8 Sample and Record Retention

The laboratory shall retain:

1. The sample materials, including vials until March 31, 2016 which will allow EPA with time to review the data and contact the laboratory with any questions about the samples. Until this time, the laboratory shall refrigerate the sediment samples. The laboratory shall periodically check the sample materials for degradation.

2. Original records, including laboratory notebooks, for a minimum of 10 years from the date that EPA publishes the final report.

After the stated time periods, the laboratory shall follow its internal protocols for disposal.

8.9 References

American Society for Testing and Materials (ASTM). 2008. E1367-03 "Standard Guide for Conducting 10-Day Static Sediment Toxicity Tests With Marine and Estuarine Amphipods." Annual Book of Standards, Water and Environmental Technology, Vol. 11.05, West Conshohocken, PA.

ASTM. 2009. E1706. "Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates.

United Stated Environmental Protection Agency (USEPA). 1994. Chapter 11 in Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods, retrieved on March 13, 2014 from http://water.epa.gov/polwaste/sediments/cs/upload/marinemethod.pdf.

USEPA. 2000. Section 11 in Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, Second Edition, retrieved on March 13, 2014 from http://water.epa.gov/polwaste/sediments/cs/upload/freshmanual.pdf.

9.0 FISH TISSUE FILLET (GREAT LAKES)

Laboratory Methods incorporated in EPA OST Manuals/QAPP.

10.0 MERCURY IN FISH TISSUE PLUGS

10.1 Summary of the Procedure

This procedure is applicable to the analysis of mercury in fish tissue plugs. The method is performance based. Laboratories may use any method that meets the requirements below to analyze the fish tissue samples (for example, EPA Method 1631). Example SOPs are provided in Appendix D of this LOM.

10.2 General Requirements for Laboratories

Competency. To demonstrate its competency, the laboratory shall provide EPA with performance data demonstrating their proficiencies in analyzing water quality samples. In addition, the laboratory must provide one or more of the following:

- Memorandum that identifies the relevant services that the laboratory provided for the National Aquatic Resource Surveys in the past five years.
- Documentation detailing the expertise of the organization, including professional certifications for water-related analyses, membership in professional societies, and experience with analyses that are the same or similar to the requirements of this method.

Also, the lab must provide a demonstration of past experience with fish tissue samples in applying the laboratory SOP in achieving the method detection limit.

Quality assurance and quality control requirements.
To demonstrate its expertise in quality assurance and quality control procedures, the organization shall provide EPA with copies of the quality-related documents relevant to the procedure. Examples include Quality Management Plans (QMP), QAPPs, and applicable Standard Operating Procedures (SOPs).

To demonstrate its ongoing commitment, the person in charge of quality issues for the organization shall sign the NCCA QAPP Certification Page.

10.2.1 Personnel

Laboratory Technician: This procedure may be used by any laboratory technician who is familiar with the NCCA Quality Assurance Project Plan, and this procedure in the NCCA Laboratory Operations Manual.

10.2.2 Equipment/Materials

The analytical method, selected by the laboratory, identifies the necessary equipment.

10.3 Sample Receipt

Because EPA initiates tracking procedures designed to recover any missing shipment, the laboratory personnel responsible for tracking samples must start the following login steps within 24 clock hours of receiving a delivery.

1. Report receipt of samples in the NARS IM sample tracking system (within 24 clock hours). Alternatively, for shipments with a large number of samples, the laboratory may email a spreadsheet with the sample login and sample condition information to NARS-IM (see Chapter 2 for contact information).

2. Inspect each sample THE SAME DAY THEY ARE RECEIVED:
   a. Verify that the sample IDs in the shipment match those recorded on the:
      i. Chain of custody forms when the batching laboratory sends the samples to the microcystins laboratory; or
      ii. Sample tracking form if the field crew sends the shipment directly to the State laboratory.
   b. Record the information in Table 10.1 into NARS IM, including the Condition Code for each sample:
      i. *OK*: Sample is in good condition
      ii. *C*: Sample container was cracked
      iii. *L*: Sample container is leaking
      iv. *ML*: Sample label is missing
      v. *VT*: Volume not sufficient for testing
      vi. *W*: Sample is warm (>8^{o}), record the temperature in the comment field, and perform the assay
      vii. *Q*: other quality concerns, not identified above.
   c. If any sample is damaged or missing, contact the EPA HQ Laboratory Review Coordinator to discuss whether the sample can be analyzed. (See contact information in Chapter 2 of the Manual).

3. Store samples in the freezer until sample preparation begins.

4. Maintain the chain of custody or sample tracking forms with the samples.

Table 10.1 Fish Tissue Plugs Login: Required Data Elements

FIELD	FORMAT	DESCRIPTION
LAB ID	text	Name or abbreviation for QC laboratory
DATE RECEIVED	MMDDYY	Date sample was received by lab
SITE ID	text	NCCA site id as used on sample label
VISIT NUMBER	numeric	Sequential visits to site (1 or 2)
SAMPLE ID	numeric	Sample id as used on field sheet (on sample label)
DATE COLLECTED	MMDDYY	Date sample was collected
CONDITION CODE	text	Condition codes describing the condition of the sample upon arrival at the laboratory.

FIELD	FORMAT	DESCRIPTION	
		Flag	Definition
		OK	Sample is in good condition
		C	Sample container is cracked
		L	Sample or container is leaking
		ML	Sample label is missing
		VT	Volume or mass not sufficient for testing (VT)
		W	Sample is warm (>8°)
		Q	Other quality concerns, not identified above
CONDITION COMMENT	text	Comments about the condition of the sample. If the condition code='W' then provide the temperature	

10.4 Quality Measures

This section describes the quality assurance and quality control measures used to ensure that the data will meet NCCA's requirements. Tables 10.2 and 10.3 provide a summary of the measurement data quality objectives and quality control requirements.

10.4.1 Assistance Visits

Assistance visits are intended to familiarize EPA with actual procedures being implemented by different laboratories; and to ensure a clear and consistent understanding of procedures and activities by both EPA and the laboratories. If EPA decides to conduct an assistance visit, a qualified EPA scientist or contractor will administer a checklist based upon the steps described in this chapter.

10.4.2 QC Samples

Once or twice during the performance period, the External QC Coordinator will provide one or two identical sets of QC samples to all participating laboratories. Each laboratory will run the QC samples following the same procedures used for the other samples. The External QC Coordinator will compare the results to the expected value to determine whether the values are within expected ranges. The contractor shall analyze the external QC samples using the same procedures as those for the field samples.

Table 10.2 Measurement data quality objectives

Variable or Measurement	MDL	Quantitation Limit
Mercury	0.47 ng/g	5.0 ng/g

Table 10.3 Quality Control

Activity	Evaluation/Acceptance Criteria	Corrective Action
Demonstrate competency for analyzing fish samples to meet the performance measures	Demonstration of past experience with fish tissue samples in applying the laboratory SOP in achieving the method detection limit	EPA will not approve any laboratory for NCCA sample processing if the laboratory cannot demonstrate competency. In other words, EPA will select another laboratory that can demonstrate competency for its NCCA samples.
Check condition of sample when it arrives.	Sample issues, such as punctures or rips in wrapping; missing label; temperature; adherence to holding time requirements; sufficient volume for test. All samples should arrive at the laboratory frozen.	Assign an appropriate condition code.
Store sample appropriately. While stored at the laboratory, the sample must be kept at a maximum temperature of -20^{o} C.	Check the temperature of the freezer per laboratory's standard operating procedures.	Record temperature of sample upon arrival at the laboratory. If at any other time, samples are warmer than required, note temperature and duration in comment field.
Analyze sample within holding time	The test must be completed within the holding time (i.e., 1 year). If the original test fails, then the retest also must be conducted within the holding time.	Perform test, but note reason for performing test outside holding time. EPA expects that the laboratory will exercise every effort to perform tests before the holding time expires.
Maintain quality control specifications from selected method/SOP (that meets the measurement data quality objectives)	Data meet all QC specifications in the selected method/SOP.	If data do not meet all QC requirements, rerun sample or qualify data. If the lab believes the data are to be qualified without rerunning sample, the lab must consult with the EPA Survey QA Lead before proceeding.
Maintain the required MDL	Evaluate for each sample	If MDL could not be achieved, then provide dilution factor or QC code and explanation in the comment field.
Use consistent units for QC samples and field samples	Verify that all units are provided in wet weight units and consistently	If it is not possible to provide the results in the same units as most other analyses, then assign a QC code and describe the reason for different units in the comments field of the database.
Maintain completeness	Completeness objective is 95% for all parameters.	Contact the EPA Survey QA Lead immediately if issues affect

Activity	Evaluation/Acceptance Criteria	Corrective Action
		laboratory's ability to meet completeness objective.

11.0 FECAL INDICATOR: ENTEROCOCCI

Laboratory methods incorporated into EPA ORD Manuals/QAPP.

12.0 ALGAL TOXINS, RESEARCH INDICATOR

See Appendix C for USGS Organic Geochemistry Research Laboratory (OGRL) Standard Operating Procedures as modified for use in NCCA 2015 relating to the Algal Toxins Research Indicator.

- **Appendix C.1 OGRL-SOP-5400 (as modified for use in NCCA 2015)**: Analysis of Cyanotoxins and Algal Toxins in Fresh and Marine Surface Water, Accumulations, and Blooms (Internal Standard Calibration of Standard Addition) – LCTX
- **Appendix C.2 OGRL-SOP-4520**: Sequential Freeze/Thaw Cell-Lysis Procedure for Total and Dissolved Algal Toxin Analysis of Water Samples
- **Appendix C.3 OGRL-2010**: Data and Information Backup for all OGRL Instruments

APPENDIX A: LABORATORY REMOTE EVALUATION FORMS

Email the completed and signed forms to Kendra Forde (forde.kendra@epa.gov).
Questions: Contact Kendra Forde at forde.kendra@epa.gov or 202-566-0417.

NCCA 2015 Document Request Form – Chemistry Laboratories

EPA and its state and tribal partners will conduct the 2015 National Coastal Condition Assessment. NCCA is a survey of the nation's coastal waters and Great Lakes. It is designed to provide statistically valid regional and national estimates of the condition of coastal waters and the Great Lakes. Consistent sampling and analytical procedures ensure that the results can be compared across the country.

As part of the 2015 NCCA, the Quality Assurance Team has been requested to conduct a technical assessment to verify quality control practices in your laboratory and its ability to perform chemistry analyses under this project. Our review will be assessing your laboratory's ability to receive, store, prepare, analyze, and report sample data generated under EPA's 2015 NCCA.

The first step of this assessment process will involve the review of your laboratory's certification and/or documentation. Subsequent actions may include (if needed): reconciliation exercises and/or a site visit. All labs will need to complete the following forms:

All laboratories will be required to complete the following forms and check the specific parameter in which your laboratory will be conducting an analysis for the 2015 NCCA:

- ☐ Water Chemistry and chlorophyll *a* (all of the analytes identified in the LOM and QAPP)
- ☐ Microcystin
- ☐ Mercury in Fish Tissue Plugs
- ☐ Sediment Chemistry
- ☐ Grain Size
- ☐ Total Organic Carbon (TOC)

If your lab has been previously approved within the last 5 years for the water chemistry indicator:

- ☐ A *signature* on the attached Laboratory Signature Form indicates that your laboratory will follow the quality assurance protocols required for chemistry labs conducting analyses for the 2015 NCCA.
- ☐ A signature on the Quality Assurance Project Plan (QAPP) and the Laboratory Operations Manual (LOM) Signature Form indicates that you will follow both the QAPP and the LOM.

If you have not been approved within the last 5 years through the laboratory verification process for the water chemistry indicator, in order for us to determine your ability to participate as a laboratory in the NCCA, we are requesting that you submit the following documents (if available) for review:

- ☐ Documentation of a successful *quality assurance audit* from a prior National Aquatic Resource Survey (NARS) that occurred within the last 5 years.
- ☐ Documentation showing participation in a previous NARS for Water Chemistry for the same parameters/methods.

Additionally, we request that all labs provide the following information in support of your capabilities, (these materials are required if neither of the two items above are provided):

- ☐ A copy of your laboratory's *accreditations and certifications* if applicable (i.e. NELAC, ISO, state certifications, NABS, etc.).
- ☐ An updated copy of your laboratory's *QAPP* and Laboratory Quality Assurance Manuals
- ☐ *Standard Operating Procedures* (SOPs) for your laboratory for each analysis to be performed (if not covered in 2015 NCCA LOM).
- ☐ Documentation attesting to experience running all analytes for the 2015 NCCA, including chlorophyll *a*.

Laboratory Signature Form – Chemistry Laboratories

I _____ certify that the laboratory, located in _____, will abide by the following standards in performing the following data analysis and reporting for the 2015 National Coastal Condition Assessment (NCCA).
This applies to the _____ chemistry indicator.

1.) Use procedures identified in the 2015 NCCA Laboratory Operations Manual (or equivalent). If using equivalent procedures, please provide the procedures and obtain approval from EPA.
2.) Read and abide by the 2015 NCCA Quality Assurance Project Plan (QAPP) and related Standard Operating Procedures (SOPs).
3.) Have an organized IT tracking system in place for recording sample tracking and analysis data.
4.) Provide Quality Control (QC) data for internal QC check, on a quarterly basis.
5.) Provide data using the template provided on the NARS Sharefile.
6.) Provide data results in a timely manner. This will vary with the type of analysis and the number of samples to be processed. Sample data must be received no later than May 1, 2016 or as otherwise negotiated with EPA.
7.) Participate in a laboratory technical assessment or audit if requested by EPA NCCA staff (this may be a conference call or on-site audit).
8.) Agree to analyze for all parameters specified in the LOM for the appropriate indicator(s) identified above, including Chlorophyll-*a,* for water chemistry.

Signature _____ Date _____

NCCA 2015 Document Request Form - Biology Laboratories

EPA and its state and tribal partners will conduct the 2015 National Coastal Condition Assessment. NCCA is a survey of the nation's coastal waters and Great Lakes. It is designed to provide statistically valid regional and national estimates of the condition of coastal waters and the Great Lakes. Consistent sampling and analytical procedures ensure that the results can be compared across the country.

As part of the 2015 NCCCA, the Quality Assurance Team has been requested to conduct a technical assessment to verify quality control practices in your laboratory and its ability to perform biology analyses under this project. Our review will be assessing your laboratory's ability to receive, store, prepare, analyze, and report sample data generated under EPA's 2015 NCCA.

The first step of this assessment process will involve the review of your laboratory's certification and/or documentation. Subsequent actions may include (if needed): reconciliation exercises and/or a site visit.

All laboratories will be required to complete the following forms and check the specific parameter in which your laboratory will be conducting an analysis for the 2015 NCCA:

- ☐ Mercury in Fish Plugs
- ☐ Benthic Macroinvertabrates
- ☐ Sediment Toxicity

If your laboratory has been previously approved within the last 5 years for the specific parameters:

- ☐ A *signature* on the attached Laboratory Signature Form indicates that your laboratory will follow the quality assurance protocols required for biology laboratories conducting analyses for the 2015 NCCA.
- ☐ A signature on the Quality Assurance Project Plan (QAPP) and the Laboratory Operations Manual (LOM) Signature Form indicates you will follow both the QAPP and the LOM.

If you have not been approved within the last 5 years through the laboratory verification process for the specific parameters, in order for us to determine your ability to participate as a lab in the NCCA, we are requesting that you submit the following documents (if available) for review:

- ☐ Documentation of a successful *quality assurance audit* from a prior National Aquatic Resource Survey (NARS) that occurred within the last 5 years.
- ☐ Documentation showing participation in previous NARS for this particular indicator.

Additionally, we request that all labs provide the following information in support of your capabilities, (these materials are required if neither of the two items above are provided):

- ☐ A copy of your laboratory's *accreditations and certifications* if applicable (i.e. NELAC, ISO, state certifications, NABS, etc.).

- ☐ Documentation of NABS (or other) *certification* for the *taxonomists* performing analyses (if applicable).
- ☐ An updated copy of your Laboratory's *QAPP* and Laboratory Quality Assurance Manuals.
- ☐ *Standard Operating Procedures* (SOPs) for your lab for each analysis to be performed (if not covered in 2015 NCCA LOM).

Laboratory Signature Form – Biology Laboratories

I _____ certify that the laboratory
located in _____, will abide by the following standards in performing biology data analysis and reporting for the 2015 National Coastal Condition Assessment (NCAA).
This applies to the _____ biological indicator.

Use procedures identified in the 2015 NCCA Lab Operations Manual (or equivalent). If using equivalent procedures, please provide the procedures and obtain approval from EPA.
Read and abide by the 2015 NCCA Quality Assurance Project Plan (QAPP) and related Standard Operating Procedures (SOPs).
Have an organized IT tracking system in place for recording sample tracking and analysis data.
Use taxonomic standards outlined in the 2015 NCCA Laboratory Operations Manual.
Participate in taxonomic reconciliation exercises during the field and data analysis season, which include conference calls and other laboratory reviews.
Provide Quality Control (QC) data for internal QC checks, including for sorting, on a monthly basis.
Provide data using the template provided on the NARS Sharefile.
Provide data results in a timely manner. This will vary with the type of analysis and the number of samples to be processed. Sample data must be received no later than May 1, 2016 or as otherwise negotiated with EPA. Samples results for independent taxonomic QC described in the LOM and QAPP must be provided to EPA prior to final datasets to allow for reconciliation to take place.
Participate in a Laboratory technical assessment or audit if requested by EPA NCCA staff (this may be a conference call or on-site audit).
Agree to utilize taxonomic nomenclature and hierarchical established for NCCA 2015.

Signature _____ Date _____

APPENDIX B: TARGET FISH SPECIES FOR WHOLE FISH ANALYSES

Table B.1 Northeast region primary and secondary marine target species - whole body fish tissue collection (Ecofish)

NORTHEAST REGION PRIMARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Ictaluridae	*Ameiurus catus*	White catfish	Primary
	Ictalurus punctatus	Channel catfish	Primary
Moronidae	*Morone americana*	White perch	Primary
Paralichthyidae	*Paralichthys dentatus*	Summer flounder	Primary
Pleuronectidae	*Pseudopleuronectes americanus*	Winter flounder	Primary
Sciaenidae	*Cynoscion regalis*	Gray weakfish	Primary
	Sciaenops ocellatus	Red drum	Primary
Sparidae	*Stenotomus chrysops*	Scup	Primary
NORTHEAST REGION SECONDARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Achiridae	*Trinectes maculatus*	Hogchoaker	
Anguillidae	*Anguilla rostrata*	American eel	Secondary
Atherinopsidae	*Menidia menidia*	Atlantic silverside	
Batrachoididae	*Opsanus tau*	Oyster toadfish	
Ephippidae	*Chaetodipterus faber*	Atlantic spadefish	
Moronidae	*Morone saxatilis*	Rock fish	Secondary
Mugulidae	*Mugil cephalus*	Black mullet	
Pomatomidae	*Pomatomus saltatrix*	Bluefish	Secondary
Sciaenidae	*Bairdiella chrysoura*	Silver perch	
	Menticirrhus saxatilis	Northern kingfish	
Serranidae	*Centropristis striata*	Black sea bass	
Triakidae	*Mustelus canis*	Smooth dogfish	
Triglidae	*Prionotus carolinus*	Northern searobin	
	Prionotus evolans	Striped searobin	

** Indicates whether species also occurs in the primary or secondary fish plug list*

Table B.2 Southeast region primary and secondary marine target species - whole body fish tissue collection (Ecofish)

SOUTHEAST REGION PRIMARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Ariidae	*Ariopsis felis*	Hardhead sea catfish	Primary
	Bagre marinus	Gafftopsail sea catfish	Primary
Paralichthyidae	*Paralichthys albigutta*	Gulf flounder	Primary
	Paralichthys dentatus	Summer flounder	Primary
	Paralichthys lethostigma	Southern flounder	Primary
Sciaenidae	*Cynoscion arenarius*	Sand weakfish (or seatrout)	Primary
	Cynoscion nebulosus	Speckled trout	Primary
	Cynoscion regalis	Gray weakfish	Primary
	Leiostomus xanthurus	Spot croaker	Primary
Sparidae	*Lagodon rhomboides*	Pinfish	
SOUTHEAST REGION SECONDARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Cichlidae	*Tilapia mariae*	Spotted tilapia	
Haemulidae	*Haemulon aurolineatum*	Tomtate	
Sciaenidae	*Bairdiella chrysoura*	Silver perch	
	Menticirrhus americanus	Southern kingfish	
Serranidae	*Centropristis striata*	Black sea bass	

** Indicates whether species also occurs in the primary or secondary fish plug list*

Table B.3 Gulf region primary and secondary marine target species - whole body fish tissue collection (Ecofish)

GULF REGION PRIMARY ECOFISH TARGET SPECIES			
FAMILY	SCIENTIFIC NAME	COMMON NAME	FISH PLUG LIST*
Ariidae	*Ariopsis felis*	Hardhead sea catfish	Primary
	Bagre marinus	Gafftopsail sea catfish	Primary
Paralichthyidae	*Paralichthys albigutta*	Gulf flounder	Primary
	Paralichthys dentatus	Summer flounder	Primary
	Paralichthys lethostigma	Southern flounder	Primary
Sciaenidae	*Cynoscion arenarius*	Sand weakfish (or seatrout)	Primary
	Cynoscion nebulosus	Speckled trout	Primary
	Cynoscion regalis	Gray weakfish	Primary
	Leiostomus xanthurus	Spot croaker	Primary
	Micropogonias undulatus	Atlantic croaker	Primary
	Sciaenops ocellatus	Red drum	Primary
Sparidae	*Lagodon rhomboides*	Pinfish	
GULF REGION SECONDARY ECOFISH TARGET SPECIES			
FAMILY	SCIENTIFIC NAME	COMMON NAME	FISH PLUG LIST*
Carangidae	*Caranx hippos*	Crevalle jack	
	Chloroscombrus chrysurus	Atlantic bumper	
Diodontidae	*Chilomycterus schoepfii*	Burrfish	
Gerreidae	*Eucinostomus gula*	Silver jenny	
Haemulidae	*Orthopristis chrysoptera*	Pigfish	
Ictaluridae	*Ictalurus furcatus*	Blue catfish	
Lepisosteidae	*Lepisosteus oculatus*	Spotted gar	
Lutjanidae	*Lutjanus griseus*	Gray snapper	
Sciaenidae	*Pogonias cromis*	Black drum	
Serranidae	*Diplectrum formosum*	Sand perch	
Triglidae	*Prionotus scitulus*	Leopard searobin	

** Indicates whether species also occurs in the primary or secondary fish plug list*

Table B.4 Western region primary and secondary marine target species - whole body fish tissue collection (Ecofish)

WESTERN REGION PRIMARY ECOFISH TARGET SPECIES			
FAMILY	SCIENTIFIC NAME	COMMON NAME	FISH PLUG LIST*
Atherinopsidae	*Atherinops affinis*	Topsmelt silverside	
Cottidae	*Leptocottus armatus*	Pacific staghorn sculpin	Primary
	Oligocottus rimensis	Saddleback sculpin	
Cynoglossidae	*Symphurus atricaudus*	California tonguefish	
Embiotocidae	*Cymatogaster aggregata*	Shiner perch	Primary
	Embiotoca lateralis	Striped seaperch	Primary
Gasterosteidae	*Gasterosteus aculeatus*	Three-spined stickleback	
Paralichthyidae	*Paralichthys californicus*	California flounder	Primary
	Citharichthys sordidus	Pacific sanddab	Primary
	Citharichthys stigmaeus	Speckled sanddab	
Pleuronectidae	*Isopsetta isolepis*	Butter sole	
	Parophrys vetulus	English sole	Primary
	Psettichthys melanostictus	Pacific sand sole	
	Platichthys stellatus	Starry flounder	Primary
Sciaenidae	*Genyonemus lineatus*	White croaker	Primary
Serranidae	*Paralabrax nebulifer*	Barred sand bass	Primary
	Paralabrax maculatofasciatus	Spotted sand bass	Primary
WESTERN REGION SECONDARY ECOFISH TARGET SPECIES			
FAMILY	SCIENTIFIC NAME	COMMON NAME	FISH PLUG LIST*
Echinodermata/ Toxopneustidae	*Tripneustes gratilla* (Hawaii ONLY)	Collector urchin	
Batrachoididae	*Porichthys notatus*	Plainfin midshipman	
	Porichthys myriaster	Specklefin midshipman	

Chimaeridae	*Hydrolagus colliei*	Spotted ratfish	
Embiotocidae	*Amphistichus argenteus*	Barred surfperch	Secondary
Paralichthyidae	*Xystreurys liolepis*	Fantail sole	
Pleuronectidae	*Pleuronichthys guttulatus*	Diamond turbot	Secondary
	Microstomus pacificus	Dover sole	Secondary
	Lepidopsetta bilineata	Rock sole	
	Lyopsetta exilis	Slender sole	
Sciaenidae	*Umbrina roncador*	Yellowfin croaker	

** Indicates whether species also occurs in the primary or secondary fish plug list.*

Table B.5 Great Lakes primary and secondary target species - whole body fish tissue collection (Ecofish)

GREAT LAKES PRIMARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Catostomidae	*Moxostoma macrolepidotum*	Shorthead redhorse	Primary
Centrarchidae	*Ambloplites rupestris*	Rock bass	Primary
	Lepomis gibbosus	Pumpkinseed	Primary
	Lepomis macrochirus	Bluegill	Primary
	Micropterus dolomieu	Smallmouth bass	Primary
	Pomoxis annularis	White crappie	
	Pomoxis nigromaculatus	Black crappie	
Cottidae	*Cottus bairdii*	Mottled sculpin	
	Cottus cognatus	Slimy sculpin	
Cyprinidae	*Couesius plumbeus*	Lake chub	
	Cyprinus carpio	Common carp	Primary
	Pimephales notatus	Bluntnose minnow	
Esocidae	*Esox lucius*	Northern pike	Primary
	Esox masquinongy	Muskellunge	Primary
Gasterosteidae	*Gasterosteus aculeatus*	Three-spined stickleback	
Gobiidae	*Neogobius melanostomus*	Round goby	
	Proterorhinus marmoratus	Tubenose goby	
Ictaluridae	*Ameiurus nebulosus*	Brown bullhead	Primary
	Ictalurus punctatus	Channel catfish	Primary
	Noturus flavus	Stonecat	
Gadidae	*Lota lota*	Burbot	Primary
Moronidae	*Morone americana*	White perch	Primary
	Morone chrysops	White bass	Primary
Osmeridae	*Osmerus mordax*	American/ rainbow smelt	
Percidae	*Gymnocephalus cernuus*	Ruffe	
	Perca flavescens	Yellow perch	Primary
	Percina caprodes	Logperch	
	Sander canadensis	Sauger	
	Sander vitreus	Walleye	Primary
Percopsidae	*Percopsis omiscomaycus*	Trout-perch	
Salmonidae	*Coregonus artedi*	Cisco/ lake herring	
	Coregonus clupeaformis	Lake whitefish	Primary
	Oncorhynchus gorbuscha	Pink salmon	
	Oncorhynchus kisutch	Coho salmon	Primary
	Oncorhynchus mykiss	Rainbow trout	Primary
	Oncorhynchus tshawytscha	Chinook salmon	Primary
	Salvelinus namaycush	Lake trout	Primary
Sciaenidae	*Aplodinotus grunniens*	Freshwater drum	Primary
GREAT LAKES SECONDARY ECOFISH TARGET SPECIES			
FAMILY	**SCIENTIFIC NAME**	**COMMON NAME**	**FISH PLUG LIST***
Catostomidae	*Catostomus catostomus*	Longnose sucker	
	Catostomus commersonii	White sucker	Secondary
	Moxostoma anisurum	Silver redhorse	
Centrarchidae	*Micropterus salmoides*	Largemouth bass	
Clupeidae	*Alosa pseudoharengus*	Alewife	
	Dorosoma cepedianum	American gizzard shad	
Cyprinidae	*Cyprinella spiloptera*	Spotfin shiner	
	Luxilus cornutus	Common shiner	
	Notropis stramineus	Sand shiner	
Esocidae	*Esox niger*	Chain pickerel	

Fundulidae	*Fundulus diaphanus*	Banded killifish	
	Fundulus majalis	Striped killifish	
Ictaluridae	*Ameiurus melas*	Black bullhead	
Salmonidae	*Prosopium cylindraceum*	Round whitefish	
	Salmo trutta	Brown trout	Secondary
	Salvelinus fontinalis	Brook trout	
	Salvelinus fontinalis x namaycush	Splake	

** Indicates whether species also occurs in the primary or secondary fish plug list*

APPENDIX C: ALGAL TOXINS RESEARCH INDICATOR STANDARD OPERATING PROCEDURES

Appendix C.1

Title:	Identifier:	Revision:	Effective Date:
Analysis of Cyanotoxins and Algal Toxins in Fresh and Marine Surface Water, Accumulations, and Blooms (Internal Standard Calibration or Standard Addition) – LCTX (As modified for NCCA 2015)	*OGRL-SOP-5400*	**2**	**8/31/2015**

USGS
science for a changing world

APPROVALS FOR USE

Author's Name (Print):	Author's Signature:	Date:
Keith A. Loftin		8/31/2015
Project Director's Name (Print) *Michael T. Meyer*	Project Director's Signature	Date: 8/31/2015

Organic Geochemistry Research Laboratory (OGRL)

Table of Contents

Analysis of Cyanotoxins and Algal Toxins in Fresh Surface Water, Accumulations, and Blooms (Standard Addition)

NOTE: Laboratory personnel may produce paper copies of this procedure printed from the controlled document file. However, it is their responsibility to ensure that they are trained on and utilizing the current version of this procedure. The procedure author may be contacted if text is unclear.

This is a direct inject analytical method developed for the separation, detection, and quantitation of cyanotoxins and algal toxins in fresh and marine surface water, and cyanobacterial accumulations and blooms by standard addition. Separation and detection of algal toxins is made using multiple reaction monitoring (MRM) mode of a liquid chromatography triple quadrupole mass spectrometer (LC/MS/MS). Quantitation is accomplished by either internal standard calibration curve or single point standard addition described in this SOP at a level equivalent to 1.0 µg/L. Standard addition can be used exclusively or when matrix effects are greater than +/- 20% (28.3% RSD) of spiked concentration. Samples analyzed by this procedure at minimum should be filtered which would be analogous to a dissolved algal toxin concentration or lysed and filtered which would be analogous to a total algal toxin concentration.

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the preparation, separation, detection, and quantitation for 14 cyanotoxins and algal toxins by liquid chromatography tandem mass spectrometry (LC/MS/MS) at the USGS Organic Geochemistry Research Laboratory (OGRL) in Lawrence, KS. The LCTX procedure applies to the following compounds in water:

anatoxin-a (ANAA), cylindrospermopsin (CYLS), domoic acid (DMAC), microcystin-HtYR (MCHtYR), microcystin-LA (MCLA), microcystin-LF (MCLF), microcystin-LR (MCLR), microcystin-LW (MCLW), microcystin-LY (MCLY), microcystin-RR (MCRR), microcystin-WR (MCWR), microcystin-YR, (MCYR), nodularin-R (NODR), and okadaic acid (OKAC). Simetone is used as an internal standard and L-phenylalanine is used to differentiate anatoxin-a from environmental phenylalanine since they have similar MRM transitions (isobaric compounds) and elute near each other chromatographically.

1.3 The minimum reporting limit (MRL) and minimum detection level (MDL) is matrix and compound dependent. However, the MRL to date has ranged from 0.10 µg/L (0.10 ppb) to 0.30 µg/L (0.30 ppb) based on a 100 µL injection depending on toxin.

2.0 TRAINING

The Project Director is responsible for ensuring that all who perform the function(s) described in this SOP for the OGRL are familiar with the objectives of and properly trained in its procedures. In addition, lab technicians using this procedure must document that they have read and understand this procedure in their training folder.

3.0 DEFINITIONS

3.1 Liquid Chromatography (LC) — An analytical instrument that relies on the interaction of an analyte with a solid stationary phase contained in a column and a liquid mobile phase as it passes through the analytical column (column) carrying the analyte.

3.2 Triple Quadrupole Mass Spectrometer (MS/MS)—An analyte detector that can determine the mass of selected fragments and fragments of fragments. This detector is typically used in conjunction with a chromatographic technique.

3.3 LC/MS/MS—A hyphenated technique where a liquid chromatograph is used for analyte separation is connected to a tandem mass spectrometer as the detector.

3.4 Chromatogram—The data that is acquired from the LC/MS/MS.

3.5 Analyte—The compound of interest.

3.6 Internal Standard— A standard (preferably an isotope labeled version of the analyte(s) of interest when possible) that is spiked into all samples, blanks and calibration samples. This compound should not be present in the environment and is used to correct for variation in analytical processes or techniques.

3.7 Reagent Water—treated water (18.2 MΩ/cm, < 1 ppb Total Organic Carbon (TOC)) generated by the laboratory system at the OGRL.

3.8 Stock Standard—a known concentration of an individual compound dissolved in a known volume of solvent. Target concentration is usually 100 µg/mL but can be greater if sufficient standard is available with adequate solubility.

3.9 LCTX Working Standard Mix— a reagent water spiked with a known concentration of all cyanotoxins and algal toxins that are determined by this method. This does not include the internal standard, simetone.

3.10 Analytical column--A stainless steel column containing a solid, stationary phase used to aid in separation on the LC.

3.11 Mobile phase—The solvent or combination of solvents that carries the analyte through the analytical column that aid in separation on the LC.

3.12 CAS#--Reference number assigned by Chemical Abstract Services to a chemical.

3.13 SOP—Standard operating procedure.

3.14 MeOH—Methanol, LC/MS grade or better.

3.15 ACN—Acetonitrile, LC/MS grade or better.

3.16 Formic Acid—Concentrated formic acid, usually 90% or greater.

3.17 THF-Tetrahydrofuran, analytical grade or better.

3.18 LCTX—an acronym for the liquid chromatography/triple quadrupole mass spectrometer method of cyanotoxins and algal toxins**.**

3.19 PPE---Personal Protective Equipment

3.20 Electrospray positive mode (ES +) —An ionization mode of positive polarity used by the tandem mass spectrometer to aid in fragmentation of positive ions.

3.21 Electrospray negative mode (ES -) —An ionization mode of negative polarity used by the tandem mass spectrometer to aid in fragmentation of negative ions.

3.22 Multiple Reaction Monitoring (MRM) — The scan type used for detection and quantitation of a parent and corresponding daughter fragment of an analyte.

3.23 Processed Sample—For purposes of this SOP, this term means that a sample has at minimum been filtered (Dissolved Cyanotoxin Analysis) or lysed and filtered (Total Cyanotoxin Analysis).

4.0 PERSONNEL HEALTH AND SAFETY

Note: This SOP is to be used in conjunction with an approved Chemical Hygiene Plan. Also, consult the Chemical Hygiene Plan for information on and use of all PPE including nitrile gloves, safety glasses, and a lab coat should be worn especially when making stock standard solutions.

4.1 Acetontrile, methanol, or tetrahydrofuran should not come in contact with skin or eyes, be inhaled, or be swallowed. Contact lenses should not be worn when working with these chemicals. Should contact occur, immediately wash with water. To prevent inhalation, use a fume hood with a suitable face velocity and cover containers before transporting. If a person breathes large amounts of any of these chemicals, move the exposed person to the fresh air at once. If any of these chemicals has been swallowed, get medical attention immediately by calling 911.

4.2 Care should be taken when working with THF, being a cyclic ether, there is concern for peroxide formation. **Do not evaporate THF to dryness!** THF is typically shipped with an inhibitor to prevent peroxide formation. There is no need to remove the inhibitor as part of processes conducted in this SOP. Use as is.

4.3 Cyanotoxins and algal toxins, by their very nature, are naturally occurring poisons that must be handled with care. The compounds covered in this SOP have a variety of indications when exposure occurs and relevant concentrations are not well defined for humans. However, in lieu of human acute and chronic toxicity information, mouse bioassays have been used to set suggested exposure thresholds. The World Health Organization has also suggested guidelines for some toxins with respect to drinking water and recreational exposure and US EPA has published health advisory thresholds in finished drinking water for anatoxin-a, cylindrospermopsin, and microcystin-LR.

4.4 Leaks may occur in fittings due to the high operating pressure of the LC. Safety goggles should be worn to protect eyes from splash.

4.5 The column compartment is hot and precautions should be taken before handling columns or touching the walls of column compartment.

4.6 The spray chamber of the MS/MS is very hot, with temperatures in excess of 650°C, and must be allowed to cool before touching.

5.0 APPARATUS AND INSTRUMENTATION

5.1 Analytical balance—capable of accurately weighing 0.0500 g ± 0.0001 g.

5.2 Top loading balance—capable of accurately weighting 5.0 g ± 0.1 g

5.3 Auto pipettes--10-to 10,000-µL, variable-volume auto pipettes with disposable plastic tips (Rainin, Woburn, MA, or equivalent).

5.4 Mechanical vortex mixer.

5.5 Data acquisition system—computer and printer compatible with all systems.

5.6 Instrument Software – LC/MS/MS software used for acquisition and data reduction supplied by LC/MS/MS manufacturer.

6.0 CHEMICALS AND REAGENTS

6.1 Mobile phase A, 0.1 % formic acid in reagent water.

6.2 Mobile phase B, 0.1 % formic acid in a mixture of 50/50 (v/v) MeOH to ACN.

6.3 Active and passive needle rinse solution for LC—Mobile phase B.

6.4 Stock solutions of analytes– See attachment A.

6.5 Stock internal standard solution, simetone, as received from Chem Services, inc. dissolved in methanol.

6.6 An aqueous 5% tetrasodium ethylene diamine tetraacetic acid (EDTA) solution made in reagent water is added to samples to minimize metal chelation. Volume is dependent on data quality (e.g. higher metals content requires more EDTA.

7.0 PROCEDURE

Note: Deviations from SOPs must be recorded in an appropriate instrument or work log. Include the name of the person recording the deviation, date it occurred and type of deviations, and whether the deviation was corrected (if applicable).

7.1 Preparation of 100 µg/mL individual stock standard solutions of cyanotoxins and algal toxins.

7.1.1 It is critical that all work with concentrated standards be conducted in a properly functioning fume hood. Remove all other items from hood that are not necessary for the work of making the stock cyanotoxin and algal toxin standards prior to initiation of stock standard preparation. Place a sign on the hood before beginning work with toxins for other personnel to stay out of this hood until the sign is removed. The sign should read “Stay out until further notice! Cyanotoxin work in progress. Contact: “your name”, office number, and phone number with questions. This will be in effect for 24 hours from the conclusion of toxin work with concentrated standards and decontamination of surfaces with 50 % aqueous ethanol solution or 50 % aqueous isopropanol solution. All materials including paper towels, gloves, pipettes, and used pipette tips should be left in the hood for 24 hours also before being bagged, tied off, and disposed in the dumpster. Pipettes can be wiped down with 50 % isopropanol or ethanol solutions.

7.1.2 When working with toxins always where nitrile gloves, appropriate safety glasses. A lab coat is recommended or wash your hands and arms with soap and water upon conclusion of work or at breaks.

7.1.3 The mass of toxin received from the distributor may be difficult to observe in the vial since standards usually only have 25 to 250 µg of material. This typically results in a thin film that is clear to offwhite. All solutions therefore must be initially made in the original vial. Target volume of stock standards is 0.25 to 1.0 mL with the appropriate solvent. See attachment A for individual stock standard concentration and the appropriate solvent or solvent mixture. Final individual stock standard solution storage can be in screw cap vials from supplier. For those standards arriving with crimp caps and septa the individual stock standard solutions will need to be transferred to separate screw cap LC/MS/MS grade vials.

7.1.4 Unless a certificate of analysis is available regarding standard purity assume 100 % purity for now. There are few certified reference materials available for these toxins and there is not an independent testing lab to confirm purity separate from the manufacturer. Aliqouts of standards will be evaluated for purity and the final concentrations will be corrected at that point. (Usually purity is corrected for when making the standards, but correction is not possible in this case since purity is unknown until measured).

7.1.5 Add appropriate volume of diluents as listed in Attachment A for a given mass of toxin and vortex capped vial for a couple of minutes. **Keep standards covered in the dark as much as possible when not working with them!** Allow standards to sit at room temperature in the dark for approximately 5 minutes and re-vortex capped vials for approximately 2 minutes. Keep standards at room temperature for 5 more minutes. Record the stock standard concentration, lot number from the manufacturer, name of the preparer and the date prepared in the working standards notebook.

7.2 Preparation of 100 µg/L LCTX Working Standard Mix.

7.2.1 Add 50 µL of each 100 µg/mL toxin standard to a labeled 123 mL amber glass bottle. Weigh in 49.3 g of reagent water to the bottle. Cap and invert bottle. (**Note: Since this is a research method, the number of standards added to the mix may change over time. The mass of reagent water to add will decrease by the same volume as the total volume of toxin solution added.).** Each working standard mix should have a lot number connected to the individual 100 µg/mL individual stock standards. The specific information should be recorded in the working standards notebook (i.e. LCTX-WSM-001).

7.2.2 Divide the 100 µg/L LCTX Working Standard Mix into subaliqouts by placing 1.5 mL of the 100 µg/L LCTX Working Standard Mix into labeled screw capped LC vials. Make 10 LC vials at a time since they will last for quite a while. Each vial label should be labeled with a lot number that ties it back to the original 100 µg/L LCTX Working Standard Mix (i.e. LCTX-WSM-001a). Keep the remainder of the 100 µg/L LCTX Working Standard Mix in the original bottle (7.2.1) and remove from freezer for use only when the 1.5 mL aliquots have been used up and make 10 more subaliqouts. Store all 100 µg/L LCTX Working Standard Mixes in the appropriate standards freezer until use.

7.3 Preparation of the 1.23 mg/mL Stock Internal Standard Solution of Simetone.

7.3.1 Weigh 123 mg of Simetone (more if purity is not 100%) into 100mL of MeOH. Mix until simetone completely dissolved. Store in Stock Standards Freezer until needed.

7.4 Preparation of 1.23 mg/L Intermediate Internal Standard of Simetone.

7.4.1 Dilute 1 mL of 1.23 mg/mL Stock Internal Standard Simetone Solution with 999 mL of MeOH. Mix and store in the freezer when not in use.

7.5 Preparation of 0.123 mg/L Working Internal Standard of Simetone.

7.5.1 Dilute 10 mL of the 1.23 mg/L Intermediate Internal Standard of Simetone with 90 mL of reagent water. Mix and store in the freezer when not in use.

7.6 **Preparation of LCTX Internal Standard (LCTX ISTD)**.

7.6.1 Dilute 2.5 mL of the 0.123 mg/L Working Internal Standard with 2.5 mL of reagent water. Label as LCTX ISTD.

7.7 Preparation of Check Standards, Blanks, and Samples for LCTX Analytical Run.

7.7.1 Obtain current LCTX run sheet from Project Management Office or off the computer in ResLab (OGRL Computer Network). Each analytical run should consist of the following: Check Standards (2)- 1.0 µg/L, Blanks after each Check Standard and every standard addition (SA) sample, and unspiked (A) and spiked samples (SA-Standard Addition), and duplicate unspiked and spiked samples (at least 1 duplicate for every 10 samples). There should be 15 to 25 samples per analytical run. For every sample there will be two vials—one containing sample and LCTX ISTD solution and the other containing sample and LCTX SA solution.

7.7.2 Make labels for all Check Standards, Blanks, and Samples as indicated by the run sheet. Remember to make labels for the standard addition samples. All labels except for the Blanks go on microvials. Only two Blank labels will be needed per analytical run and those labels will be placed on 2 mL screw capped LC vials. Labels should have the sample name, LCTX Run number, date of preparation, and initials of personnel preparing the analytical run. Initials should be cross-walked to full names in log book.

7.7.3 Apply labels to all vials and organize by analytical run.

7.7.4 Preparation of Blanks

7.7.4.1 Place 2 mL of reagent water into a labeled 2 mL screw capped LC vial. There should be a total of two vials with Blank solution per analytical run.

7.7.5 Preparation of Samples

7.7.5.1 Invert all samples 3 times before pipetting.

7.7.5.2 Pipette 1.5 mL of filtered sample into a glass LC/MS/MS vial and cap.

7.8 Make sure the Source of the MS/MS is clean. If source is not cleaned and you are not trained ask your supervisor for training.

7.9 Mobile Phase Preparation for LC

7.9.1 Preparation of Mobile Phase A: 0.1 % Formic acid in reagent water.

7.9.1.1 Add 2 mL of concentrated formic acid to 2 L of reagent water. Cap and invert 3 times. pH will be approximately 2.6 to 2.7 typically.

7.9.1.2 Place on channel A of the LC.

7.9.2 Preparation of Mobile Phase B: 0.1 % Formic acid in 50/50 Methanol/Acetonitrile.

7.9.2.1 Add 2 mL of concentrated formic acid to 1 L of LC/MS/MS grade or better methanol and 1 L of LC/MS/MS grade or better acetonitrile. Cap and invert 3 times.

7.9.2.2 Place on channel B of the LC.

7.9.3 Preparation of Mobile Phase C: Reagent Water.

7.9.3.1 Add 2 L of reagent water into a 2 L mobile phase bottle.

7.9.3.2 Place on channel C of the LC.

7.9.4 Preparation of Mobile Phase D: 50/50 Methanol/Acetonitrile.

7.9.4.1 Add 1 L of LC/MS/MS grade or better methanol to 1 L of LC/MS/MS grade or better acetonitrile in a 2 L mobile phase bottle.

7.9.4.2 Place on channel D of the LC.

7.10 Make sure all frits, guard cartridge, and analytical column are in place for LCTX. Analytical column is a Waters Corp. Atlantis T3 analytical column. A Waters Corp. Atlantis dC18 analytical column can also be substituted resulting in slight changes in analyte retention time. The guard cartridge is a Waters Atlantis dC18 cartridge.

7.11 Prime the LC pumps by opening the purge valves, setting flow rate at 2 to 5 mL/min proportion at 25% for each of the 4 mobile phases. Let prime for at least 5 minutes. Change flow to 95% A and 5% B to reflect starting conditions of separation for 5 minutes. When priming is finished, reduce flowrate back to initial flowrate conditions of LCTX method (usually around 0.7 mL/min), and close purge valve LC pump.

7.12 This is a performance based method and is suitable for any bioinert LC/MS/MS system as long as quality control criteria are met. SOP is written currently for an Agilent 1260 bioinert LC/6460 triple quadrupole mass spectrometer with a jet stream source attached. Multimode sources are suitable as well when used in electrospray mode only. The LCTX method is adapted from Loftin et al., 2008 and Graham et al., 2010.

7.12.1 Defragment partitioned hard drive of instrument computer weekly.

7.12.2 Open Agilent MassHunter Acquisition software.

7.12.3 Open the current LCTX Project

7.12.4 Open an old LCTX worklist from a previous run and resave with the current analytical run number and date.

7.12.5 Enter the correct sample names and save the batch (see appendix B for example layout). Check that the correct acquisition method is being used.

7.12.6 Recheck worklist for typographical errors. Resave if any changes.

7.12.7 Place vials in appropriate position in autosampler tray (as shown in appendix B unless project scientists requests a change). Blanks go in vial 1 and vial 2 slots, internal standard solution in vial 3, and standard addition solution is 100 µg/L calibration standard. Place QC, calibration standards, and samples in order in well plate trays starting with P1-A1 according to worklist.

7.13 Equilibration of LC/MS/MS

7.13.1 Open the LCTX method.

7.13.2 Start the worklist in multiple vial mode.

7.13.3 Run the first 3 injections of the worklist as 1 µg/L control standards. If using a new column, then may need to run up to 6 injections to equilibrate column.

7.13.4 Evaluate retention time stability, peak shape and abundance. Values should be within 60 seconds, consistent peak shape based on historical data, and within 30% of historical abundance, respectively.

7.13.5 If data is not consistent, then begin troubleshooting which may include:

7.13.5.1 Check that LC backpressure is within typical ranges.

7.13.5.2 Make sure purge valve is closed.

7.13.5.3 Check for leaks.

7.13.5.4 Check that spray from electrospray needle is positioned correctly and has a concentric spray.

7.13.5.5 Infuse a standard in the MS/MS to check MS/MS performance.

7.13.5.6 Notification of supervisor as needed and remedial action to correct instrument performance.

7.14 Submission of Worklist.

7.14.1 If control standards data looks comparable between injections, then proceed with worklist. Control standards should be within +/- 20% of expected concentration or abundance.

7.14.2 Verify periodically that internal standard, blanks, controls, and standard addition samples look appropriate. Confirm that peak shapes and retention times are consistent compared to historical analysis runs (e.g. retention times within 1 minute of historical value unless method needs to be modified with approval from supervisor. If not, remedy the problem following the troubleshooting steps in Section 7.13.5.

7.15 Post Run Instrument Clean-Up.

7.15.1 The last line of the worklist should include a blank injection using the LCTX clean method. This will use mobile phases C and D to clean any residual traces due to sample matrix out of the column under the clean conditions which are at a LC higher temperature. No acid modifier is added to mobile phases C and D for proper column storage.

7.16 Data Reduction with Agilent MassHunter Quantitation software

7.16.1 Once data has been acquired, open the Agilent MassHunter Quantitation software.

7.16.2 Create a new Batch and load newly acquired data from worklist into the batch.

7.16.3 Load the appropriate LCTX quant method.

7.16.4 Edit the LCTX quant method to update retention times and MRM ratios as necessary using a mid to upper range calibration standard. Check integration of all compounds.

7.16.5 Save method with batch folder and process calibration data. Use linear or quadratic curve fits. 1/x weighting is permissible. R^2 values should be 0.98 or greater. Save when done.

7.16.6 Quantitate all samples

7.16.7 Evaluate calibration data, make sure blanks are blank below the minimum reporting level (MRL) of the method, duplicates and control standards are within +/- 20% (28.3% RSD) of expected concentration or abundance.

7.16.8 For Standard Addition Calculations, use the standard addition LCTX quant method or export the results table into a spreadsheet program such as Microsoft Excel.

7.16.9 When quantitation is complete, have a supervisory chemist provide quality control of the data set as described in Section 10.

7.16.10 Reanalysis of samples is necessary when quality control or instrument performance renders the data outside of acceptable QC metrics as established in Section 10 of the SOP, Table 5.11.1 of the NCCA 2015 QAPP and best professional scientific judgement by a supervisory chemist. When using standard addition for quantitation, check if concentrations prior to correction for dilution are greater than 2.5 µg/L. If so, dilute the original sample and reanalyze by standard addition as described in this SOP.

7.16.11 If any samples exhibit data quality issues confer with a supervisory chemist for evaluation of problem.

7.16.12 When data quality is deemed acceptable then store an electronic data analysis report for record keeping.

7.17 Refer to the 2010 OGRL SOP on backing up data for data archival.

8.0 REFERENCES

8.1 2010 Backing up Data

8.2 Toxic Cyanobacteria in Water: A guide to their public health consequences, monitoring and management. Chorus, I.; Bartram, J.; Eds., Spon Press: London, 1999.

8.3 Graham, J.L., Loftin, K.L., Meyer, M.T., Ziegler, A.C. 2010. Cyanotoxin mixtures and taste-and-odor compounds in cyanobacterial blooms from the Midwestern United States, *Environ. Sci. Technol.*, 44, 7361-7368.

8.4 Loftin, K.A., Meyer, M.T., Rubio, F., Kamp, L., Humphries, E., Whereat, E. 2008. Comparison of two cell lysis procedures for recovery of microcystins in water samples from

Silver Lake in Dover, Delaware, with microcystin producing cyanobacterial accumulations. USGS OFR 2008-1341, 9 p.

9.0 RECORDS AND ARCHIVAL

The person performing this SOP is responsible for submitting the following records to be archived to the Project Documents Archival manager or stored in the appropriate location in the laboratory (usually next to instrument computer).

9.1 Instrument Maintenance Log

9.2 Worklist Log

9.3 Tune files Log

9.4 Computer data files for each sample and control are stored, copied, backed up, and archived according to OGRL-SOP-2010.

10.0 QUALITY CONTROL

10.1 Supervisory chemist visually reviews QC data for each run or uses software to identify excursions from permissible results described in Sections 7.16.5 and 7.16.7, and MRLs listed in Table 5.11.1 of the NCCA 2015 QAPP.

10.1.1 Analytical control is maintained by the use of carryover blanks (COB), laboratory duplicates (DUP), and Control Standards (CCV).

10.1.2 Standard addition (spiked sample duplicate) results provide checks for and qualify matrix dependent shifts in retention times and Multiple Reaction Monitoring (MRM) ratios, and provide a basis for sample matrix-corrected results when responses deviate from expected (e.g. +/- 20% or 28.3% RSD).

10.1.3 Target analytes will be quantitated by internal standard curve unless matrix effects are greater than +/- 20%. Larger deviations will trigger reanalysis and quantitation by standard addition.

11.0 ATTACHMENTS

11.1 Appendix A: Analyte List

11.2 Appendix B: Instrument Worklist Example

11.3 Appendix C: LCTX LC/MS/MS method

11.4 Appendix D: LCTX Clean LC/MS/MS method

12.0 REVISIONS TO THIS SOP

Rev. 1 9/29/2008 Initial version

Rev. 2 8/31/2015

Appendix A: Analyte List

Toxin	Type	CAS#	Stock Concentration (µg/mL)	Diluent (solvent)
anatoxin-a	Cyanotoxin	64285-06-9	100	Water
cylindrospermopsin	Cyanotoxin	143545-90-8	100	Methanol
domoic Acid	Algal toxin	14277-97-5	100	Methanol
microcystin-HiLR	Cyanotoxin	NA	100	Methanol
microcystin-HtYR	Cyanotoxin	NA	100	Methanol
microcystin-LA	Cyanotoxin	96180-79-9	100	Methanol
microcystin-LF	Cyanotoxin	154037-70-4	100	Methanol
microcystin-LR	Cyanotoxin	101043-37-2	100	Methanol
microcystin-LW	Cyanotoxin	111755-37-4	100	Methanol
microcystin-RR	Cyanotoxin	111755-37-4	100	Methanol
microcystin-WR	Cyanotoxin	NA	100	Methanol
microcystin-YR	Cyanotoxin	101043-37-2	100	Methanol
nodularin-R	Cyanotoxin	118399-22-7	100	Methanol
okadaic acid	Algal toxin	78111-17-8	100	Methanol
L-phenylalanine	Amino acid	63-91-2	100	Water

Appendix B: Example Instrument Run Sheet Layout

Worklist Number	**Sample ID**	**Sample Type**	**Injection Volume (µL)**	**Standard**
1	1 µg/L LCTX Standard Mix a	Column Equilibration Sample	1	100 µg/L LCTX Standard Mix
2	1 µg/L LCTX Standard Mix b	Column Equilibration Sample	1	100 µg/L LCTX Standard Mix
3	1 µg/L LCTX Standard Mix c	Column Equilibration Sample	1	100 µg/L LCTX Standard Mix
4	Blank 1	Instrument Blank	0	Blank
5	0.001 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	100	0.10 µg/L LCTX Standard Mix
6	0.010 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	1	1 µg/L LCTX Standard Mix
7	0.030 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	3	1 µg/L LCTX Standard Mix
8	0.050 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	5	1 µg/L LCTX Standard Mix
9	0.080 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	8	1 µg/L LCTX Standard Mix
10	0.10 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	10	1 µg/L LCTX Standard Mix
11	0.25 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	25	1 µg/L LCTX Standard Mix
12	0.50 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	50	1 µg/L LCTX Standard Mix
13	0.75 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	75	1 µg/L LCTX Standard Mix
Worklist Number	**Sample ID**	**Sample Type**	**Injection Volume (µL)**	**Standard**

14	1 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	1	100 µg/L LCTX Standard Mix
15	5 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	5	100 µg/L LCTX Standard Mix
16	8 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	8	100 µg/L LCTX Standard Mix
17	10 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	10	100 µg/L LCTX Standard Mix
18	25 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	25	100 µg/L LCTX Standard Mix
19	50 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	50	100 µg/L LCTX Standard Mix
20	75 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	75	100 µg/L LCTX Standard Mix
21	99 µg/L LCTX Standard Mix	Internal Standard Curve Calibration	99*	100 µg/L LCTX Standard Mix
22	Blank 2	Instrument Blank	0	Blank
23	NCC-439871 A	Sample	99	
24	NCC-439872 A	Sample	99	
25	NCC-439873 A	Sample	99	
26	NCC-439874 A	Sample	99	
27	NCC-439875 A	Sample	99	
28	NCC-439876 A	Sample	99	
29	NCC-439877 A	Sample	99	
30	NCC-439878 A	Sample	99	
Worklist Number	**Sample ID**	**Sample Type**	**Injection Volume (µL)**	**Standard**
31	NCC-439880 A	Sample	99	
32	NCC-439881 A	Sample	99	

33	NCC-439871 L	Instrument Duplicate	99	
34	NCC-439881 SA	Spiked Sample Duplicate	99 - sample 1 - 100 µg/L LCTX Standard Mix	
35	Blank 3	Instrument Blank	0	Blank
36	1 µg/L LCTX Standard Mix CC1	Calibration Check	1	100 µg/L LCTX Standard Mix
37	Blank 4	Instrument Blank	0	Blank
38	NCC-439882 A	Sample	99	
39	NCC-439883 A	Sample	99	
40	NCC-439884 A	Sample	99	
41	NCC-439885 A	Sample	99	
42	NCC-439886 A	Sample	99	
43	NCC-439887 A	Sample	99	
44	NCC-439888 A	Sample	99	
45	NCC-439889 A	Sample	99	
46	NCC-439890 A	Sample	99	
47	NCC-439891 A	Sample	99	
48	NCC-439882 L	Instrument Duplicate	99	
49	NCC-439891 SA	Spiked Sample Duplicate	99 - sample 1 - 100 µg/L LCTX Standard Mix	
50	Blank 5	Instrument Blank	0	Blank
Worklist Number	**Sample ID**	**Sample Type**	**Injection Volume (µL)**	**Standard**
51	1 µg/L LCTX Standard Mix CC2	Calibration Check	1	100 µg/L LCTX Standard Mix
52	Blank 6	Instrument Blank	0	Blank

1 Only 99 µL is injected to accommodate a 1 µL stacked injection of internal standard (simetone) for 100 µL injection loops. 100 µL of standard mix can be injected on larger injection loops.

2 L = instrument sample duplicate

3 SA = Spiked Sample Duplicate. Final concentration can be modified by changing the injection volume of the standard spiked. This example shows a 1.0 µg/L equivalent final concentration.

Appendix C.2

Title: *Sequential Freeze/Thaw Cell-Lysis Procedure for Total and Dissolved Algal Toxin Analysis of Water Samples*	Identifier: *OGRL-SOP-4520*	Revision: **2**	Effective Date: **1/18/2016**

APPROVALS FOR USE		
Author's Name (Print): *Keith A. Loftin*	Author's Signature:	Date: *01/18/16*
Project Director's Name (Print) *Mike T. Meyer*	Project Director's Signature	Date: **01/22/16**
Organic Geochemistry Research Laboratory (OGRL)		

STANDARD OPERATING PROCEDURE

PROCESSING WATER SAMPLES FOR ALGAL TOXIN ANALYSIS

Table of Contents

Processing Water Samples for Microcystin Analysis

NOTE: Laboratory personnel may produce paper copies of this procedure printed from the controlled document file. However, it is their responsibility to ensure that they are trained on and utilizing the current version of this procedure. The procedure author may be contacted if text is unclear.

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the sequential freeze/thaw cell-lysis process for preparing water samples for algal toxin analysis at the USGS Organic Geochemistry Research Group (OGRL) Laboratory in Lawrence, KS.

1.2 *Algal toxins* are toxins produced and released by phytoplankton. These algal blooms can be extremely toxic to many different species of birds and mammals (including humans).

1.3 This Standard Operating Procedure (SOP) describes the cell-lysis process of freezing and thawing water samples as a means to lyse the algal cells and release of algal toxins for analysis.

2.0 TRAINING

The OGRL Director or designee is responsible for ensuring that all who perform the functions described in this SOP for the OGRL are familiar with the objectives of and properly trained in its procedures. In addition, one must document that they have read and understand this procedure in their training folder.

3.0 DEFINITIONS

3.1 *Total Algal Toxins*—For purposes of this SOP, this term refers cell-lysis of all phytolankton in a given water sample followed by filtration to remove particulates.

Cell-lysis results in intracellular algal toxins transferred to the dissolved phase of the water sample.

3.2 *Dissolved Algal Toxins*—For purposes of this SOP, this term refers to filtration to remove particulates of a given water sample. Given that this sample is filtered in the absence of artificial (laboratory induced) cell-lysis, the algal toxins measured in the water sample do not represent intracellular algal toxins, but dissolved-phase algal toxins from naturally lysed algae.

3.3 *Frozen Water Sample*—A water sample that has been placed overnight in a freezer and is frozen completely through.

3.4 *Thawing Water Sample*—A water sample that has been removed from a freezer to thaw protected from light by aluminum foil.

3.5 *Thawed Water Sample*—A water sample that contains no ice and is composed only of liquid.

3.6 *Filtering*—The process of forcing a sample through a filter to remove particulates.

3.7 *Sample ID*—Each sample in a defined project will have a unique ID that is generally five digits long with a letter.

3.8 *Project Code*—This is the three digit code noted on all sample labels. It is unique and informs the lab employees which project the sample is a part of.

3.9 *Project Title*—This is the title of the project. It will generally include information such as the purpose of the study and who is concerned with the results. An example would be 'EPA Lake Assessment.'

3.10 *Reslab*—This is the name of the shared network used by all members of the Organic Geochemistry Research Laboratory.

4.0 PERSONNEL HEALTH AND SAFETY

4.1 **Note:** This SOP is to be used in conjunction with an approved Chemical Hygiene Plan. Also, consult the Chemical Hygiene Plan for information on and use of all personal protective equipment (PPE).

4.2 **Toxins:** The nature of this work can expose an individual to algae and algal toxins if appropriate standard safety protocols are not followed. Notify supervisor when initiating work with environmental samples that may contain toxins and as always report any safety incidences at the earliest opportunity to the laboratory safety officer.

4.3 Always where gloves, at minimum safety glasses, work in the hood when possible and to the extent necessary. Do not ingest, inhale, get in eyes, or contact with skin. If contact with skin made then wash with copious amounts of soap and water. If eye contact made immediately use the eyewash station to rinse eyes then seek medical attention as necessary. For ingestion or inhalation, seek appropriate medical attention. The toxins are not known to be volatile, but can be aerosolized.

5.0 EQUIPMENT AND SUPPLIES

Descriptions of commonly used pieces of equipment, their advantages, and their limitations are listed below.

5.1 *Nitrile Gloves*- Required for handling all environmental samples potentially containing toxins.

5.2 *Freezer Space*- Space should be set aside for the water samples in a freezer with a temperature range less than or equal to –20˚C (+/-5 ˚C).

5.3 *Refrigerator Space*- Space should be set aside for the water samples in a refrigerator or the walk-in with a temperature range between 2˚C and 10˚C.

5.4 *Aluminum Foil*- Foil is used for covering the sinks full of thawing samples. **Algal toxins have been reported as light sensitive, it is necessary to cover all of the samples at all times!**

5.5 *Vacuum Filtration*-The process by which particulates are removed from samples by vacuum.

5.6 *Syringe Filtration*-The process by which particulate are removed from samples by use of syringe and filter.

5.7 *Clear LC/MS Screw Top Vials*- These are 2mL clear glass vials. One vial is needed for each sample. A fine tip permanent marker is used to print pertinent information onto the labeling sticker, which is attached to the vial.

5.8 *Screw Top Cap*- This blue cap is used to seal the clear screw top vial.

5.9 *1000 mL Beaker*- This beaker is used to collect unwanted water sample.

5.10 *Permanent Marker*- Used for labeling the 1000mL dump beaker.

5.11 *Labeling Tape*- Colored tape that is used to label the 1000mL beaker.

5.12 *Labeling Stickers*- Used for labeling the screw top vials during the filtration process.

5.13 *Fine Tip Permanent Marker*- Used for writing on labeling stickers during the filtration process.

5.14 *10mL pipette and pipette tips*- Used for transferring 10mL of the unfiltered sample to the syringe filter.

5.15 *Empty Chromacol Cardboard Box*- This box is used to store the chromacol vials in a freezer after processing is complete. A spreadsheet that includes a list of the vials being stored is placed inside the box. A label is also necessary on the outside of the box.

5.16 *Empty Vial File or Tray*- An item used to store all clear screw top vials belonging to a designated IMN run. It is labeled and stored in a freezer for future analysis and archival/storage.

5.17 *Clear tape*- This is used to affix printed labels to the chromacol vials.

5.18 *1 oz. amber glass bottle* – a sample storage container used for frozen storage/archival of sample filtrate.

6.0 PROCEDURE FOR FREEZE/THAW CYCLING

Note: Deviations from SOPs must be recorded in an appropriate instrument or work log. HDPE or Teflon sample bottles may be used in place of amber glass for selected projects at the initiation of a study. Additionally, different filter procedures may be used prior to the initiation of a study. These deviations from this SOP are not

acceptable after a study is initiated. Deviations to this procedure are subject to approval by the principle investigator.

6.1 If sample is to be processed for Dissolved Algal toxin analysis only skip to section 7.0. If sample is to be processed for both Total and Dissolved Algal toxin, then the sample will need to be homogenized by inversion of the sample at least 3 times. Split the homogenized sample in half and label each sample appropriately. Designate 1 bottle as Total and the other Dissolved. Take the sample split for Total Algal toxin analysis through the remaining Freeze/Thaw procedure starting with step 6.2. Skip to Section 7.0 of this SOP to begin processing for Dissolved Algal toxin analysis. Any glass container that will be frozen should be no more than half full of sample.

6.2 It is important to have as much communication between all personnel involved in the project as possible.

6.2.1 At the beginning of every shift, read through the “Log Note” left from the students who last worked on the project (see section 10.3.1.3).

6.2.2 Speak with the student login assistant and find out if any new samples have been received for the afternoon.

6.3 If there are new samples that have been logged in, ask the login assistant for the physical location of the samples. Also, find out if the samples have undergone the first freeze or freeze/thaw cycle (sometimes samples are stored frozen before shipment to OGRL and may or may not thaw during shipment).

6.4 Create a “Processing Spreadsheet” for the new samples (see section 9.1).

6.5 Each morning all samples from the freezer and refrigerator are thawed in an empty sink for the day.

6.5.1 Cover all samples with aluminum foil while in sink and do not have samples touching each other to allow air to circulate between the bottles.

6.6 Print out the “Sample Checklist” (see section 10.1) and note where all the samples are located in the cycling process.

6.6.1 To make the checklist easy to read, choose a different colored pen/highlighter to mark: the thawing samples, the samples that have been sent to the freezer for the next freeze cycle and the samples ready for filtration.

6.6.1.1 If the sample has just completed its first, then add 1 line by permanent marker to the lid. Repeat with a second line for the completion of the second freeze/thaw cycle, and a third line when the third freeze/thaw cycle is complete. Record dates of each freeze/thaw step in the spreadsheet for each sample.

6.6.1.2 If the sample has just completed its third thaw, it is ready for filtration and then vialing (see section 7.0). These samples will be kept in the refrigerator before filtration begins. If samples will not be filtered within 24 hours then do not do the third thaw until ready for filtration.

6.7 Make sure that all samples are accounted for and all spreadsheets are updated on the computer spreadsheet (see section 9.0).

6.8 All samples that are still thawing will be kept overnight in a refrigerator (Thawing time is very dependent on sample volume).

6.9 Samples that are completely thawed out will follow the sample procedure outlined in sections 6.5.

6.10 All spreadsheets must be updated and printed out for storage in the project binder located in the Project Management office (see section 9.0)

7.0 PROCEDURE FOR FILTERING/VIALING

7.1 One of two filtration techniques (vacuum filtration or syringe filtration) will be used on a set of project samples as indicated by the principle investigator or the project management office. Filter type and mesh size can be modified by the principle investigator to meet project needs, but changes should be recorded in sample spreadsheet.

7.2 Vacuum filtration

7.2.1 Get a clean 47 mm diameter 100 mL two-piece glass funnel and funnel clamp for each sample.

7.2.2 Assemble the filter assembly with a 0.7 micron, 47 mm diameter glass fiber filter in between the two filter pieces and clamp together.

7.2.3 Attach filter assembly to a clean 250 to 1000 mL side arm vacuum flask.

7.2.4 Connect vacuum flask with vacuum tubing to house vacuum.

7.2.5 Invert the capped sample bottle vigorously at least 3 times to homogenize sample.

7.2.6 Add approximately 30 mL of sample to funnel and apply vacuum until filter is dry.

7.2.7 All filtrate should be stored in clean glass vials for freezing. Glass vials should not be more than half full.

7.2.8 Make sure all vials have appropriate identifying information (e.g. sample ID, data, personnel, and “TF” for total and filtered or “DF” for dissolved and filtered.

7.2.9 Transfer 1 mL of filtered sample to a labeled 2 mL screw capped vial.

7.2.10 Store the remaining filtrate in

7.2.11 When finished filtering update all spreadsheets (see section 9.0).

7.3 Syringe Filtration

7.3.1 Attach an unused 25 mm, 0.7 micron glass fiber membrane syringe filter to an unused 10 mL HDPE syringe with luer lock fitting after removing syringe plunger.

7.3.2 Lay syringe plunger on a clean chemwipe.

7.3.3 Add 10 mL of sample to syringe barrel with syringe filter in place.

7.3.4 Replace removed syringe plunger back into syringe barrel and filter 1 mL of sample directly into labeled, glass 2 mL LC/MS vial. Cap vial.

7.3.5 Filter remaining sample, to larger 1 oz. amber glass bottle.

8.0 REFERENCES

Toxic Cyanobacteria in Water: A guide to their public health consequences, monitoring, and management. Eds. I. Chorus, J. Bartram, Spon Press: London, 1999.

Graham, J.L., Loftin, K.A., Meyer, M.T., Ziegler, A.C., 2010, Cyanotoxins mixtures and taste-and-odor compounds in cyanobacterial blooms from the midwestern United States, Environmental Science and Technology, 44, 7361-7368.

9.0 RECORDS AND ARCHIVAL

The person performing this SOP is responsible for submitting the following records to be archived to the Project Office Manager.

9.1 PROCESSING SPREADSHEETS (All spreadsheets are maintained in "ResLab" on the network.)

9.1.1 The processing sheets are important because they allow OGRL staff to keep track of the freeze/thaw cycling for each sample. They also indicate when each sample was filtered/vialed and the current location.

9.1.2 The template for this spreadsheet should include: the project code, sample ID, date received, number of bottles processed with this sample ID, a section to fill-in the date for each freeze and thaw (repeated three times), date the sample was filtered/vialed, storage location, initials of student and a notes section.

9.2 Liquid Chromatography Tandem Mass Spectrometry and Enzyme-Linked Immunosorbent Assay Run Sheets

9.2.1 The Project Management office and the Principle Investigator should be notified as samples are ready for analyses so run sheets may be populated and samples analyzed as appropriate.

10.0 QUALITY CONTROL

10.1 SAMPLES CHECKLIST

10.1.1 The samples checklist is created in the morning and about an hour before the personnel will leave for the day. After completing the checklist, personnel will update the Processing Spreadsheets with the new information.

10.1.1 To create the checklist, search through each Processing Spreadsheet to find which samples have not yet been filtered/vialed. Copy and paste the entire row of the sample's processing information onto the checklist and keep adding samples.

10.1.2 **Samples not completely thawed.** When a sample is still somewhat or totally frozen, mark the sample ID on the Samples Checklist with a colored highlighting marker. *Place the sample back in the sink to thaw or in the refrigerator for overnight storage as is appropriate.*

10.1.3 **Samples that are thawed and ready to freeze.** When a sample is completely thawed and ready to enter the next freeze cycle, mark the sample ID on the Samples Checklist with a colored highlighting marker (a different color than the marker used in section 8.2.3.3.1.1). *Mark a new line on the top of the sample bottle and place it into the freezer.*

10.1.4 **Samples that are thawed and ready for filtration.** When a sample is completely thawed and ready for filtration and vialling, mark the sample ID on the Samples Checklist with a colored highlighting marker (a different color than the markers used in sections 8.2.3.3.1.1 and 8.2.3.3.2.1). *Set the bottle aside under a cover and filter/vial.*

10.2 CHECKLIST FOR FILTRATION

10.2.1 The Checklist for Filtration helps the personnel accurately complete all filtration steps. While filtering a sample, check off each step in the process.

10.2.2 The checklist should include: the project code, sample ID, each step of filtration/vialing and the initials of the student.

10.3 **NOTES**

10.3.1.1 After cleanup at the end of the work shift it is necessary for personnel to communicate their progress on the project to the Project Management Office and Principle Investigator.

11.0 ATTACHMENTS

No attachments

12.0 REVISIONS TO THIS SOP

No revisions

1/18/2016 reviewed.

Appendix C.3

Title: *Data and Information Backup for all OGRL Instruments*	Identifier: *OGRL-2010*	Revision: **5**	Effective Date: **1/12/16**

15.1.1.1 APPROVALS FOR USE

Author's Name (Print): *Keith A. Loftin*	Author's Signature:	Date: *1/12/16*
Project Director's Name (Print) *Michael T. Meyer*	Project Director's Signature	Date: **1/12/16**

15.1.2 Organic Geochemistry Research Group (OGRG)

STANDARD OPERATING PROCEDURE

DATA AND INFORMATION BACKUP FOR HP GCMS, LCMS, AND HPLC INSTRUMENTS

Table of Contents

Data and Information Backup for all OGRL instruments

NOTE: Laboratory personnel may produce paper copies of this procedure printed from the controlled document file. However, it is their responsibility to ensure that they are trained on and utilizing the current version of this procedure. The procedure author may be contacted if text is unclear.

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the process Data and Information Backup for instruments for the USGS Organic Geochemistry Research Laboratory (OGRL) in Lawrence, KS.

2.0 TRAINING

The Project Director is responsible for ensuring that all who perform the function(s) described in this SOP for the OGRL are familiar with the objectives of and properly trained in its procedures. In addition, one must document that they have read and understand this procedure in their training folder.

3.0 DEFINITIONS

3.1 Computer—PC that is used to operate and control OGRL instruments.

3.2 External Backup Drive—external data storage drive used for transferring information from instrument computer's hard drive to an external flash drive.

4.0 PERSONNEL HEALTH AND SAFETY

Note: This SOP is to be used in conjunction with an approved Chemical Hygiene Plan. Also, consult the Chemical Hygiene Plan for information on and use of all PPE.

4.1 Obey and follow all Safety Regulations when entering the Laboratory.

5.0 EQUIPMENT AND SUPPLIES

Descriptions of commonly used pieces of equipment, there advantages and their limitations are listed below.

5.1 External Backup Drive — Each instrument is currently equipped with this device.

6.0 PROCEDURE

Note: Deviations from SOPs must be recorded in an appropriate instrument or work log.

6.1 Each instrument is equipped with an external backup drive to archive instrument methods, worklists, and data folders (hereafter referred to as data).

6.2 Data is manually archived weekly during scheduled instrument downtime.

6.3 Data archive is then backed up onto the USGS KS WSC network drive and also maintained on the external backup drive. The USGS KS WSC network drive has a redundant mirror site in case of network failure.

6.4 Over time given the operation of the instruments large quantities of data stored in files on the instrument computer hard drive will have to be permanently removed from the instrument computer (e.g. when 75% of computer's memory is consumed). Each instrument is backed up using the same general procedure. If possible perform backups when the instrument computer is not in operation.

6.5 Printed copies of instrument sequences and analytical methods are also maintained at each instrument.

7.0 REFERENCES

No references are cited in this SOP.

8.0 RECORDS AND ARCHIVAL

The person performing this SOP is responsible for submitting the following external drives to be archived to the Project Documents Archival manager.

9.0 QUALITY CONTROL

No quality control measures have been defined for this procedure.

10.0 ATTACHMENTS

There are no attachments to this SOP.

11.0 REVISIONS TO THIS SOP

6/6/00- Initial Version
2/15/02- Revisions 2 , Added Section 11.0
6/4/03-Reviewed, no changes
1/12/04 – Reviewed, no changes
1/12/16 – Reviewed, changed archive procedures from tape drive back up to external flash drive storage.

APPENDIX D: EXAMPLE SOPS FOR MERCURY IN FISH TISSUE PLUG ANALYSES

Eurofins Document Reference	EFGS-SOP-137-R02	**Revision**	2
Effective Date	6/17/2013	**Status**	Final
Historical/Local Document Number	FGS-SOP-137.02		
Local Document Level	Level 3		
Local Document Type	SOP		
Local Document Category	NA		

Prepared by	Ryan Nelson
Reviewed and Approved by	Dave Wunderlich and Patrick Garcia-Strickland

Table of Contents

Approvals:

Prepared by: _____ Date: 6/17/13

Approved by: _____ Date: 6/17/2013

Approved by: _____ Date: 6/18/13

[redacted]

1 Revision Log:

Revision: 06	Effective Date: This version	
Section	**Justification**	**Changes**
Cover	Required change	Changed company name from Frontier Global Sciences to Eurofins Frontier Global Sciences.
All	Formatting requirement per LOM SOP-LAB-201	Reformatted document to new corporate specifications.
13.1, 13.2	Required	Added hardware and software components
14.8	Required	Updated mercury standard prep
14.9	Required	Updated standard and reagent documentation procedures
15.2 – 15.4	Required	Updated calibration information
16.7	Required	Added instrument maintenance and troubleshooting

2 Reference:

2.1 EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, 2002.

2.2 Method 1669, "Method for Sampling Ambient Water for Determination of Metals at EPA Ambient Criteria Levels," U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Engineering and Analysis Division (4303), 401 M Street SW, Washington, DC 20460, April 1995 with January 1996 revisions.

2.3 Bloom, N.S.; and Tsalkitzis, E. Standard Operating Procedure FGS-012 Determination of Total Mercury in Aqueous Media (Modified EPA Method1631). Frontier GeoSciences Inc., Quality Assurance Manual 1995.

2.4 Bloom, N.S.; Ultra-Clean Sample Handling, Environmental Lab 1995, March/April, 20.

2.5 Bloom, N.S.; Horvat M., and Watras C.J. Results of the International Mercury Speciation Intercomparison Exercise. Wat. Air Soil Pollut. 1995, 80, 1257.

2.6 Bloom, N.S.; Crecelius, E.A. Determination of Mercury in Seawater at Sub-nanogram per Liter Levels.Mar.Chem.1983, 14, 49.

2.7 Bloom, N.S.; Crecelius, E.A. Distribution of Silver, Lead, Mercury, Copper, and Cadmium in Central Puget Sound Sediments Mar. Chem 1987, 21, 377-390.

2.8 Bloom, N.S.; Fitzgerald, W.F. Determination of Volatile Mercury Species at the Picogram Level by Low-Temperature Gas Chromatography with Cold-Vapor Atomic Fluorescence Detection. Anal. Chem. Acta. 1988, 208, 151.

2.9 Cossa, D.; Couran, P. An International Intercomparison Exercise for Total Mercury in Seawater. App.Organomet. Chem.1990, 4, 49.

2.10 Fitzgerald, W.F.; Gill, G.A. Sub-Nanogram Determination of Mercury by Two-Stage Gold Amalgamation and Gas Phase Detection Applied to Atmospheric Analysis. Anal. Chem. 1979, 15, 1714.

2.11 Gill, G.A.; Fitzgerald, W.F. Mercury Sampling of Open Ocean Waters at the Picogram Level Deep Sea Res.1985, 32, 287.

2.12 EPA Method 30.B, Determination of total vapor phase mercury emissions from coal-fired combustion sources using carbon sorbent traps.

2.13 Chemical Hygiene Plan, Eurofins Frontier Global Sciences, current version.

2.14 National Environmental Laboratory Accreditation Conference, NELAC Standard September 8, 2009.

2.15 Department of Defense Quality Systems Manual for Environmental Laboratories, prepared by DoD Environmental Quality Workgroup, Final Version 4.2, October 2010.

3 Cross Reference:

Document	Document Title
SOP FGS-003	Pipette Verification, Calibration and Maintenance
SOP FGS-007	Cleaning of Sampling Equipment and Bottles
SOP FGS-008	Ultra Clean Aqueous Sample Collection
SOP FGS-012	Oxidation of Aqueous Samples for Total Mercury Analysis
SOP FGS-061	Gold Trap Construction
SOP FGS-094, App F	Standard Operating Procedure Training Record
SOP FGS-099	Waste Disposal Procedure for Client Sample Waste
SOP FGS-121	Determination of Total Mercury by Flow Injection AFS (Mod 1631E)
SOP FGS-155	Calibration of Volumetric Dispensers

4 Purpose:

4.1 This SOP is designed to ensure that all reproducible traceable procedures in EPA 1631 are followed in the standardization of the total mercury analyzers and in the analysis of samples for total mercury, as well as to establish the limits wherein data will be considered acceptable.

5 Scope:

5.1 This Standard Operating Procedure (SOP) describes a method for the determination of total mercury (Hg) in filtered and unfiltered water by oxidation, purge and trap, desorption, and cold vapor atomic fluorescence spectrometry (CVAFS).

5.2 This method is designed for the determination of mercury in the range of 0.5-40 ng/L (ppt). Application may be extended to higher levels by selection of a smaller sample size, as long as the instrument value (intensity) remains within the calibration curve.

5.3 The Control Limits are established from EPA 1631E.

6 Basic Principles:

6.1 For analysis of aqueous samples, an aliquot of oxidized sample is neutralized with hydroxylamine-hydrochloride (NH_2OH-HCl) to destroy free halogens, and added to a bubbler.

6.2 Stannous chloride ($SnCl_2$) is added to the bubbler to reduce the Hg(II) to volatile Hg(0), and the bubblers are sealed with Keck clips. Blanked gold traps are placed at the end of soda-lime pre-traps. The bubbler is purged with nitrogen (N_2) for 20 minutes. All gas that flows into the bubbler should only leave the system through the soda-lime pre-trap and then the gold trap.

6.3 The gaseous mercury amalgamates to the gold traps, which are removed and individually placed in the analytical train. The gold trap is heated, thus releasing the mercury into the argon gas stream flowing into the instrument.

7 Reference Modifications:

7.1 There were no significant modifications to this method.

8 Definitions:

8.1 Analytical Duplicate (AD): A representative sample (that yielded a result within the calibration curve) is analyzed a second time during the analytical run. The second analysis should be at the same aliquot as the original.

8.2 Analytical Run – The continuous analysis of one or more batches during the same 12 hour-shift. Each analytical day requires a minimum five-point calibration curve, ICV, at least 3 IBLs, and CCV/CCB every ten runs. An analytical day must conclude with a CCV/CCB.

8.3 Analytical Spike and Analytical Spike Duplicate (AS/ASD): A representative sample is selected and spiked, with a dilution of the primary source, during the analytical run, at a target concentration of 1-5X the ambient concentration of the sample. These QC samples are used to indicate sample matrix effects on the analyte of interest. Non-detectable samples are spiked at 1 – 5 x of the MRL/PQL.

8.4 Batch: 20 client samples or less grouped for preparation. See Quality Assurance Section for batch requirements.

8.5 Calibration Standards (CAL) – a series of standards that will be used to calibrate the instrument, made from a primary source stock standard. A calibration blank plus at least five different concentrations are required, beginning with one at PQL concentration.

8.6 Certified Reference Material (CRM) – a standard of known composition that is certified by a recognized authority and representing a sample matrix. It is used to verify the accuracy of a method.

8.7 Continuing Calibration Blank (CCB): An instrument blank that is used to monitor the ambient blank concentration after the Continuing Calibration Verification (CCV).

8.8 Continuing Calibration Verification (CCV): An aliquot of standard from the same source as the calibration standard, at a value of 20ng/L (2.0ng in ~100mL bubbler water). This standard is analyzed after every 10 analytical runs, and determines whether the instrument is maintaining calibration.

8.9 Continuing Demonstration of Capability (CDOC)

8.10 Control Limit (CL) – the limit of the range of acceptability for the quality control samples

8.11 Equipment Blank (EB): Reagent water processed through the sampling devices and placed in a sample container prior to using the equipment to collect samples and used to demonstrate that the sampling equipment is free from contamination.

8.12 Field Blanks (FB): A sample of reagent water placed in a sample container in the field and used to demonstrate that samples have not been contaminated by sample collection or transport activities. EPA-1631E recommends the analysis of at least one field blank per 10 samples collected at the same site at the same time. Analyze the blank immediately before analyzing the samples in the batch.

[redacted]

8.13 Initial Calibration Verification (ICV): A standard that is prepared from a secondary source stock standard with a value of 15ng/L (1.5ng in ~100mL bubbler). This standard is run immediately following the calibration curve and verifies instrument calibration. It is always followed by the IBLs.

8.14 Initial Blank Level (IBL): An instrument blank that is used to demonstrate the ambient blank concentration of the instrument. One per bubbler is needed at the beginning of the analytical run.

8.15 Initial Demonstration of Capability (IDOC).

8.16 Laboratory Control Sample (LCS and LCSD) or Quality Control Sample (QCS): A sample (and duplicate) containing a known concentration of mercury that is used to monitor complete method performance. The preferred LCS is a matrix matched Certified Reference Material (CRM), but a blank spike meets the requirement also. In LIMS, the LCS is always referred to as a Blank Spike (BS), whether it is matrix matched or not.

8.17 Limit of Detection (LOD) – equal to MDL and verified on a quarterly/annual basis, depending on the preparation, by spiking within three times the established LOD and showing a positive result on the instrument.

8.18 Limit of Quantitation (LOQ) – equal to PQL and verified on a quarterly/annual basis, depending on the preparation, by spiking within 2 times the LOQ and showing a recovery between 70 – 130%.

8.19 LIMS: Laboratory Information Management System. Computer software used for managing samples, standards, and other laboratory functions.

8.20 May: This action, activity, or procedural step is optional.

8.21 May Not: This action, activity, or procedural step is prohibited .

8.22 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): A representative sample is selected and spiked with a dilution of the primary source at a known concentration. The MS and MSD are run through the entire analytical process just as the samples are. These QC samples will indicate sample matrix effects on the analyte of interest.

8.23 Method Blank (MBLK) or Preparation Blank (PB): For waters, reagent water that is prepared and analyzed in a manner identical to that of samples. For digested solids, preparations blanks consist of the same reagents used to digest the samples, in the same volume or proportion and are carried through the complete sample preparation and analytical procedure. Boiling chips are used as a blank matrix for solids. Preparation blanks are referred to as BLK in LIMS.

8.24 Method Detection Limit (MDL): A limit derived from 40 CFR, Part 136, Appendix B. This method produces a defined value that is the minimum concentration that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero from a given matrix.

8.25 Method Duplicates/Method Triplicates (MD/MT): A second or third separate sample dilution, taken from the same source sample, prepared and analyzed in the laboratory separately. An MSD may be used as a duplicate.

8.26 Reagent water: 18 MΩ minimum, reagent water starting from a pre-purified (distilled, Reverse Osmosis, etc.) source.

8.27 Must: This action, activity, or procedural step is required.

8.28 Ongoing Precision and Recovery (OPR): A dilution of a secondary source resulting in an instrumental concentration of 5.0 ng/L mercury.

8.29 PM: Project Manager.

8.30 Practical Quantitation Limit (PQL), Method Reporting Limit (MRL): The minimum concentration that can be reported quantitatively. The PQL is often described as 1-10 times higher than MDL. Eurofins Frontier defines the PQL as the lowest concentration that can achieve 70-130% recovery for 10 replicate sample preparations. In LIMS, the PQL is referred to as the MRL.

8.31 Primary Source: The stock standard used to make the calibration standard. Procedural Method: A method where standards and samples are run through the analytical procedure exactly the same. By NELAC definition, this SOP is a procedural method.

8.32 Secondary Source: The stock standard used to make the OPR standard.

8.33 Shall: This action, activity, or procedure is required.

8.34 Should: This action, activity, or procedure is suggested, but not required.

8.35 Stock Standard Solution (SSS) – a standard of analyte that is purchased from a certified source for the preparation of working standards.

8.36 Total mercury: As defined by this method, all bromine monochloride-oxidizable mercury forms and species found in aqueous solutions. This includes, but is not limited to, Hg(II), Hg(0), strongly organo-complexed Hg(II) compounds, adsorbed particulate Hg(P), and several tested covalently bound organomercurials (i.e. CH_3HgCl, $(CH_3)_2Hg$, and $C_6H_5HgOOCCH_3$). The recovery of mercury bound within microbial cells may require additional preparation steps (i.e. UV oxidation, or oven digestion).

8.37 Travel or Trip Blank (TB): A sample of reagent water placed in a sample container in the laboratory and used to demonstrate that samples have not been contaminated by transport activities.

9 Interferences:

9.1 Gold and iodide are known interferences. At a mercury concentration of 2.5 ng/L and at increasing iodide concentrations from 30 to 100 mg/L, test data have shown that mercury recovery will be reduced from 100 to 0 percent. At iodide concentrations greater than 3 mg/L, the sample should be pre-reduced with $SnCl_2$ (to remove brown color immediately prior to analysis) and additional or more concentrated $SnCl_2$ should be added to the bubbler containing sample. If samples containing iodide concentrations greater than 30 mg/L are analyzed, it may be necessary to clean the analytical system with 4N HCl after the analysis.

9.2 Water vapor has the potential to create recovery interferences. To prevent interference from water, ensure that soda-lime pre-traps and gold traps remain dry.

9.3 The presence of high concentrations of silver and/or gold can cause $SnCl_2$ to precipitate out of solution and adhere to the bubbler walls. High concentrations of these metals can sometimes be found in the matrix spike samples from the digestion sets that are shared with the trace metals group. When analyzing digestates where the matrix spike samples have been spiked with silver or gold, the matrix-spiked samples must not be used for mercury analysis. Instead, an alternate matrix spike and matrix spike duplicate (MS/MSD) should be prepared and analyzed. If this is not possible, an Analytical Spike/Analytical Spike Duplicate (AS/ASD) must be analyzed on the ambient sample.

10 Safety Precautions, Pollution Prevention and Waste Handling:

10.1 Personnel will don appropriate laboratory attire according to the Chemical Hygiene Plan. This includes, but is not limited to, laboratory coat, safety goggles and nitrile gloves under clean gloves.

10.2 The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Chemists should refer to the MSDS (Material Safety Data Sheets) for each chemical they are working with.

10.2.1 Note: Use particular caution when preparing and using BrCl, as it releases extremely irritating, corrosive fumes similar in effect to free chlorine. Always handle this reagent in an approved fume hood

10.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease-causative agents. Eurofins Frontier will reimburse the expense of Hepatitis A and B immunizations for any laboratory staff member who desires this protection.

10.4 Hydrochloric acid: Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion. Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering. For more information see MSDS.

10.5 See Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP) for general information regarding employee safety, waste management, and pollution prevention.

10.6 Pollution prevention information can be found in the current Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP), which details and tracks various waste streams and disposal procedures.

10.7 All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations. Any waste generated by this procedure should be disposed of according to SOP FGS-099 "Waste Disposal Procedure for

Client Sample Waste," which provides instruction on dealing with laboratory and client waste.

11 Personnel Training and Qualifications:

11.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on CRMs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

11.2 The analyst/laboratory technician must have read this SOP and other relevant SOPs and have the training documented on the applicable form(s). The analysis may be questioned on SOP by supervisor(s) and/or trainers.

11.3 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

11.4 Reading of the SOP must be documented on the correct form such as "Standard Operating Procedure Training Record," Appendix F in FGS-094, the last page of this SOP, Appendix A "Standard Operating Procedure Training Record" or a similar document."

11.5 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

11.6 All training documents including IDOCs, CDOCs, SOP reading, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11.7 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

12 Sample Collection, Preservation, and Handling:

12.1 Aqueous samples are collected in rigorously cleaned fluoropolymer (e.g. Teflon) or PETG bottles and caps (as described in FGS-007 "Cleaning of Sampling Equipment and Bottles for Mercury Analysis"). Certified clean glass bottles with fluoropolymer lids may be used if mercury is the only analyte of interest.

12.1.1 Aqueous samples are preserved upon receipt with 0.2N BrCl that has tested low in mercury. Samples are typically preserved to 1% BrCl v/v, but may require further oxidation due to high levels of organic matter or mercury. Refer to FGS-012 "Oxidation of Aqueous Samples for Total Mercury Analysis" for oxidation of aqueous samples. Samples requiring greater than 10% BrCl must have a method blank prepared at the time of preservation. Preservation levels should be limited to 1%, 2%, 3%, 5%, 10%, and 100%.

12.1.2 Preservation levels other than 1% are written on the LIMS label of the sample bottle. Preservation levels are also documented in the LIMS bench sheet by adjusting the initial and final volumes. For example, a sample preserved at 2 % BrCl must say "2" on the LIMS label, and have an initial volume of 100mL and a final volume of 102mL in the bench sheet.

12.2 All samples should be collected utilizing clean techniques, so as not to cross-contaminate samples with mercury. See FGS-008 "Ultra Clean Aqueous Sample Collection" and EPA Method 1669 for aqueous sample techniques.

13 Apparatus and Equipment:

13.1 LIMS – Element, version 5.85 or higher; Computer – Windows XP, 7 or 8

13.2 *Tekran 2500 Atomic Fluorescence Spectrophotometer (AFS) or equilivalent: A high sensitivity AFS Detector (IDL<1pg) with a required wavelength of 253.7 nm and associated software.*

13.3 Flow meter/needle valve: A unit capable of controlling and measuring gas flow to the cold vapor generator at 200-500 mL/min.

13.4 Teflon Fittings: Connections between components and columns are made using Teflon FEP tubing and Teflon friction fit tubing connectors.

13.5 Soda-Lime pre-trap: A 10cm x 0.9cm diameter Teflon tube containing 2-3 g of reagent grade, non-indicating 8-14 mesh soda-lime ($Ca(OH)_2$+NaOH) aggregates, packed between portions of silanized glass wool. This trap is purged of mercury by placing it on the output of a clean cold vapor generator and purging it with ~3-5% HCl and ~600 µL of $SnCl_2$ for approximately 20 minutes with N_2 at 40 mL/min.

13.6 Cold-vapor generator (bubbler): A 150 mL, tall, flat-bottom borosilicate flask with standard taper 24/40 neck, fitted with a sparger having a coarse glass frit which extends to within 0.2 cm of the flask bottom.

13.7 Gold Traps: Made from 12 cm lengths of 6 mm OD quartz tubing, with a 4-way crimp 3.0 cm from one end. The tube is filled with approximately 2.5 cm of 20/40 mesh gold-coated quartz sand, the end of which is then plugged with quartz wool. Gold-coated sand traps are heated to 450-500°C (the coil should have a barely visible red glow when the room is darkened) with a coil consisting of 75 cm of 24-gauge nichrome wire at a potential of 10 VAC. Potential is applied and finely adjusted with an auto-transformer. Refer to SOP FGS-061 regarding the construction of gold traps used for total mercury analysis.

13.8 Agilent Integrator Recorder or equivalent: Any multi-range chart recorder or integrator with 0.1-5.0 mV input and variable speeds is acceptable. Data capture software may also be used.

13.9 Pipettes: Calibrated variable pipettes with a range of 5 µL – 10 mL. Used to make solutions and sample dilutions. Pipettes are to be calibrated weekly according to SOP FGS-003 and FGS-155.

14 Reagents and Standards:

All reagents, except those made daily, must be entered into LIMS

14.1 Reagent Water: 18-MΩ ultra pure deionized water starting from a pre-purified (distilled, R.O., etc.) source is used. To remove any remaining trace metals and organics, an activated carbon cartridge is placed between the final ion exchange bed and the 0.2-µm filter. Reagent water used in the mercury lab is checked weekly for total mercury concentrations, and must test below 0.25ng/L.

14.2 Hydrochloric Acid (HCl): Concentrated (36-38% weight basis). Must be trace-metal purified and reagent grade. HCl is typically monitored through performance of the BrCl. Sometimes it will be necessary to test the HCl directly. To do so, add 1 mL, using a calibrated pipette, of HCl to approximately 100mL of purged bubbler water. Enter 1mL as aliquot in the Excel spreadsheet. Do not prep blank correct. Analyze one replicate per bottle. This reagent should test below 5.0 ng/L. This solution is considered stable until the expiration date on the bottle, set by the manufacturer.

14.3 0.2N Bromine Monochloride (BrCl):

14.3.1 37.5 g of KBr is added to a 2.5-L bottle of concentrated HCl (pre-analyzed and found to be below 0.25 ng/L Hg). The bottle is then inverted in a fume hood to mix the acid and KBr. The solution then sits overnight allowing for the KBr to be dissolved.

14.3.2 27.5 g of $KBrO_3$, certified to be low in Hg, is slowly added to the acid. When all of the $KBrO_3$ has been added, the solution should have gone from yellow to red to orange.

14.3.3 Loosely cap the bottle, and allow to sit for 30 minutes in a fume hood before tightening the lid. Once capped invert bottle to make sure all of the solids goes into solution. **CAUTION: This process generates copious quantities of free halogens (Cl_2, Br_2, BrCl) which are released from the bottle. Add the $KBrO_3$ SLOWLY and in a well operating fume hood.**

14.3.3.1 To test the BrCl, add 1 mL, using a calibrated pipette, of the BrCl to a prep blank vial containing approximately 4 mL reagent water. Add 200 µL Hydroxylamine-HCl to the vial; pour the entire contents into a bubbler containing approximately 100 mL of purged water. Assume a 100 mL aliquot in the Excel spreadsheet. This reagent must test below 0.20ng/L. Do not prep blank correct. Analyze one replicate per bottle.

14.3.3.2 The expiration time for this reagent is set by default to six months in LIMS. There is no suggested holding time in EPA method 1631E, therefore the holding time can be extended, as long as the primary reagent has not expired. The mercury concentration of the BrCl is monitored through the preparation of water preparation blanks.

14.4 Hydroxylamine hydrochloride: dissolve 300g of NH_2OH-HCl in reagent water and bring the volume up to 1L. This solution may be purified by the addition of 1mL $SnCl_2$ solution and purging overnight at 500mL/min with mercury-free N_2. The working reagent is a 25% solution that is made by adding one part reagent water to one part 50% hydroxylamine hydrochloride. This reagent must test below 0.25ng/L.

14.4.1 To test the Hydroxylamine-HCl (NH_2OH-HCl), add 1 mL of the 50% reagent, using a calibrated pipette, to approximately 100 mL of purged bubbler water.

Assume a 100 mL aliquot in the Excel spreadsheet. This reagent must test below 0.20 ng/L. Do not prep blank correct. Analyze one replicate per bottle.

14.4.2 The expiration time for this reagent is set by default to six months in LIMS. There is no suggested holding time in EPA method 1631E; therefore the holding time can be extended, as long as the primary reagent has not expired.

14.5 Stannous Chloride ($SnCl_2$): Weigh out 500 g $SnCl_2$ using a calibrated balance that also has been verified for the day. Dissolve with three 100 mL aliquots of concentrated HCl and transfer to a 1L I-CHEM glass bottle, which contains approximately 300 mL of reagent water. Bring this solution up to approximately 1 L of volume and purge overnight with mercury-free N_2 at 500 mL/min to remove all traces of mercury. Store tightly capped. The working reagent is a 25 % solution that is made by adding one part reagent water to one part 50 % stannous chloride.

14.5.1 To test the Stannous Chloride ($SnCl_2$), add 1 mL of the 50% reagent, using a calibrated pipette, to approximately 100 mL of purged bubbler water. Assume a 100 mL aliquot in the spreadsheet. This reagent must test below 0.20 ng/L. Do not prep blank correct. Analyze one replicate per bottle.

14.5.2 The expiration time for this reagent by default is set to six months in LIMS. There is no suggested holding time in EPA method 1631E; therefore the holding time can be extended, as long as the primary reagent has not expired.

14.6 Argon Grade 4.7 or better (ultra high-purity grade): Argon that has been further purified by the removal of mercury using a gold trap that is located in line between the gas output and the analyzer gas input.

14.7 Nitrogen Grade 4.5 (standard laboratory grade): Nitrogen that can be further purified of mercury using a gold trap that is located in line between the gas output and bubbler

14.8 Preparation of Total Mercury Standard Solutions:

14.8.1 Mercury standard solutions are prepared in ultra clean volumetric glassware and gravimetrically calibrated pipettes. Resulting solutions must be stored in glass or Teflon bottles and preserved to at least 2 % BrCl. All working standards must be tested prior to use.

14.8.1.1 New working standards and standard dilutions are tested prior to use. Three reps of the new standard are analyzed in the same run as three reps of the current NIST 1641D standard. Analyze 200 µl of the NIST 1641D and assume 100 ml in the bubbler. The mean percent recovery of the three standards should be ±5 % (95-105 %) of the true value and also within 5 % of the average NIST 1641D recovery (e.g. If the average of NIST 1641D recovery is 97 %, the range for the standard is 95-102 %). If the standard does not test within this control limit, it is retested. If it still does not meet the control limit, it is discarded and remade, unless otherwise approved by the Quality Assurance Officer. NOTE: When making serial dilutions to create various standard levels; the lowest concentration may be used to test any of the higher concentration steps (for example: if a 10ng/mL calibration standard is created from a 1000ng/mL spiking standard, only the 10ng/mL standard requires testing.

If the 10ng/mL standard passes, then both standards are considered to be passing within the control limits.)

14.8.2 Total Mercury Stock Standard Solution (Stock): Certified mercury standard purchased from High Purity Standards (1000 µg/mL (1 000 000 ng/mL) primary source) or Absolute Standards (100 µg/mL (100 000 ng/mL) secondary source), or any equivalent standard.

14.8.3 Total Mercury Spiking Standard Solutions (Spiking Standard): Spiking standards are made from either the primary or secondary sources.

14.8.3.1 To make standards, use an ultra clean volumetric flask and a calibrated pipette. Add reagent water until flask is about half full. Add 2 % 0.2N BrCl and the specific spike volume noted below (these volumes may be changed as long as ratio and resulting concentration remains the same). Bring up to the mark with reagent water and mix well prior to testing. When spiking samples, no more than 200 µL of any spiking standard is added to the sample to minimize effects on volume. It is also recommended that staff pipette no less than 25 µL. If possible, minimize headspace during standard storage. Expiration date is currently set at 6 months or when the stock standard expires, whichever is shorter.

14.8.3.2 100,000 ng/mL Spiking Standard: Made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 10 mL of the stock standard to 100 mL of reagent water containing 2 % BrCl. (Can also be made by preserving Secondary Stock Standard to 2% BrCl).

14.8.3.3 10,000 ng/mL Spiking Standard: If made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 1.0 mL of the stock standard to 100 mL of reagent water containing 2 % BrCl. If made from Secondary Stock Standard, dilute 10mL of stock standard to 100mL with reagent water containing 2% BrCl.

14.8.3.4 1,000 ng/mL Spiking Standard: If made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 0.250 mL of the stock standard to 250 mL RO water containing 2 % BrCl. If made from Secondary Stock Standard dilute 2.5mL of stock standard to 250mL with RO water containing 2% BrCl.

14.8.3.5 100 ng/mL Spiking Standard: Made from a stock standard or dilution of a stock standard with a concentration of 100,000 ng/mL. Dilute 0.100 mL of the 100,000 ng/mL dilution to 100 mL of reagent water containing 2 % BrCl. Expiration date is currently set at 3 months or when the stock standard expires, whichever is shorter.

14.8.4 Calibration Standard (10 ng/mL): Must be made from a dilution of the Primary Stock Standard (High Purity, or equivalent vendor). Typically made by diluting 0.5mL of a 10,000 ng/mL Primary Spiking Standard to 500 mL of reagent water containing 2 % BrCl. Transfer to glass or Teflon bottle. The calibration standard is considered stable for three months or until the stock standard expires.

14.8.5 Calibration Standard (1 ng/mL): Must be made from a dilution of a Primary Stock Standard. Typically made by diluting 1.0mL of a 100 ng/mL Primary Spiking Standard to 100mL with Reagent water containing 2% BrCl.

14.8.6 Initial Calibration Verification (ICV): A 10 ng/mL ICV solution is prepared using the Secondary Stock Standard (Absolute Standards, or equivalent vendor). Use 0.100 mL (100 µL) of the Secondary Stock Standard to 1000 mL Milli-Q containing 2 % BrCl. Transfer to one 1000 mL glass or Teflon bottle. The ICV standard is considered stable for three months or until the stock standard expires. It is recommended to alternate expiration date with the CAL standard.

14.8.7 Continuing Calibration Verification (CCV): For CCV analysis, use 200 µL of the 10 ng/mL CAL standard (documented in LIMS as SEQ-CAL3). The True Value is 20 ng/L.

14.8.8 Certified Reference Material (CRM) for Total Mercury in Water: A 1.5679 mg/L solution (1.557 mg/kg at a density of 1.007 g/mL) is prepared by adding a 5.0 mL of CRM NIST 1641d (from ampoule) into a 1000 mL flask containing of reagent water. This solution is diluted to 1000 mL, and an additional 10 mL of 0.2N BrCl is added, resulting in a final volume of 1010 mL. Preparing the solution in this manner makes a 1:200 dilution of the stock CRM. This solution is considered stable for one year, or until the stock standard expires. Results are corrected for the additional 1 % BrCl in the analysis Excel spreadsheet and in LIMS.

14.8.9 Ongoing Precision and Recovery (OPR) for "Strict" 1631E: A 5.0 ng/L solution is prepared by adding 100 µL of the 100 ng/mL secondary spiking standard into 2000 mL reagent water. An additional 1 % BrCl (20 mL) of BrCl is added, so that the final volume is 2020 mL. This standard is analyzed at 100 mL at the instrument, and preparation blank corrected exactly in the same manner as samples

14.9 Documentation of Standards and Reagents:

14.9.1 Standards and Reagents are documented in LIMS upon receipt or creation. A LIMS generated label is affixed to each standard and reagent that has the name of the solution, the person who prepared or received it, the date it was prepared or received, and the expiration date.

14.9.2 Each bottle of standard must be labeled with the following: the date of receipt or creation, the initials (or name) of who entered the standard into LIMS, the concentration and analyte, the expiration date and the LIMS ID. This information must also appear on the certificate of analysis of stock standards.

14.9.3 Stock standards and CRMs are logged into LIMS upon receipt by Shipping and Receiving (S&R) or the Quality Assurance department (QA). These do not require testing, provided there is a Certificate of Analysis on file in QA. When receiving a solid CRM, QA shall generate a work order in LIMS for total solids analysis.

14.9.4 For all standards, LIMS documentation must include the following: a description of the standard, department, expiration date of the standard (not to exceed the expiration of the parent standard), the name of the person who made (or

received) the standard or reagent, the date it was prepared (or received), final volume, a reference date (date entered into LIMS), concentration units (µg/mL), the vendor and vendor lot. The solvent lot is used to document the Lot Number or LIMS ID of the BrCl that was used. In the comments section, the analyst must enter the sequence and applicable results for documentation of standard testing. Other notes may be entered in here as well. The correct parent standard must be noted, as well as the amount used. Analytes are entered individually from the list. LIMS will calculate the true value of the standard based on the amount of the parent used and the final volume. Click the appropriate radio button under Standard type. A Spike Mix is a standard that is used in a bench sheet, and a Calibration standard is a standard used only in sequences. A Reference Standard is a Certified Reference Material (CRM). The standard must not be used until it has passed control limits and is approved by the mercury supervisor, mercury laboratory manager, or QA for use.

14.9.4.1 If the new standard is a calibration standard, a separate standard ID must be created for each calibration point based on the final concentration in the sequence (example: THg CAL1 0.10 ng or THg CAL2 0.50 ng). These are given the same expiration as the standard they are made from, and will need to be generated every three months as each new working calibration standard is made and tested.

14.9.4.2 To generate new "CAL" standards in LIMS, go to the Laboratory drop down menu and select Standards. Open the current CAL1 standard and click "Copy". Update the appropriate information, including the Prepared Date, Expiration Date, Prepared By, and the Reference Date. For these standards, which are to be used in the sequence, the final volume is equal to the assumed aliquot in the bubbler (100 mL). Check that the vendor lot is correct. Remove the old (expired) parent standard. Choose the new parent standard, and enter the amount of standard added to the bubbler for that calibration point. All depleted or expired standards are moved into the Expired Standards Department once they are no longer being used.

14.9.4.3 Each bottle of standard must be labeled with the following: the date of receipt or creation, the initials (or name) of who entered the standard into LIMS, the concentration and analyte, the expiration date and the LIMS ID. This information must also appear on the certificate of analysis of stock standards.

14.9.5 Neat reagents are logged into LIMS with a unique identifier upon receipt by Shipping and Receiving Department and given a default expiration of 3 years, unless otherwise noted by the manufacturer.

14.9.6 Working reagents are prepared by the analyst, logged into LIMS and assigned a unique identifier. Reagents entered into LIMS must have the information listed in section 14.9.2. In addition the parent neat reagents are added by their unique identifier and the amount of each reagent is entered. It is not necessary to enter analytes from the list for reagents. The Solvent Lot is not applicable to working reagents. The radio button must be clicked to Reagent. If the reagent

[redacted]

requires testing, it must test clean prior to using. All reagents used during analysis and prep should be added to bench sheet.

14.9.7 Depleted or expired standards and reagents are segregated and removed from use.

15 Calibration:

15.1 The analyst should label the strip chart/integrator printout with the corresponding dataset ID as well as print and sign their name. For strip chart printouts, the analyst should label the baseline ratios accordingly (usually X=1 and X=20) and label with the analysis day start time and strip chart drum speed (usually 1 mm/min). The analyst should note the end time as well. If using an integrator, the date and time should be checked and corrected if necessary.

15.2 The calibration sequence determines the range of sample concentrations that are reportable. The calibration sequence starts with a 5-point curve using the total mercury calibration standard solution. The five points are: *0.05ng (0.50 ng/L),* 0.10 ng (1.00 ng/L), 0.50 ng (5.00 ng/L), 2.00 ng (20.00 ng/L), and 4.00 ng (40.0 ng/L). An ICV/OPR and IBLs (one for every bubbler used are analyzed immediately following the standard curve.

15.2.1 Using the 10 ng/mL calibration standard, add 5 µL, 10 µL, 50 µL, and 200 µL to the bubblers sequentially from the left to right. Add 300 µL $SnCl_2$ to the bubblers and seal bubbler tops using Keck Clips.

15.2.2 Place blanked gold traps securely at the end of soda-lime traps (pinched section of gold trap closest to the soda-lime trap). Purge bubblers with N_2 for a minimum of 20 minutes.

15.2.3 Attach individual gold traps to the analytical train and burn in sequential order. Peaks produced should be labelled, as well as recorded in the Excel spreadsheet in real time.

15.3 For the second round, add *400 µL* of the 10 ng/mL mercury calibration standard to the first bubbler. Add 50µL of the *10 ng/mL* ICV(OPR) standard to the second bubbler *(5.0 ng/L). The third and fourth bubblers are used for the first and second IBLs and nothing should be added to these bubblers.* To ensure that nothing is added, keep it sealed with a Keck Clip. Add 300 µL $SnCl_2$ to all bubblers except the fourth and seal bubbler tops with Keck Clips.

15.4 For the third round, use the first and second bubbler to finish the IBLs needed for 1631. The third and fourth bubbler can be used for the first portion of the batch. If the curve does not pass or needs to be investigated any batch portions analyzed in this round will need to be reanalyzed.

15.5 Once the instrument is calibrated and the ICV/IBLs are analyzed and judged to be in control, the instrument is operational. The sample concentrations must fall within the range of the calibration standards or be diluted and reanalyzed.

15.6 The purge efficiency of the bubbler system is 100 % and is independent of volume at the volumes used in this method. Calibration of this system is typically performed using units of mass. For purposes of working in concentration, the volume is assumed to be 100 mL.

15.7 This completes the instrument calibration for total mercury analysis.

16 Procedure:

16.1 *When analyzing on the Tekran 2600, follow the procedure in EFGS-121 while still adhering to the QA/QC criteria of this method.*

16.2 Pre-analysis and Organization:

16.2.1 Prior to analyzing samples it is imperative to reference LIMS for all project specific information, such as QC requirements, suggested dilutions, project manager information, and specifics regarding spike levels.

16.2.2 The analyst should then locate samples and check the work order in LIMS for notes about specific project requirements.

16.2.3 The analyst should compare the sample IDs to the work order and see that the samples are accounted for, and notify the project manager of any discrepancies in analysis required, sample identification, etc.

16.2.4 All mercury analyses receive a unique dataset identifier. This is comprised of the instrument type and number, the date and the calibration number for that day. The format is as follows: THg8-091218-1, where "THg "refers to a total mercury analysis; "8" refers to the analyzer number 8; 091218 refers to the date (December 18, 2009 in the YYMMDD format); and "1" refers to the first calibration of the day.

The sequence number is assigned by LIMS when the data gets imported into LIMS. The alpha-numeric code is based on the following format: 3B02001, where the 3 refers to the year (2013), the "B" is the month (A= January, B=February...L=December), "02" is the day of the month (February 2nd) and the final 3 digits is the nth sequence created on that particular year/month/day combination.

16.2.5 In general, the analyst should organize their samples in the order listed on the bench sheet. The first samples analyzed should be the preparation blanks, then the LCS if analyzing solid samples, followed by actual samples. If possible, run total and dissolved samples side by side to facilitate verification that total concentration is greater than dissolved concentration. See QA section.

16.2.6 All samples specified as being *High QA* should be analyzed prior to any Standard QA projects that are being analyzed on the same instrument on the same day. However, if concentrations are known, analyze samples with low concentrations prior to samples with high concentrations

16.3 Instrument Start Up:

16.3.1 Begin blanking gold traps. To do this, attach one trap at a time to the analytical train and burn to the instrument. Ensure the Argon is flowing at appropriate levels (~25-40 mL/min). The pinched portion of the gold trap should be on the left (closest to the analytical trap). Continue to burn traps in sequential order.

16.3.2 Rinse out the bubbler three times with reagent water and fill with about 100 mL of reagent water. Using a pre-purged pipette, add 3-5 mL HCl. Initially add 600 µL of $SnCl_2$.

16.3.3 Prepare one soda-lime trap for each bubbler. To prepare soda-lime traps, hold soda-lime between two glass wool plugs in a Teflon tube. Cap the tubes with Teflon plugs and attach to the bubbler. Once the soda-lime traps have been attached, the bubbler system (soda lime trap and bubbler water/acid/ $SnCl_2$) must purge for a minimum of 20 minutes before beginning the instrument calibration sequence.

16.4 Analyzing Aqueous Samples:

16.4.1 All aqueous samples should be preserved with BrCl according to FGS-012 at least 24 hours prior to analysis. In the event a sample requires further oxidation prior to analysis, additional BrCl is added and the sample should not be analyzed for at least 12 additional hours. In special cases where rush turn-around-time is required and an oxidation period of less than 24 hours may be used, a heated oven digestion procedure can be utilized.

16.4.2 While bubbling and burning the standard curve, the analyst should prepare a minimum of three BrCl method blanks (BLK) at 1% BrCl. Add 1 mL BrCl and 200 µL hydroxylamine hydrochloride (NH_2OH-HCl) to each bubbler. The aliquot is assumed to be 100 mL. Any sample requiring an increased amount of reagent must be accompanied by at least one method blank that includes an identical amount of reagent.

16.4.3 After the instrument calibration sequence, preparation blanks and the LCS/LCSD are analyzed.

16.4.4 All known field, equipment, and trip blanks should be analyzed before any other sample types, usually after the BLKs. Aliquots of 100 mL should be analyzed, provided there is adequate collected sample volume. Sample aliquot sizes of 125 mL can be analyzed upon request by the project manager.

16.4.5 For all waters, select the appropriate dilution (refer to LIMS, historical data, etc.).

16.4.5.1 For sample aliquots of 25 µL to 10.0 mL, use calibrated pipettes to dispense the aliquots directly into bubbler. Due to minimal amounts of BrCl in aliquots of 10 mL or less, NH_2OH-HCl is not added. It is highly recommended that the analyst should not pipette less than 25 µL. A dilution of the sample should be made to allow a larger aliquot to be analyzed.

16.4.5.2 For sample aliquots greater that 10 mL, gravimetrically weigh out the selected volume (±0.2 g) into a clean 125 mL Teflon bottle. Once quantity is weighed out, neutralize BrCl with 200 µL NH_2OH-HCl no more than five minutes prior to adding the sample to bubblers. The sample should turn from a yellowish color to a clear/cloudy solution, depending on the matrix.

16.4.6 If the material is a seawater or highly dense liquid, it may be necessary to account for the density if the aliquot is gravimetrically determined. Density

checks can be performed at the time of analysis to determine if further determinations are necessary.

16.4.7 The procedure for analysis is similar to that of the calibration. Samples to be analyzed are pipetted or poured into the bubbler (one sample per bubbler) along with 300µL $SnCl_2$. Bubbler tops are sealed with Keck Clips to ensure nominal sample leakage. Blanked gold traps are securely placed at the end of the soda-lime trap. Purge bubblers with N_2 for a minimum of 20 minutes, remove gold traps, and sequentially place in the analytical train. Burn individual traps to analyzer, labeling resulting peaks with corresponding sample in real time.

16.4.7.1 Sample IDs, aliquot volume, BrCl percentage (group ID), peak height/peak area, and dilution factor (if applicable) associated with each sample should be entered into the THg Waters Template Excel spreadsheet.

16.4.7.2 While purging one set of samples, the analyst should begin preparing the next round of water samples in the same fashion to maximize efficiency.

16.5 End of analysis close-down procedure:

16.5.1 Turn off gas flow.

16.5.2 Carryout all end of day cleaning and restocking tasks.

16.6 The analytical data is compiled into an Excel file. The data is then copied and pasted into an Excel template that is LIMS compatible.

16.7 Maintenance and Troubleshooting

16.7.1 ISSUE: No peaks at all

16.7.1.1 *Ensure that the system is powered.*

16.7.2 ISSUE: Low sensitivity

16.7.2.1 *Make sure that you have freshly changed soda lime in the soda lime trap, and that it is from a good source.*

16.7.2.2 *Do not use old calibration standards to calibrate the system.*

16.7.2.3 *Make sure you are running fresh $SnCl_2$ solution.*

16.7.2.4 *Make sure that your stock Hg standard has not expired and is from a reliable source and that it is not compromised.*

16.7.2.5 Check the lamp voltage

16.7.3 ISSUE: High blanks

[redacted]

16.7.3.1 Check reagent (including water) quality

16.7.3.2 Check for system contamination

16.7.4 ISSUE: Nonlinearity of the calibration curve

16.7.4.1 *Check and investigate high blanks.*

16.7.4.2 *Contaminated and expired soda lime. Change soda lime.*

16.7.4.3 *Make sure your calibration standards are fresh and properly prepared.*

17 Calculations:

17.1 Average all instrument blanks (PH_X) using the peak area values from the TekMDS software. Subtract the average (IB) from the peak area for each standard and sample.

17.2 Calculate the calibration factor (CF_x) for mercury in each of the five standards using the mean instrument-blank-subtracted peak area and the following equation:

$$CF_x = PA_x - IB / C_x$$

Where:

17.2.1 PAx=peak area (or peak height) for mercury in standard

17.2.2 IB= mean peak height (or peak area) for mercury in bubbler blank

17.2.3 Cx=mass in standard analyzed (ng/L)

17.2.4 CFx=Calibration Factor of each concentration

17.2.4.1 Average the five calibration factors to establish mean value: CF(Avg) (units/ng/L).

17.3 Sample results are then corrected for the average peak area values of at least three preparation blanks (PBs), unless otherwise requested. This result is shown as the Initial Result on the Excel spreadsheet and in LIMS.

17.4 Total Mercury in Water:

Instrument Value (ng/L) = (Peak Height – BB) / $CF_{(Avg)}$

Final Result (ng/L) = [(Instrument Value x DF) – (BLK)] x (Vf /Vi)

Where:

17.4.1 $CF_{(avg)}$ = average calibration factor for curve (in units/ng/L).

17.4.2 BB = average bubbler blank peak area or peak height (in units)

17.4.3 V_f = Final volume of sample (in mL) from bench sheet.

17.4.4 V_i = initial volume of sample analyzed in mL prior to addition of BrCl.

17.4.5 DF = Dilution Factor - takes into account any instrumental dilution of the sample

17.4.6 BLK = average of the preparation blanks in ng/L.

17.5 A linear regression can be used as alternate calibration. A linear regression will not change values significantly. If linear regression is used, the correlation coefficient (R) must be ≥0.995.

18 Statistical Information/Method Performance:

18.1 The Method Detection Limit (MDL) is determined according to 40 CFR Part 136 Section B. Ten replicates (9 degrees of freedom) spiked 3-10 times the expected MDL are run. The standard deviation (s) is taken from the resulting data and the MDL is calculated as follows: MDL=2.821*s. This value should not be interpreted as the method reporting limit.

18.2 The Practical Quantitation Limit (PQL) is the reporting limit for this method and is included as the lowest calibration point (2003 NELAC regulation 5.5.5.2.2.1.h.3). The PQL is determined by running ten samples with a concentration that will produce a recovery of 70-130 %. The PQL is referred to as the Method Reporting Limit (MRL) in LIMS.

18.3 Using clean handling techniques and reagents tested low for Hg content, the LOD value for Total Hg in water is typically less than 0.2 ng/L, while the PQL is 0.50 ng/L.

18.4 Current LODs, LOQs, MDLs, and PQLs are stored at: Cuprum\General and Admin\Quality Assurance\MDLs & PQLs.

19 Quality Assurance/Quality Control:

19.1 A minimum of three preparation blanks and one LCS/LCSD (preferably NIST 1641d), must be analyzed per preparation batch. The upper control limit for each preparation blank is equal to the PQL.

19.2 Matrix Spikes: One Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be performed for every 10 samples. The recovery of the MS/MSD must be between 71%-125% recovery, and the Relative Percent Difference (RPD) below 24%. If an MS/MSD is out of control, the analyst should investigate to identify the source of the failure. The MS and MSD may be used as duplicates. Some failures may be qualified using QA Qualification Flow Charts (Appendix A).

19.2.1 For aqueous samples, the MS/MSD is spiked at 1 to 5 times the ambient concentration, with 0.25 ng, in the bubbler, being the minimum spiking level. Sample aliquots for the MS/MSD should be the same as the ambient sample aliquot, if sufficient sample volume exists. Spikes are added to the split aliquots for volumes of 10mL or greater. For less than 10mL aliquots, spikes are added directly to the bubbler. NEVER ADD SPIKE DIRECTLY TO THE ORIGINAL SAMPLE VESSEL UNLESS OTHERWISE STATED.

19.3 Matrix Duplicates – One Matrix Duplicate (MD) may be analyzed for every batch of 20 samples. Upon request, a Matrix Triplicate (MT) may be performed. The MSD may serve as the MD if necessary. The Relative Percent Difference (RPD) and the Relative Standard Deviation (RSD) of duplicate samples must be less than 24%. Some failures may be qualified using QA Qualification Flow Charts.

19.3.1 For aqueous samples, analyze the parent, duplicate and triplicate at the same dilution.

19.4 Laboratory Control Standard (LCS) or Quality Control Sample (QCS): For every batch of samples, at least one LCS is processed and analyzed. The recovery of the LCS must be within 80-120% for the aqueous NIST 1641d. An LCS Duplicate (LCSD) should accompany the LCS.

19.4.1 A Certified Reference Material (CRM) is the preferred LCS, but a Blank Spike may serve as an LCS if an appropriate CRM does not exist. The spiking level is based on client request, historical data, or a default of mid-curve. A duplicate blank spike must also be prepared as an LCSD.

19.5 Ongoing Precision and Recovery (OPR): An OPR must be analyzed at the beginning and end of each analytical batch, or at the end of each 12-hour shift. The recovery of the OPR must be within 77-123% to be considered in control.

19.6 All calibration standards must be traceable to the original standard source. The calibration curve must be established at the beginning of the analytical run. It must include at least five different concentrations, with the lowest concentration equal to the PQL. The average response factor of each calibration standard is used to calculate the sample values. The RSD of the response factors must be less than 15% of the mean or the calibration fails.

19.7 ICV and CCV control limit is 77-123%. The CCV is analyzed every 10 analyses, and at the end of an analytical run. CCBs are always analyzed after the CCVs.

19.8 Field Blanks: To be compliant with EPA 1631, clients must submit a field blank for each set of samples (samples collected from the same site at the same time, to a maximum of 10 samples).

19.8.1 If no field blanks are submitted by the client, their data will be flagged with "FB-1631." "Required equipment/field/filter blank not submitted by the client. The sample has been analyzed according to 1631E, but does not meet 1631E criteria."

19.9 Method or Preparation Blanks (BLK): Method blanks are used to demonstrate that the analytical system is free from contamination that could otherwise compromise sample results. Method blanks are prepared and analyzed using sample containers, labware, reagents, and analytical procedures identical to those used to prepare and analyze the samples.

19.9.1 A minimum of three 1 % BrCl method blanks per analytical batch are required. Any sample requiring an increased amount of reagent must be accompanied by at least one method blank that includes an identical amount of reagent.

19.9.2 If the result for any 1 % BrCl method blank is found to contain ≥0.50 ng/L Hg (0.25 ng/L for DOD), the system is out of control. Mercury in the analytical system must be reduced until a method blank is free of contamination at the 0.50 ng/L level.

19.9.3 For method blanks containing more than 1% BrCl, the control limit is equal to 0.50 ng/L multiplied by the final preservation percentage of BrCl. For example, for a method blank preserved to 2 % BrCl, the control limit for the blank is 0.50

ng/L * (102/101), or 0.50 ng/L. For 3% BrCl the control limit is (103/101)*0.50ng/L, or 0.51ng/L.

19.10 Instrument Blanks (IBL): A minimum of three instrument blanks must be analyzed with each analytical batch. To analyze an instrument blank, attach a clean gold trap to the bubbler. Purge and analyze as previously described and determine the amount of Hg remaining in the system.

19.10.1 An instrument blank must be performed on all bubblers used during the analytical run (normally four, but three at a minimum).

19.10.2 If the instrument blank is found to contain more than 0.50ng/L, the system is out of control. The problem must be investigated and remedied and the samples run on that bubbler must be reanalyzed. If the blanks from other bubblers contain less than 0.50 ng/L, the data associated with those bubblers remain valid, provided that all other QC criteria are met.

19.10.2.1.1 The mean result for all instrument blanks must be <0.25ng/L with a standard deviation of 0.10 ng/L.

19.11 The analytical day must close with a CCV/OPR/CCB.

19.12 Because the method is done in real-time, it is EFGS' position that a single non-compliant QC sample result does not automatically invalidate a data set. All data points that can be explained and rerun with a passing result can be qualified. If the source of error cannot be corrected for a QC standard that day, none of the data can be validated. In the event that the system becomes out of control during the analysis day, all results bracketed between valid QC data points shall still be considered valid (CCV, OPR, CCB, etc).

19.13 The Control Limits are established from EPA 1631E.

20 Corrective Action

20.1 The data is reviewed as in the QC section (or matrix specific QC section) for all parameters that pass specific requirements. If the data does not meet QC requirements it is qualified or submitted for reruns. Data may be qualified (based on scientific peer review) by the Group Supervisor, Project Manager, Lab Manager, or QA Officer.

20.2 Control Chart data is generated through LIMS to monitor the performance of the CCV, LCS, MS, and MSD. This is done by the QA department.

20.3 Due to the real-time nature of the CVAFS method, failures must be investigated as they happen. If the source of the problem can be identified, and corrected, the samples may be rerun. If source of problem cannot be isolated, see the Senior Analyst, Group Supervisor, or Laboratory Manager for instructions.

20.4 The Senior Analyst, Group Supervisor, Laboratory Manager, or QA Officer must be informed if QC fails. It is also advisable to always alert the Project Managers.

21 List of Attachments

Table 1: QC Requirements for Total Mercury

Appendix A: Example - Standard Operating Procedure Training Record

[redacted]

Table 1: QC Requirements for Total Mercury

QC Parameter	Acceptance Criteria
Initial Calibration Verification (ICV)	77-123% Recovery
Continuing Calibration Verification (CCV)	77-123% Recovery
Ongoing Precision and Recovery (OPR)	77-123% Recovery
Initial Calibration Blank (ICB)/ Continuing Calibration Blank (CCB)	Individually, IBL and CCB ≤0.50ng/L, but the mean of all the IBLs shall be <0.25ng/L with a standard deviation of 0.10ng/L.
Laboratory Control Standard (LCS) or Quality Control Standard (QCS)	80-120% Recovery for NIST1641d and 75-125% for all other CRMs. RSD<24%
Calibration Curve RSD (Referred to as "Corr. RSD CF" in Excel spreadsheet).	RSD of Calibration Response Factor ≤15%
Lowest Calibration Point	75-125%
1% BrCl Method Blank (BLK)	Less than 0.50ng/L (0.25ng/L for DOD projects) (individually)
Matrix Duplicate (MD) and Analytical Duplicate (AD)	< 24% RPD
Matrix Spike and Matrix Spike Duplicate (MS/MSD) ; Analytical Spike (AS) and Analytical Spike Duplicate (ASD)	71-125% Recovery < 24% RPD

Appendix A: Example - Standard Operating Procedure Training Record

By signing this document, I the employee, certifies to have read, understood and agreed to follow the test method and quality procedure as described in this procedure.

Reading of SOP FGS-137.02:
Mercury in Water by Oxidation, Purge & Trap and CV-AFS (EPA Method 1631, Rev E.

SOP name and Revision number

Employee name (print)

Employee name (sign) Date:

Supervisor name (sign) Date:

Initial SOP Training (leave blank if not applicable)

Initial reading of method and training	Initials	Date	Supervisor
1. Read method			
2. Observe the method			
3. Detailed review of method and associated literature			
4. Supervised practice of method with trainer			
5. Unsupervised practice of the method with trainer			
6. Review of work with trainer and/or peer-review			
7. IDOC to determine precision and accuracy			
8. Determination of blanks			

[redacted]

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Local Document Type	SOP		
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Prepared by	Ryan Nelson
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[redacted]

Table of Contents

[redacted]

Approvals:

Prepared by: _____ Date: 5/20/13

Approved by: _____ Date: 5/16/13

Approved by: _____ Date: 5/20/13

1 Revision Log:

Revision: 05	Effective Date: This version	
Section	**Justification**	**Changes**
Cover	Required change	Changed company name from Frontier Global Sciences to Eurofins Frontier Global Sciences.
All	Formatting requirement per LOM SOP-LAB-201	Reformatted document to new corporate specifications.
8.9	Required	Updated spiking levels for the matrix spike
14.3, 14.4	Required	Updated max contamination levels of reagent acids
17.3	Required	Replaced MDL with LOD
18.2 – 18.5	Required	Updated QC limits
18.3	Required	Incorporated QA MOC 2011-007

2 Reference:

2.1 Chemical Hygiene Plan, Eurofins Frontier Global Sciences, current version.

2.2 EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, 2002.

2.3 National Environmental Laboratory Accreditation Conference, NELAC Standard September 8, 2009.

2.4 Department of Defense Quality Systems Manual for Environmental Laboratories, prepared by DoD Environmental Quality Workgroup, Final Version 4.2, October 2010

3 Cross Reference:

Document	Document Title
SOP FGS-003	Pipette Verification, Calibration and Maintenance
SOP FGS-008	Ultra Clean Aqueous Sample Collection
SOP FGS-038	Data Review and Validation
SOP FGS-094, App F	Standard Operating Procedure Training Record
SOP FGS-099	Waste Disposal Procedure for Client Sample Waste
SOP FGS-121	Determination of Total Mercury in Various Matrices by Flow Injection Atomic Fluorescence Spectrometry (EPA Method 1631E)
SOP FGS-155	Calibration of Volumetric Dispensers

4 Purpose:

4.1 The purpose of this Standard Operating Procedure (SOP) is to describe the method for digesting biological tissue samples prior to analysis by CV-AFS for total mercury.

5 Scope:

5.1 This method is for the preparation of biological tissue samples for the determination of total mercury at concentrations less than 1 ng/g. Through the analysis of smaller digestate aliquots, contaminated tissues of up to 10,000 ng/g can be directly measured. Using clean handling techniques and low-level reagents, the typical detection limit for samples prepared by this method is less than 1 ng/g.

5.2 Total mercury, as defined by this method, is all HNO3/H2SO4/BrCl-oxidizable mercury forms and species found in tissue matrices. This includes, but is not limited to, Hg(II), Hg(O), HgS, strongly organo-complexed Hg(II) compounds, adsorbed particulate Hg,

and several covalently bound organo-mercurials (i.e., CH_3HgCl, $(CH_3)_2Hg$, and $C_6H_5HgOOCCH_3$).

6 Basic Principles:

6.1 Samples are collected using clean sample handling protocols into commercially available clean glass containers with Teflon-lined caps (i.e., I-Chem glass jars) or 125 mL or 250 mL HDPE jars. Freezing (< -15°C) preserves tissue samples until sample preparation is performed.

6.2 A subsample of homogenized sample is digested with 10 mL of 70:30 HNO_3/H_2SO_4.

6.3 The digested sample is diluted up to 40 mL with 10% (v/v) BrCl.

7 Reference Modifications:

7.1 No significant modifications were made to this method.

8 Definitions:

8.1 Batch – no more than 20 client samples grouped for preparation. 3 Preparation Blanks, *1 CRM* or 1 LCS/LCSD (or BS/BSD) set and 1 MD are prepared per every 20 samples; 1 MS/MSD set is prepared for e*very 10 samples*.

8.2 Celsius (C), conversion of Celsius to Fahrenheit: (C * 1.8) + 32.

8.3 Fahrenheit (F), conversion of Fahrenheit to Celsius: (F - 32) * 5/9.

8.4 Method Detection Limit (MDL) – the limit derived from an exercise as described in 40 CFR, Part 136, Appendix B. The exercise produces a defined value that is the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero from a given matrix.

8.5 Certified Reference Material (CRM) – a standard of known composition that is certified by a recognized authority and representing a sample matrix. It is used to verify the accuracy of a method.

8.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD), is a sample containing known concentrations of the analytes of interest that is taken through the entire preparation and analysis process in the same manner as the samples to monitor complete method performance. A Certified Reference Material (CRM) is preferred as the LCS, but a blank spiked sample also meets the requirement.

8.7 Preparation Blank (BLK) – Method blanks consist of the same reagents used to digest the samples, in the same volume or proportion, and are carried through the complete sample preparation and analytical procedure. Teflon boiling chips are added to the preparation blanks.

8.8 Matrix Duplicate (MD) – a representative sample is selected and digested in the same manner. This QC sample will indicate sample homogeneity on the analytes of interest

8.9 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – a representative sample is selected and spiked with a secondary source at *two* to five times the ambient

concentration or at *two* to five times the MRL, whichever is greater. These QC samples will indicate sample matrix effects on the analytes of interest.

8.10 May: This action, activity or procedure is optional.

8.11 May Not: This action, activity or procedure is prohibited.

8.12 Shall: This action, activity or procedure is required.

8.13 Should: This action, activity or procedure is suggested, but is not required.

9 Interferences:

9.1 Due to the high levels of halogens (i.e., iodine) typically found in tissue digestates, it is recommended that aliquots of no more than 5.0 mL of the digestate be analyzed. Otherwise, soda-lime traps may be overloaded and the gold traps may lose the ability to amalgamate and retain mercury.

9.2 The high acidity and halogen levels that are found in tissue digestates necessitate the changing of the bubbler water after every 10 mL of digestate analyzed. Failure to do so can lead to low recoveries that would be reflected in the analysis of QC samples.

10 Safety Precautions, Pollution Prevention and Waste Handling:

10.1 Personnel will don appropriate laboratory attire according to the Chemical Hygiene Plan. This includes, but is not limited to, laboratory coat, safety goggles, and nitrile gloves under clean gloves.

10.2 The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Chemists should refer to the MSDS (Material Safety Data Sheets) for each chemical they are working with.

10.2.1 Note: Use particular caution when preparing and using BrCl, as it releases extremely irritating, corrosive fumes similar in effect to free chlorine. Always handle this reagent in an approved fume hood.

10.2.2 Note: Use particular caution when preparing and using the Nitric/Sulfuric Mixture. Always handle this reagent in an approved fume hood.

10.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease-causative agents. Eurofins Frontier will reimburse the expense of Hepatitis A and B immunizations for any laboratory staff member who desires this protection.

10.4 Nitric acid (HNO_3): Corrosive. Strong oxidizer. Contact with other material may cause a fire. Causes eye and skin burns. May cause severe respiratory tract irritation with possible burns. May cause severe digestive tract irritation with possible burns. For more information see MSDS.

10.5 Sulfuric acid (H_2SO_4): Corrosive. Causes eye and skin burns. May cause severe eye irritation with possible burns. May cause severe respiratory tract irritation with possible burns. May cause severe digestive tract irritation with possible burns. Cancer hazard.

Animal studies suggest this acid may cause fetal effects. May cause kidney damage. May cause lung damage. May be fatal if inhaled. Hygroscopic. Strong oxidizer. Contact with other material may cause a fire. For more information see MSDS.

10.5.1 Eyes: Get medical aid immediately. Do NOT allow victim to rub or keep eyes closed. Extensive irrigation with water is required (at least 30 minutes).

10.5.2 Skin: Get medical aid immediately. Flush skin with soap and water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Destroy contaminated shoes.

10.6 See Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP) for general information regarding employee safety, waste management, and pollution prevention.

10.7 Pollution prevention information can be found in the current Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP), which details and tracks various waste streams and disposal procedures.

10.8 All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations. Any waste generated by this procedure should be disposed of according to SOP FGS-099 "Waste Disposal Procedure for Client Sample Waste," which provides instruction on dealing with laboratory and client waste.

11 Personnel Training and Qualifications:

11.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on CRMs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

11.2 The analyst/laboratory technician must have read this SOP and other relevant SOPs and have the training documented on the applicable form(s). The analyst may be questioned on SOP by supervisor(s) and/or trainers.

11.3 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

11.4 Reading of the SOP must be documented on the correct form such as "Standard Operating Procedure Training Record," Appendix F in FGS-094, the last page of this SOP, Appendix A "Standard Operating Procedure Training Record" or a similar document."

11.5 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

11.6 All training documents including IDOCs, CDOCs, SOP reading, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11.7 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

12 Sample Collection, Preservation, and Handling:

12.1 Samples must be collected in accordance with established ultraclean sampling techniques (see FGS-008 "Ultra Clean Aqueous Sample Collection"). Samples may be in commercially available clean glass containers with Teflon-lined caps (i.e., I-Chem glass jars), or 125 mL or 250 mL HDPE jars.

12.2 Tissue sample preservation - The tissue sample must be frozen in the sampling container at less than -15°C or freeze-dried and stored at room temperature. The holding time for tissue samples is 1 year.

12.3 Just prior to digestion, samples are thawed and if necessary homogenized. The sample is well mixed to ensure the most representative sample possible.

13 Apparatus and Equipment:

13.1 LIMS – Element, version 5.85 or higher; Computer – Windows XP, 7 or 8

13.2 40 mL or 20 mL I-Chem Vials: Borosilicate glass, series 300 vials with Teflon-lined septa in lids. The size used depends on the amount of sample available. The vials are volumetrically accurate to ± 0.5 mL when filled such that the meniscus is just to the bottom of the vial neck. The person performing the preparation should verify this.

13.3 Hot plate: A hot plate with the ability to achieve and maintain a temperature of 75 °C.

13.4 *Pipettors: All-plastic, pneumatic, fixed volume and variable pipettes in the range of 5 µL to 10 mL. Pipettes are to be calibrated weekly according to SOP FGS-003 and FGS-155.*

13.5 Clean hood.

13.6 Analytical Balance: A laboratory analytical balance capable of weighing to ± 1 mg, with documented calibration.

13.7 Calibrated thermometer: Submerged in water in a 20 mL I-Chem vial. This vial is placed on the hotplate during the digestion process. The analysts must record the actual digestion temperature and the serial number of the thermometer used in the digestion logbook.

13.8 Sample Digestion Log.

13.9 Stainless steel tools for homogenization

13.10 Tissue Homogenization Log.

13.11 Disposable spatula.

13.12 Teflon boiling chips.

13.13 Teflon reflux cap to fit the 40 mL and 20 mL I-Chem vials.

14 Reagents and Standards:

14.1 Reagent Water: 18 MΩ ultra-pure deionized water starting from a pre-purified (distilled, R.O., etc.) source. As a final mercury and organic removal step, the activated carbon cartridge on the 18-MΩ system is placed between the final ion exchange bed and the 0.2 µm filter.

14.2 Nitric Acid (HNO_3): Trace metal purified reagent-grade HNO3 is pre-analyzed and lot sequestered. Several brands (Baker, Fisher, Omnitrace) have been found to have lots with acceptably low levels of trace metals. This reagent should be from a lot number that has been previously tested to be low for the analytes of interest. This reagent shall be entered into LIMS and the expiration date is set to the same as the manufacturer's expiration date.

14.3 Sulfuric acid (H_2SO_4) - Trace metal purified reagent-grade H_2SO_4 is pre-analyzed to < 50 ng/L Hg and lot sequestered before purchase. This reagent shall be entered into the LIMS and is considered stable until the expiration date on the bottle (set by the manufacturer.

14.4 Nitric/Sulfuric Acid Mixture: Carefully add 300 mL of pre-analyzed, low mercury (< 50 ng/L) concentrated sulfuric acid to 700 mL of pre-analyzed, low mercury concentrated nitric acid to a pre-marked Teflon bottle. Stir constantly. This reagent shall be entered into the LIMS with an expiration date of six months. **CAUTION: THIS MIXTURE BECOMES VERY HOT AND EMITS CAUSTIC FUMES**.

14.5 Potassium Bromide (KBr), neat: this reagent is pre-certified by the vendor to be low in mercury and is entered into the LIMS with a five year expiration date.

14.6 Potassium Bromate ($KBrO_3$), neat: this reagent is pre-certified by the vendor to be low in mercury and is entered into the LIMS with a five year expiration date.

14.7 0.2N Bromine Monochloride (BrCl):

14.7.1 37.5 g of KBr is added to a 2.5 L bottle of concentrated HCl (pre-analyzed and below 5 ng/L Hg). The bottle is inverted in a fume hood to mix the acid and KBr. The solution sits overnight, allowing the KBr to dissolve.

14.7.2 27.5 g of KBrO3 (certified to be low in Hg) is slowly added to the acid. As the KBrO3 is added, the solution should go from yellow to red to orange.

CAUTION: This process generates copious quantities of free halogens (Cl_2, Br_2, BrCl) which are released from the bottle. Add the $KBrO_3$ SLOWLY in a well operating fume hood.

14.7.3 Loosely cap the bottle and allow to sit for 30 minutes (in a fume hood) before tightening. Once tightly capped, invert bottle to make sure all of the solids go into solution.

14.7.4 This reagent shall be entered into the LIMS with a six month expiration date.

14.8 10% (v/v) of 0.2N BrCl: 200 mL of 0.2N BrCl is diluted up to 2.0 L with reagent water in a clean, empty HCl bottle. This bottle is fitted with a 10 mL repipettor. The expiration time for this reagent is set by default to six months in the LIMS.

15 Procedure:

15.1 If needed, the sample is dissected and homogenized with acid-washed stainless steel tools.

15.1.1 The process used for homogenization, number of samples, work order number, client name, and initials of the technician are entered into the Tissue Homogenization Log.

15.2 Weigh at least a 0.5 g aliquot (but not more than 0.65 g) for common and unknown samples, and up to 1.0 g ± 0.025 g for low-level or large-grain samples. This aliquot is placed into a 40 mL I-Chem glass vial.

15.2.1 If limited sample is available, use 20 mL glass vials and drop the initial mass of the samples to 0.25g ± 0.025 g.

15.2.2 It is imperative that all biological tissue samples are thoroughly homogenized. The importance of representativeness cannot be understated.

15.2.3 Batch requirements for this digestion limit the number of samples to 20. In each batch, there must be three method blanks (BLKs), a Blank Spike and Blank Spike Duplicate (BS/BSD) that is *preferably a Certified Reference Material (CRM) or a Laboratory Control Spike* (LCS, *prepared at 8 ng/g*), a Matrix Duplicate (MD), and a Matrix Spike and Matrix Spike Duplicate (MS/MSD).

15.3 10.0 mL of 70:30 (v/v) HNO_3/H_2SO_4 solution is pipetted in and the sample is swirled. *Note: 5.0 mL of 70:30 (v/v) HNO_3/H_2SO_4 solution is used for limited samples prepared in 20 mL vials (15.2.1).*

15.4 The vial is placed on a hot plate operating at 75±5°C with a Teflon reflux can in place instead of the vial's lid. An aluminum rack id often used to keep the vials from tipping over while on the hot plate.

15.4.1 A calibrated thermometer submerged in water is placed in a 20 mL I-Chem vial. This I-Chem vial with a calibrated thermometer is placed on the hot plate during the digestion process. The analysts must record the actual digestion temperature and the serial number of the thermometer used in the digestion logbook.

15.5 After the samples start to reflux, the samples are heated at 75±5°C for an additional 2 hours or until all organic matter is dissolved.

15.6 The samples are allowed to cool and are diluted to 40 mL (*or to 20 mLs for limited sample digestions as described in 15.2.1*) with a 10% (v/v) solution of 0.2N BrCl, capped with their respective lids, and are thoroughly shaken. Sample digestates should be allowed to settle prior to an aliquot being taken for analysis.

15.7 Analysis for total mercury is according to Eurofins Frontier *SOP FGS-121.*

16 Calculations:

16.1 This preparation procedure does not involve calculations.

17 Statistical Information/Method Performance:

17.1 Method Detection Limit (MDL) and Practical Quantitation Limit (PQL) studies are based on 40 CFR 136, Appendix B. The MDL and PQL must be performed for each analyte/matrix/preparation combination.

17.2 The Practical Quantitation Limit (PQL) is the reporting limit for this method and is included as the lowest calibration point (2003 NELAC regulation 5.5.5.2.2.1.h.3). The PQL is determined by running ten replicate samples with a concentration that will produce a recovery of 70-130% for most analytes, but the recovery requirements are analyte dependent. The PQL is referred to as the Method Reporting Limit (MRL) in LIMS.

17.3 The current LOD value for Total Hg in tissue prepared by the Nitric and Sulfuric Acids (70:30) Digestion is 0.16 ng/g, while the PQL is 0.8 ng/g.

17.4 Current LODs and PQLs are stored at: \General and Admin\Quality Assurance\MDLs & PQLs.

18 Quality Assurance/Quality Control:

18.1 Maximum Sample Batch Size: 20 samples.

18.2 Preparation Blanks: Minimum of three per batch. Each preparation blank must be less than *one-half* the PQL for the method.

18.2.1 The preparation blanks are prepared with a similar mass of Teflon boiling chips as the samples, with the same reagents, and put through the same preparation process as the samples.

18.3 *Certified Reference Material (CRM, representing the sample matrix when commercially available); a Laboratory Control Spike (LCS) and Laboratory Control Spike Duplicate (LCSD) prepared at 8 ng/g is used when a suitable CRM is not available*: One per batch in duplicate. The control limits are *77-123%* recovery.

18.4 Matrix Duplicate (MD) Sample: One per batch. The control limit for the RPD is ≤ *24%*.

18.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Samples: One set per *10* samples. The control limits are *71-125%* recoveries and an RPD of ≤ *24%*.

18.6 Follow the flow charts in SOP FGS-038 "Data Review and Validation" to determine if any QC falling outside the established control limits can be qualified.

18.7 All of the quality control limits for the analysis method are included on the "Data Review Checklist.

18.7.1 The data review checklists are located at: \\cuprum\General and Admin\Quality Assurance\Data Review\Current Data Review Checklists.

19 Corrective Action:

19.1 Limiting the source of contamination/error in the preparatory stage can decrease QC problems during analysis. Limiting such contamination/error sources may include: cleaning all digestion tools in a 10% HCl solution, ensuring all samples are thoroughly homogenized, changing gloves whenever appropriate, flushing repipettors at least

three times before dispensing into vials and, in general, following ultra-clean procedures.

19.2 A failing QC point does not necessary fail the entire dataset. If upon analysis a QC sample is out of control, some investigation must be performed to assess if the difficulties are related to matrix effects. The cause and method of determining the set's failure must be documented on the checklist and in the MMO notes, and the Group Supervisor shall be informed. See SOP FGS-038 "Data Review and Validation" for flow charts regarding analytical issues.

19.3 Additional corrective actions are listed in the SOP for total mercury analysis (Eurofins Frontier SOP FGS-121).

20 List of Attachments

Appendix A: Example - Standard Operating Procedure Training Record

Appendix A: Example - Standard Operating Procedure Training Record

By signing this document, I the employee, certifies to have read, understood and agreed to follow the test method and quality procedure as described in this procedure.

Reading of SOP EFGS-011.05:
Digestion of Tissues for Total Mercury Analysis Using Nitric and Sulfuric Acids (70:30).

SOP name and Revision number

Employee name (print)

Employee name (sign) Date:

Supervisor name (sign) Date:

Initial SOP Training (leave blank if not applicable)

Initial reading of method and training	Initials	Date	Supervisor
1. Read method			
2. Observe the method			
3. Detailed review of method and associated literature			
4. Supervised practice of method with trainer			
5. Unsupervised practice of the method with trainer			
6. Review of work with trainer and/or peer-review			
7. IDOC to determine precision and accuracy			
8. Determination of blanks			

APPENDIX B

Quality Assurance Project Plan for Sample Preparation for the 2013-2014 National Rivers and Streams Assessment Fish Fillet Indicator

Quality Assurance Project Plan for

Sample Preparation for the 2013-2014 National Rivers and Streams Assessment Fish Fillet Indicator

December 12, 2013

Prepared jointly by:

United States Environmental Protection Agency
Office of Water
Office of Science and Technology
Standards and Health Protection Division

Tetra Tech, Inc.
under:
Office of Science and Technology
Contract No. EP-C-09-019

and

CSC
under:
Technical, Analytical, and Regulatory Mission Support for the Water Security Division
Contract No. EP-C-10-060

Quality Assurance Project Plan for Sample Preparation for the 2013-2014 National Rivers and Streams Assessment Fish Fillet Indicator

A. PROJECT MANAGEMENT

This Quality Assurance Project Plan (QAPP) has been prepared by the EPA Office of Science and Technology (OST). It presents performance requirements, acceptance criteria, and objectives for the preparation of tissue samples from whole fish composite samples collected by field crews during the 2013 and 2014 sampling seasons of the National Rivers and Streams Assessment (NRSA). It does not address the fish sample collection because that process is already covered by a separate QAPP (USEPA 2013a) prepared by the Office of Wetlands, Oceans, and Watersheds (OWOW). OST will revise this QAPP at a later date to include the details of the analyses of the fillet tissue samples prepared under this QAPP for various environmental contaminants.

This QAPP was prepared in accordance with the most recent version of EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans* (USEPA 2001), which was reissued in 2006. In accordance with EPA QA/R-5, this QAPP is a dynamic document that is subject to change as project activities progress. Changes to procedures in this QAPP must be reviewed by the OST Project Manager and the EPA Standards and Health Protection Division (SHPD) Quality Assurance Coordinator for the NRSA to determine whether the changes will impact the technical and quality objectives of the project. If so, the QAPP will be revised accordingly, circulated for approval, and forwarded to all project participants listed in the QAPP distribution list (Section A3). Key project personnel and their roles and responsibilities are discussed in the QAPP section to follow (Section A4), and project background perspective and description is provided in Sections A5 and A6, respectively.

A1. Approvals

Leanne Stahl, OST Project Manager, EPA — 12/16/13 Date

Denise Hawkins, Chief, FSBOB, EPA — Dec. 16, 2013 Date

Robert Shippen, SHPD QA Coordinator, EPA — 12/16/13 Date

Marion Kelly, OST QA Officer, EPA — 12/17/13 Date

Blaine Snyder, Tetra Tech Project Leader — 12/16/13 Date

Susan Lanberg, Tetra Tech QA Officer — 12/13/2013 Date

Harry McCarty, CSC Project Leader — 12/12/2013 Date

Marguerite Jones, CSC QA Officer — 12/12/2013 Date

Laura Flynn Jenkins, EPA Project Officer — 12/18/13 Date
Contract No. EP-C-10-060

A2. Table of Contents

TABLES

FIGURES

APPENDICES

LIST OF ACRONYMS AND ABBREVIATIONS

EPA	Environmental Protection Agency
FSBOB	Fish, Shellfish, Beach, and Outreach Branch
ID	Identification
NRSA	National Rivers and Streams Assessment
ORD-WED	Office of Research and Development - Western Ecology Division
OST	Office of Science and Technology
OW	Office of Water
OWOW	Office of Wetlands, Oceans, and Watersheds
PBDE	Polybrominated diphenyl ether
PCB	Polychlorinated biphenyl
PFC	Perfluorinated compound
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
QSA	Quality system audit
SHPD	Standards and Health Protection Division
SOP	Standard operating procedure
SOW	Statement of work
TBD	To be determined

A3. Distribution List

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A4. Project/Task Organization

EPA's 2013-2014 National Rivers and Streams Assessment (NRSA) is a probability-based survey designed to assess the condition of the nation's river and stream resources. It includes collection and analysis of physical, chemical, and biological indicator data that will allow a statistically valid characterization of the condition of the nation's rivers and streams. EPA used an unequal probability design to select 1808 streams and rivers (both wadeable and non-wadeable) from across the 48 contiguous United States. To improve the ability to assess changes over time (i.e., trends analysis), the design includes revisits to 811 sites that were sampled during the 2008-2009 NRSA. The Office of Wetlands, Oceans, and Watersheds (OWOW) within the Office of Water (OW) is responsible for the overall planning and implementation of the 2013-2014 NRSA.

EPA's Office of Science and Technology (OST) within OW is collaborating with the Office of Research and Development Western Ecology Division (ORD-WED) in Corvallis, Oregon to plan and implement the fish fillet indicator under the 2013-2014 NRSA. OST is responsible for management of this indicator. ORD-WED developed the study design and selected all the sampling locations, including approximately 450 sites from which whole fish composite samples will be collected for fillet analysis. Statisticians in the Western Ecology Division will also be analyzing the fillet tissue concentration data.

Routine composite samples for the NRSA fish fillet indicator consist of five similarly sized adult fish of a single species commonly consumed by humans. All of the fish samples collected for this indicator are being shipped as whole fish to Microbac Laboratories in Baltimore, Maryland, the designated sample preparation laboratory. Staff at Microbac will prepare the fish samples for analysis (i.e., filleting the fish samples and homogenizing the fillet tissue). OST currently plans to analyze the fillet tissue samples from all sites for mercury and from the urban sites only for perfluorinated compounds (PFCs). The 2013-2014 NRSA may also include future analysis of fillet tissue samples for polybrominated diphenyl ethers (PBDEs) and polychlorinated biphenyls (PCBs).

In 2013, OWOW developed the *National Rivers and Streams Assessment 2013-2014 Quality Assurance Project Plan* (USEPA 2013a) and the *National Rivers and Streams Assessment Field Operations Manual* (USEPA 2013b). The procedures and associated quality assurance/quality control (QA/QC) activities for collecting and shipping NRSA whole fish samples for fillet analysis were described in both documents. OST developed whole fish collection and shipping procedures for the 2013-2014 NRSA based on the protocols used for EPA's National Lake Fish Tissue Study and the 2008-2009 NRSA. This additional QAPP covers activities associated with preparing NRSA whole fish samples for fillet tissue analyses.

The fish fillet indicator project team currently consists of managers, scientists, statisticians, and QA personnel in OST and the ORD Western Ecology Division, along with contractors providing scientific and technical support to OST from CSC and Tetra Tech (see Figure 1). Project team members are providing support for developing and reviewing technical and program information related to all aspects of the indicator, including training materials, standard operating procedures, QAPPs, analytical QA reports, briefings and reports on indicator results, and outreach materials. Responsibilities for key members of the project team are described below.

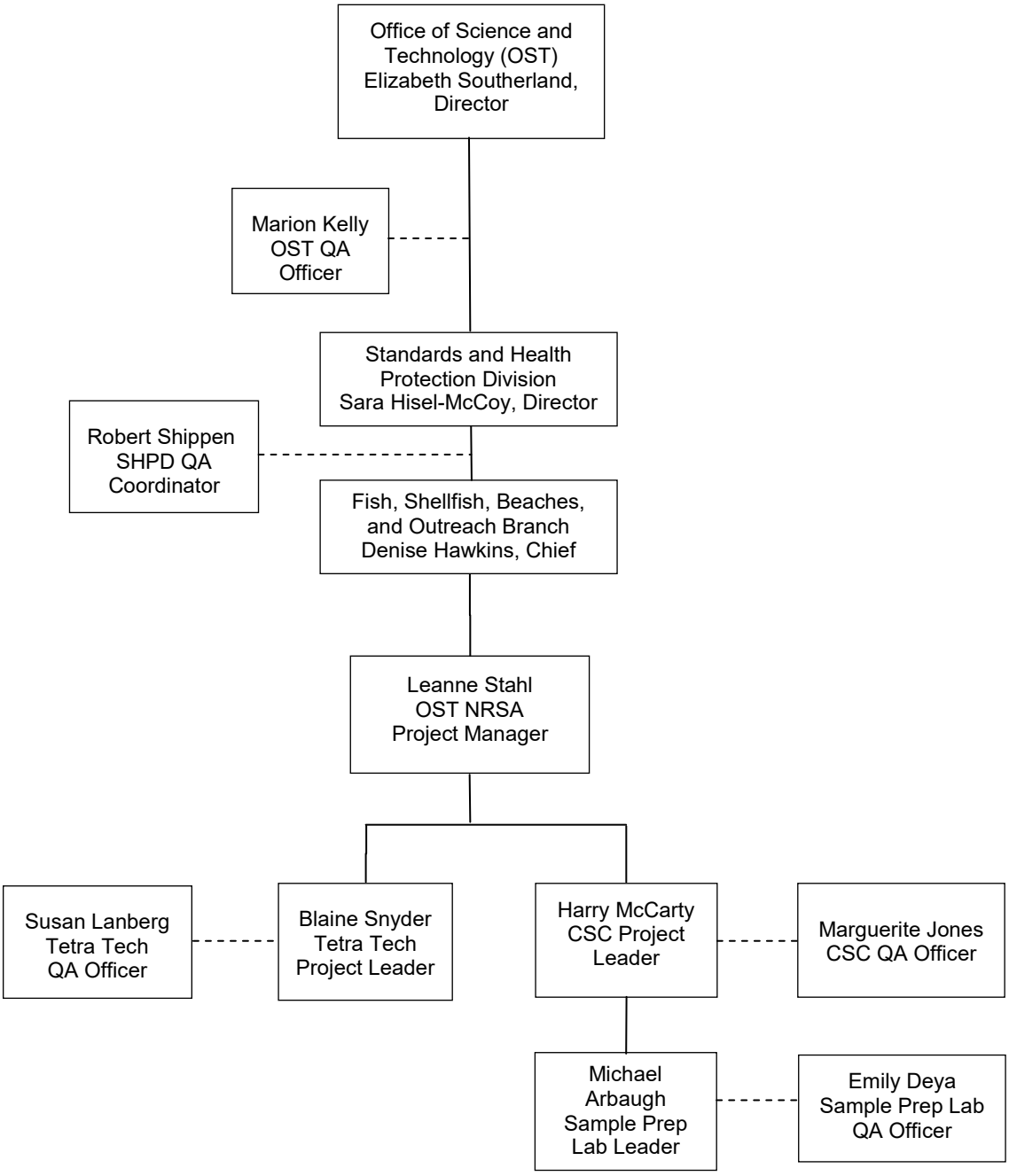

Figure 1. NRSA fish fillet indicator project team organization

Leanne Stahl of OST is the **NRSA fish fillet indictor Technical Leader and OST Project Manager** who is providing overall direction for planning and implementation of this fillet tissue study being conducted under the NRSA. This role involves the following responsibilities related to the fish fillet indicator:

- developing technical information for whole fish sample collection for fillet analysis that includes preparation of the sampling SOP and coordination with the NRSA Project

Leader in OWOW to integrate field sampling technical information for the fish fillet indicator into NRSA documents and training materials

- providing technical support to conduct training on the fish tissue sampling requirements in coordination with the NRSA Project Leader in OWOW
- developing the fish preparation SOP, implementing training for laboratory processing of NRSA fish samples, and providing technical direction for and oversight of fish preparation activities, including technical support for review of fish preparation QA data
- managing analysis of fish samples for target chemicals, including obtaining technical support for chemical analysis of fish tissue, directing development of this QAPP, providing for QA review of the analytical results, developing the data files for statistical analysis of the data, reviewing and approving the final analytical QA report, and providing oversight for development of the database to store NRSA fillet tissue results
- facilitating communication among fish fillet indictor project team members and coordinating with all of these individuals to ensure technical quality and adherence to QA/QC requirements
- developing and managing work assignments under OST or other EPA contracts to provide technical support for the NRSA, providing oversight of all OST contractor activities, and reviewing and approving study deliverables for each work assignment
- scheduling and leading meetings and conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study
- working with QA staff to identify corrective actions necessary to ensure that study quality objectives are met
- managing the development of and/or reviewing and approving all major work products associated with the NRSA fish fillet indicator
- collaborating with the NRSA project team for reporting the indicator results in technical journal articles and federal technical reports

Marion Kelly is the **OST Quality Assurance Officer** who is responsible for reviewing and approving all QAPPs that involve scientific work being conducted by OST. Robert Shippen is the **Standards and Health Protection Division (SHPD) QA Coordinator** who is responsible for reviewing and recommending approval of all QAPPs that include scientific work being conducted by SHPD within OST. The OST QA Officer and SHPD QA Coordinator are also responsible for the following QA/QC activities:

- reviewing and approving this QAPP
- reviewing and evaluating the QA/QC requirements and data for all the NRSA fish fillet indicator activities and procedures
- conducting external performance and system audits of the procedures applied for all NRSA fish fillet indicator activities
- participating in Agency QA reviews of the study

Blaine Snyder is the **Tetra Tech Project Leader** who is responsible for managing all aspects of the technical support being provided by Tetra Tech staff for the NRSA fish fillet indicator. His specific responsibilities include the following:

- providing direct technical support for the following NRSA fish fillet indicator activities or providing leadership and oversight for Tetra Tech staff supporting these activities:
  - developing standard operating procedures for fish sampling, handling, and shipment
  - preparing NRSA fish fillet indicator training materials and project information to incorporate into NRSA documents
  - providing field sampling and fish preparation training
  - planning and implementing NRSA fish fillet indicator logistics
  - conducting fish sampling at NRSA sites designated by the OST Project Manager
  - obtaining and performing QA reviews of NRSA field sampling data related to the fish fillet indicator
  - preparing fish tissue sample preparation instructions for whole fish samples collected from designated NRSA sites
  - evaluating weekly fish processing reports for adherence to the technical and quality requirements in the fish preparation SOP
  - preparing summary project information and graphics for development of project fact sheets, presentations, and other EPA meeting and outreach materials
- monitoring the performance of Tetra Tech staff participating in this study to ensure that they are following all QA procedures described in this QAPP that are related to Tetra Tech tasks being performed to support this study (see list above)
- ensuring completion of high-quality deliverables within established budgets and time schedules
- participating in meetings and conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study

Susan Lanberg is the **Tetra Tech QA Officer**, whose primary responsibilities include the following:

- assisting Tetra Tech's Project Leader with the review of this QAPP
- approving this QAPP
- providing oversight for the implementation of QA procedures related to Tetra Tech tasks that are described in this QAPP
- reporting deviations from this QAPP to the Tetra Tech Project Leader and assisting in implementing corrective actions to resolve these deviations

Harry McCarty is the **CSC Project Leader** who is responsible for managing all aspects of the technical support being provided by CSC staff for the NRSA fish fillet indicator. His specific responsibilities include the following:

- providing direct technical support for the following NRSA fish fillet indicator activities or providing leadership and oversight for CSC staff supporting these activities:
  - preparing information related to technical and quality assurance requirements for preparation of fish tissue samples and for analyses of fillet tissue samples for target chemicals designated by OST, validation of analytical data, and database development to support project planning and development of NRSA fish fillet indicator documents (including this QAPP) or characterization of this indicator in NRSA documents
  - obtaining laboratory services for whole fish sample preparation and fillet tissue analyses, and providing technical and QA oversight of laboratory operations
  - conducting reviews of fish preparation QA/QC data associated with each batch of up to 20 fish samples and preparing a report about the results of each batch review for distribution to the OST Project Manager and the fish preparation laboratory
  - obtaining freezer space that meets the requirements for long-term storage of archived fish tissue samples, organizing the archived fish tissue samples by project to facilitate retrieval of the samples, and developing and maintaining an inventory of the archived samples
  - preparing summary project information and graphics for development of project fact sheets, presentations, and other EPA meeting and outreach materials
- monitoring the performance of CSC staff participating in this study to ensure that they are following all QA procedures described in this QAPP that are related to CSC tasks being performed to support this study (see list above)
- ensuring completion of high-quality deliverables within established budgets and time schedules
- participating in meetings and conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study

Marguerite Jones is the **CSC QA Officer**, whose primary responsibilities include the following:

- assisting CSC's Project Leader with the development and review of this QAPP
- approving this QAPP
- providing oversight for the implementation of QA procedures related to CSC tasks that are described in this QAPP
- reporting deviations from this QAPP to the CSC Project Leader and recommending corrective actions to resolve these deviations

Tony Olsen is the **Senior Statistician** at the ORD Western Ecology Division in Corvallis, Oregon who is supporting the NRSA fish fillet indicator by providing technical expertise for

study planning and implementation. He is providing direct technical support for the following activities or providing leadership and oversight for ORD staff supporting these activities:

- developing the study design for the NRSA
- selecting probability-based sites and tracking each site for final statistical classification
- completing statistical analysis of analytical data for NRSA fillet tissue samples
- developing cumulative distribution functions for analytical data sets with sufficient data points
- participating in development of final reports for publication

A5. Problem Definition/Background

Obtaining statistically representative occurrence data on multiple contaminants in fish tissue is a priority area of interest for EPA. Since 1998, OW has collaborated with ORD to conduct a series of national- and regional-scale assessments of contaminants in fish tissue through statistically based studies of U.S. lakes and rivers. These EPA studies are referred to as the National Lake Fish Tissue Study, the 2008-2009 NRSA, and the Great Lakes Human Health Fish Tissue Study conducted under the 2010 National Coastal Condition Assessment. Including the fish fillet indicator in the 2013-2014 NRSA is providing the first opportunity for analysis of probability-based national fish contamination trends in U.S. rivers. Results from the 2013-2014 NRSA fish fillet indicator will be compared to corresponding results from the 2008-2009 fish fillet indicator (which generated a national baseline for fish contamination data in U.S. rivers) to examine temporal trends.

A6. Project/Task Description

OST is collaborating with OWOW and with ORD-WED in Corvallis, Oregon, to plan and implement the fish fillet indicator within the framework of the 2013-2014 NRSA. Fish composite samples will be collected during May through September of 2013 and 2014 at a statistical subset of approximately 450 sites in the NRSA framework (Figure 2).

Following are the key design components for the 2013-2014 NRSA fish fillet indicator:

- Sampling approximately 450 randomly selected sites during 2013 and 2014 (see Appendix A).
- Collecting one fish composite sample for human health applications (i.e., five similarly sized adult fish of the same species that are commonly consumed by humans) from each site.
- Shipping whole fish samples to a commercial laboratory for storage and fish sample preparation, which includes collection of tissue plug samples for mercury analysis, before filleting the fish, removing both fillets from each fish, homogenizing the fillet tissue composites, and preparing fillet tissue aliquots for analysis of mercury, perfluorinated compounds (PFCs), PCBs, PBDEs, and lipids.

- Analyzing the fillet tissue samples for mercury (total) and 13 PFCs, including perfluorooctane sulfonate (PFOS) (details to be addressed in a subsequent revision of this QAPP).
- Preparing and storing fillet tissue aliquots for potential future PCB and PBDE analyses.

Figure 2. NRSA fish fillet indicator sampling locations

Microbac will store the 2013-2014 NRSA fillet tissue samples and prepare the fish tissue samples for analysis as outlined in the third bullet above. Microbac staff are also preparing multiple aliquots of archived fillet tissue from each fish sample and storing them in a freezer to allow for future analysis of these samples for other contaminants, particularly PCBs and PBDEs.

A7. Quality Objectives and Criteria

The overall quality objective for the preparation of the 2013-2014 NRSA fish fillet indicator samples is to obtain a complete set of samples for each chemical or chemical group of interest to OST. Completeness is defined as the percentage of samples collected in the study for which usable sample aliquots are produced. The goal for completeness is 95%, which recognizes that a few samples sent from the field may not arrive in acceptable condition for inclusion in the study.

A8. Special Training/Certification

All laboratory staff involved in the preparation of fish tissue samples must be proficient in the associated tasks, as required by the National Rivers and Streams Assessment 2013 Tissue Preparation, Homogenization, and Distribution Procedures (Appendix B).

Specialized training is being provided for laboratory technicians who will be collecting fish tissue plug samples (for mercury analysis only) and preparing fillet tissue samples for this project. This training is being conducted jointly by OST, CSC, and Tetra Tech at Microbac for all laboratory staff involved with 2013-2014 NRSA fillet tissue sample preparation to accomplish the following objectives:

- Present NRSA fillet tissue preparation, homogenization and distribution procedures described in Appendix B,
- Demonstrate mercury plug sample collection techniques with fish from invalid NRSA samples,
- Demonstrate filleting and homogenizing techniques with fish from invalid NRSA samples,
- Provide hands-on opportunities for fish preparation laboratory staff to develop proficiency with plug sample extraction and with filleting and homogenizing fish samples, including equipment cleaning procedures and production of equipment rinsate samples.

A9. Documents and Records

The statement of work (SOW) issued by CSC to Microbac for the fillet tissue preparation subcontract provides the specific requirements for the preparation laboratory deliverables. That SOW is the basis for Appendix B to this QAPP. The major deliverables requirements are summarized below:

- The sample preparation laboratory must prepare and submit a weekly progress report to CSC to document the status of fish sample preparation activities and provide information specified in the SOW.
- The laboratory must report the results of the rinsate analyses for mercury, PCBs, and PBDEs, and the triplicate lipid results associated with the sample batch to CSC.
- The laboratory must provide shipping information (airbills, shipping forms, etc.) to CSC for tissue or rinsate samples sent from the laboratory.

The laboratory will maintain records and documentation associated with these efforts for a minimum of five years after completion of the study. Additional copies will be maintained by CSC for at least five years and will be transferred to EPA on request.

B. DATA GENERATION AND ACQUISITION

B1. Sampling Process Design (Experimental Design)

The objective of the 2013-2014 NRSA fish fillet indicator is to investigate the occurrence of mercury, PFCs, and other contaminants in the edible tissue (fillets) of harvestable-sized adult freshwater fish that are typically consumed by humans. This fish contamination study will provide statistically representative data on the concentrations of mercury and PFCs in fish from

the nation's rivers that are applicable to human health. Fish tissue data from this study will also provide EPA with the first opportunity to analyze trends in the levels of river fish contamination by comparing 2013-2014 NRSA fillet tissue results to the fillet tissue data generated during the 2008-2009 NRSA. The 2013-2014 NRSA fish fillet indicator may also include future analysis of tissue samples for PCBs and PBDEs. In this event, the description of the 2013-2014 NRSA fish fillet indicator objective will be expanded to add these chemical groups.

The details of the sampling process design, sampling methods, and sample handling and custody procedures are described in EPA's *National Rivers and Streams Assessment 2014-2014 Quality Assurance Project Plan, May 2013*, prepared by OWOW with input by other study participants (USEPA 2013a). However, to provide some context for the readers of this QAPP, those aspects of the NRSA are summarized below.

The target population for the 2013-14 NRSA consists of all streams and rivers within the 48 contiguous states that have flowing water from April through September, excluding portions of tidal rivers up to the head of salt. This target population applies to all the NRSA core indicators (i.e., in situ measurements, water chemistry, chlorophyll a, periphyton, benthic macroinvertebrate assemblage, fish assemblage, and physical habitat). The 2013-2014 NRSA survey design incorporates two major components: the NRSA14 survey design and the NRSA09 survey design. These design components address both NRSA objectives of estimating current status and estimating change in status for all flowing waters. The NRSA09 survey design is a subsample of 2008-2009 NRSA target river sites (Strahler order 5^{th} and greater) and stream sites (Strahler order 1^{st} through 4^{th}) sampled in 2008 and 2009. The NRSA14 survey design is a new survey design that involved selection of new sites in the following four river and stream categories: major rivers (Strahler order 5^{th} and greater), other rivers (Strahler order 5^{th} and greater), large streams (Strahler order 3^{rd} and 4^{th}), and small streams (Strahler order 1^{st} and 2^{nd}). Both the NRSA09 and NRSA14 designs are explicitly stratified by state. Within each state, the unequal probability of selection was based on river and stream categories and ecological reporting sites. Application of both the NRSA09 survey design and the NRSA14 survey design resulted in selection of 1808 base sampling sites for the 2013-2014 NRSA, which are distributed among the six river and stream categories as follows: 409 previously sampled rivers, 402 previously sampled streams, 227 new major rivers, 236 new other rivers, 256 new large streams, and 278 new small streams.

Note: The terms "NRSA09" and "NRSA14" above were developed by ORD-WED to specifically identify components of the statistical design for the 2013-2014 NRSA.

Table 1. Site Selection Summary for the 2013-2014 NRSA Survey Design

Ecological Reporting Region	NRSA09 Design			NRSA14 Design					Design Total
	Rivers	Streams	Totals	Rivers Major	Rivers Other	Large Streams	Small Streams	Total	
Coastal Plain (CPL)	52	48	100	29	33	34	42	138	238
Northern Appalachians (NAP)	52	41	93	30	34	39	44	147	240
Northern Plains (NPL)	43	39	82	16	17	17	25	75	157
Southern Appalachians (SAP)	52	60	112	29	30	31	38	128	240
Southern Plains (SPL)	41	34	75	20	21	20	20	81	156
Temperate Plains (TPL)	44	49	93	28	27	30	28	113	206
Upper Midwest (UMW)	39	40	79	19	20	20	18	77	156
Western Mountains (WMT)	43	61	104	29	26	32	32	119	223
Xeric Region (XER)	43	30	73	27	28	33	31	119	192
Total	409	402	811	227	236	256	278	997	1808

The target population for the 2013-2014 NRSA fish fillet indicator (a supplemental indicator) consists of all Strahler order 5th and greater streams (which are categorized as rivers) within the 48 contiguous states that have flowing water from April through September, excluding portions of tidal rivers up to the head of salt. A statistically representative subset of 453 river sites distributed throughout the 48 states was designated as the group of sampling sites for the 2013-2014 NRSA fish fillet indicator. To optimize the capability for estimating change in fish contaminant levels, the 2013-2014 NRSA fish fillet indicator sampling sites include the 409 river locations previously sampled during the 2008-2009 NRSA and 44 of the new major river sites from the NRSA14 design (one new major river site in each of the 44 states where new major river sites were selected).

To meet the study objective, one fish composite sample was collected from each site. A routine fish composite sample consists of five adult fish that are selected for each composite based on the following criteria:

- All are of the same species;
- All satisfy legal requirements of harvestable size (or weight) for the sampled site, or at least be of consumable size if new legal harvest requirements are in effect;

- All are of similar size, so that the smallest individual in a composite is no less than 75% of the total length of the largest individual;
- All are collected at the same time, i.e., collected as close to the same time as possible, but no more than one week apart. (Note: Individual fish may have to be frozen until all fish to be included in the composite are available for delivery to the designated laboratory.)

Accurate taxonomic identification is essential in preventing the mixing of closely related target species. Under no circumstances are individuals from different species used in a composite sample.

The sample collection goal at each NRSA fish fillet indicator site is to obtain a composite sample of fish that are adequate in size to provide a minimum of 536 grams of fillet tissue for chemical analysis. Field crews will collect fish between May and September during both field seasons in 2013 and 2014.

B2. Sampling Methods

Sampling method procedures and requirements for collection of human health fish samples are detailed in EPA's *National Rivers and Streams Assessment 2013-2014 Quality Assurance Project Plan, May 2013* (USEPA 2013a) and *National Rivers and Streams Assessment Field Operations Manual* (USEPA 2013b). These sampling procedures and requirements are summarized below.

The field objective is for sampling teams to obtain one representative fish composite sample from each sampling site. Collecting fish composite samples is a cost-effective means of estimating average chemical concentrations in the tissue of target species, and compositing fish ensures adequate sample mass for analysis of multiple chemicals. The sampling procedures specify that each composite should consist of five similarly sized adult fish of the same species. OST developed a recommended fish species list with OWOW concurrence that contained 18 priority target fish species. In June 2013, OST expanded this list to include 16 alternative fish species. Sampling teams will use this list as the basis for selecting appropriate fish species for the NRSA fish fillet indicator samples. The method applied for fish collection is at the discretion of the field team, but electrofishing is preferred.

In preparing fish samples for shipping, field teams record sample number, species name, specimen length, sampling location and sampling data and time on a fish collection form. Each fish is wrapped in solvent-rinsed, oven-baked aluminum foil, with the dull side in using foil sheets provided by EPA. Individual foil-wrapped specimens are placed into a length of food-grade polyethylene tubing, each end of the tubing is sealed with a plastic cable tie, and a fish specimen label is affixed to the outside of the food-grade tubing with clear tape. All of the wrapped fish in the sample from each site are placed in a large plastic bag and sealed with another cable tie, then placed immediately on dry ice for shipment to Microbac in Baltimore, Maryland. Field crews are directed to pack fish samples on dry ice in sufficient quantities to keep samples frozen for up to 48 hours (50 pounds are recommended), and to ship them via priority overnight delivery service (e.g., Federal Express), so that they arrive at Microbac in less than 24 hours from the time of sample collection. Alternatively, field crews may transport whole fish samples on wet or dry ice (depending on the distance) to an interim facility where the fish

samples are frozen and stored for up to two weeks before overnight shipping to Microbac on dry ice as described above.

B3. Sample Handling and Custody

This section describes the sample handling and custody procedures that apply once the whole fish tissue samples are shipped from the field to the sample preparation laboratory. Fish samples for the 2013-2014 NRSA are being collected by various organizations participating with EPA in this study, including state and tribal agencies, other federal agencies, and contractors. Although samples will be shipped frozen on dry ice, they must be inspected promptly on receipt. As samples are received, the sample custodian at the sample preparation laboratory will:

- Check that each shipping container has arrived undamaged and verify that samples are still frozen and in good condition.
- Check the temperature of one of the samples in the cooler using a thermometer that reads to at least -20 degrees Celsius (ºC), or an infra-red (IR) temperature "gun" and record the reading on the sample tracking form.
- Verify that all associated paperwork is complete, legible, and accurate.
- Compare the information on the label on each individual fish specimen to the sample tracking form for each composite and verify that each specimen was included in the shipment and is properly wrapped and labeled.
- Notify CSC of the fact that samples were received and of any discrepancies in the paperwork identified above.
- Provide CSC with a copy of the sample tracking form for each sample (via email). (CSC will check that the samples were collected from sites on the list of valid whole fish tissue sampling locations (uniquely designated by the site identification number), and notify EPA immediately if samples have been received from sites not on that list.)
- Transfer the samples to the freezer for long-term storage.

The sample preparation laboratory will notify CSC immediately about any problems encountered upon receipt of samples. Problems involving sample integrity, conformity, or inconsistencies for fish tissue samples should be reported to CSC in writing (e.g., by email) as soon as possible following sample receipt and inspection.

Following sample processing, the sample preparation laboratory must store sample aliquots frozen to less than or equal -20 °C until they are distributed to the laboratories performing analyses under separate CSC purchase orders. (The freezers are maintained by the sample processing laboratory under a separate agreement with CSC and are continuously monitored by an automated temperature monitoring system.)

B4. Fish Sample Preparation Methods

Microbac has been selected as the fish sample preparation laboratory (prep lab) for the NRSA fish fillet indicator. In this role, Microbac is responsible for extracting a fish plug sample from designated fish in the sample before filleting the fish, filleting each valid fish sample, homogenizing the fillet tissue, preparing the required number of fish tissue aliquots for analysis and archive, shipping the fish tissue aliquots for each analysis to the designated analytical laboratory, and storing archived fish tissue samples in a freezer at their facility. The specific procedures for NRSA fillet tissue sample preparation activities are described in Appendix B.

Before beginning sample processing, trained lab technicians complete a relative fish length comparison to confirm that field crews attached the correct label to each fish in the composite sample. Each fish is then weighed to the nearest gram, rinsed with deionized water, placed on a clean glass cutting board, and scaled. Prior to filleting each fish in the sample, the lab technicians extract fish plug samples from designated fish (typically two fish per composite sample). The filleting process involves removing the fillet (with skin on and belly flap [ventral muscle] attached) from both sides of each fish. Fillets are composited using the "batch" method, in which all of the fillets from the individual specimens that make up the sample are homogenized together, regardless of each specimen's proportion to one another (as opposed to the "individual" method, in which equal weights of tissue from each specimen are added together).

An electric meat grinder is used to prepare homogenate samples. Entire fillets (with skin and belly flap) from both sides of each fish are homogenized, and the entire homogenized volume of all fillets from the fish sample is used to prepare the tissue sample. Tissues are mixed thoroughly until they are completely homogenized as evidenced by a fillet homogenate that consists of a fine paste of uniform color and texture. Homogeneity is confirmed by conducting triplicate analyses of the lipid content in one of every twenty samples. The collective weight of the homogenized tissue from each sample is recorded to the nearest gram (wet weight) after processing. Microbac prepares fillet tissue samples and sample aliquots according to the specifications listed in Steps 18 to 28 of the fish sample preparation procedures in Appendix B.

B5. Fish Sample Preparation Quality Control

The project-specific QC procedures include preparation and testing of equipment rinsate samples and homogeneity testing, using lipids as a surrogate. The QC procedures are performed in two distinct phases: (1) as part of an initial demonstration of capabilities after the kickoff meeting and training workshop with EPA, and (2) during normal operations (i.e., NRSA fish sample preparation procedures).

B.5.1 Initial demonstration of capabilities

After the kickoff meeting and training workshop, Microbac staff will prepare three test fish samples provided by Tetra Tech. Each test sample will consist of a single large fish that will be processed separately. Each of these test samples will be carried through the entire sample preparation and aliquoting procedures separately. The resulting sample aliquots will not be distributed to other laboratories. In between processing each individual fish sample, Microbac

staff will clean all of the sample preparation equipment as described in Step 29 of Appendix B. After each cleaning, Microbac staff will prepare the entire series of equipment rinsates and solvent blanks described in Step 32 of Appendix B.

Microbac also will collect three lipid aliquots from each sample prepared during the initial demonstration and use them for triplicate determinations of lipids, as described in Step 36 of Appendix B. Microbac will analyze the rinsate samples for mercury, PBDE congeners, and PCB congeners using the procedures described in Table 2, or have them analyzed by a subcontract laboratory under their control, as shown below.

Table 2. Methods for Determination of Lipids and Analyses of Rinsate Samples

Parameter	Method	Laboratory
Lipids	SW-846 9071B	Microbac
Mercury	EPA 245.1	Microbac
PBDEs	EPA 1614	Vista Analytical (under subcontract to Microbac)
PCBs	EPA 1668A	Cape Fear (under subcontract to Microbac)

The results of the analyses of the rinsates and the homogeneity testing (three sets each) will be submitted to CSC for review. Microbac will not begin 2013-2014 NRSA sample preparation until CSC and EPA determine that the sample preparation laboratory has successfully demonstrated proficiency in meeting QC requirements for equipment cleaning and tissue homogenization.

From the lipid results, Microbac will calculate the mean lipid content (in percent), the standard deviation (SD), and the relative standard deviation (RSD) using the formulae below, or the corresponding functions in Excel.

$$\text{mean } \%\text{lipids} = \frac{\sum_{i=1}^{3} (\%\text{lipids})_i}{3}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{3} (\%\text{lipids}_i - \text{mean lipids})^2}{2}}$$

$$RSD = \frac{SD}{\text{mean}}$$

If the RSD of the triplicate results is less than or equal to 15%, then CSC and EPA will judge the homogenization effort to be sufficient for all samples in that preparation batch.

If the results for the rinsate samples are below the limits in Table 3 for mercury, PBDEs, and PCBs, then CSC and EPA will judge the equipment cleaning effort to be sufficient for all samples in that preparation batch.

Table 3. Acceptance Limits for Rinsate Samples

Parameter	Acceptance Limit	Basis for Limit
Mercury	1 µg/L for total mercury	Method detection limit for an aqueous sample
PBDEs	0.5 ng/mL per congener	Instrument detection limit for a 0.5-mL final volume of solvent concentrated from the original 50-mL rinsate sample
PCBs	0.5 ng/mL per congener	Instrument detection limit for a 0.5-mL final volume of solvent concentrated from the original 50-mL rinsate sample

B.5.2 Normal Operations

During normal sample preparation efforts, the Microbac will prepare one set of rinsate samples and will conduct one set of triplicate lipid determinations per batch of 20 composite fish samples, as described in Steps 32 to 37 of Appendix B. The batch-specific rinsate and homogeneity results will be reviewed by CSC and EPA against the same QC specifications used for the initial demonstration of capabilities. The sample preparation laboratory may continue to process up to one additional batch of 20 samples (based on sample preparation instructions provided by CSC) during that review process. However, the sample preparation laboratory may not continue beyond that next batch of samples until receiving notification from CSC that review of the prior batch rinsate and homogeneity test results is complete and the results were deemed satisfactory.

B6. Instrument/Equipment Testing, Inspection, and Maintenance

There are no analytical instruments used in the preparation of the fillet tissue samples. However, the balances used to weigh the whole fish and the tissue sample aliquots are inspected and serviced on a regular schedule and the homogenization equipment (meat grinder) will be inspected when it is reassembled after cleaning between samples.

All analytical instrumentation associated with the rinsate analyses will be inspected and maintained as described in the respective analysis methods and laboratory SOPs.

B7. Instrument/Equipment Calibration and Frequency

The balances used to weigh the whole fish and the tissue sample aliquots are calibrated on a regular schedule and calibrations are verified at the beginning of each day on which the balances are used.

All analytical instrumentation associated with the rinsate analyses will be calibrated as described in the respective analysis methods. The methods in Table 3 all require multi-point initial calibrations and periodic calibration verifications, and all the methods contain QC acceptance criteria for calibration.

B8. Inspection/Acceptance of Supplies and Consumables

The inspection and acceptance of any laboratory supplies and consumables associated with the rinsate analyses are addressed in the individual laboratory operating procedures to be used, and/or in the laboratory's existing overall quality system documentation. There are no additional requirements specific to this project, and therefore, none are described here.

B9. Non-direct Measurements

Non-direct measurements are not required for this project. (The analytical results from the 2008-2009 NRSA to which any new data are to be compared are primary data that EPA generated under an approved QAPP for that study.)

B10. Data Management

Data management practices employed in this study will be based on standard data management practices used for EPA's National Lake Fish Tissue Study and other OST fish contamination studies (e.g. Great Lakes Human Health Fish Tissue Study). The data management (i.e., sample tracking, data tracking, data inspection, data quality assessment, database development) procedures have been regularly applied to other technical studies by CSC. These procedures are being employed because they are effective, efficient, and have successfully withstood repeated internal and external audits, including internal review by EPA Quality Staff, public review and comment, judicial challenge, and an audit by the Government Accountability Office. These procedures, as implemented for the NRSA fish fillet indicator, are summarized below.

- Microbac is required to maintain all records and documentation associated with the preparation of study samples and rinsates for a minimum period of five years after completion of the study.
- All required reports and documentation, including raw data, must be sequentially paginated and clearly labeled with the laboratory name, and associated sample numbers. Any electronic media submitted must be similarly labeled.
- Microbac will adhere to a comprehensive data management plan that is consistent with the principles set forth in Good Automated Laboratory Practices, EPA Office of Administration and Resources Management (USEPA 1995). Microbac's data management plan is incorporated in their overall quality system documentation, e.g., their quality management plan, a copy of which will be maintained on file at CSC.

C. ASSESSMENT AND OVERSIGHT

C1. Assessments and Response Actions

The laboratory contract prepared to support this study stipulates that the sample preparation laboratory has a comprehensive QA program in place and operating at all times during the performance of their contract, and that in performing laboratory work for this study, the laboratory shall adhere to the requirements of that QA program (Microbac 2012). A copy of that plan will be maintained on file at CSC.

Sections C1.1 through C1.5 describe other types of assessment activities and corresponding response actions identified to ensure that data gathering activities in the NRSA fish fillet indicator are conducted as prescribed and that the performance criteria defined for the study are met.

C1.1 Surveillance

The CSC Project Leader will schedule and track all work performed by the sample preparation laboratory. The Project Leader will coordinate with staff at Microbac regarding fish tissue sample shipments to other laboratories once analysis contracts are funded and in place.

When CSC is advised that samples are being shipped from the field to the sample preparation laboratory, the Project Leader will contact designated sample preparation laboratory staff by email to notify them of the forthcoming shipment(s) and request that they contact CSC if the shipments do not arrive intact as scheduled. Within 24 hours of scheduled sample receipt, CSC will contact the laboratory to verify that the samples arrived in good condition, and if problems are noted, will work with the laboratory and EPA to resolve the problem as quickly as possible to minimize data integrity problems.

CSC's project leader will obtain fish sample processing instructions for each batch of 20 samples from the OST Project Manager and transmit those instructions to the sample processing laboratory by email. The sample preparation laboratory may not begin processing any samples until this QAPP is approved and CSC provides the sample processing instructions.

CSC will communicate periodically with laboratory staff by telephone or email to monitor the progress of sample preparation and lipid and rinsate analysis. If technical problems are encountered during sample preparation and rinsate analysis, CSC will identify a technical expert within CSC to assist in resolving the problem, and work with EPA to identify and implement a solution to the problem. The sample preparation laboratory will be permitted to work one batch ahead of the production and CSC/EPA review of the lipid and rinsate analyses to ensure that the homogenization and equipment cleaning procedures are adequate.

If the laboratory fails to deliver QC data on time, or if the laboratory notifies CSC of anticipated reporting or sample processing delays, CSC will notify the OST Project Manager of the situation. To the extent possible, CSC will adjust schedules and shift resources within CSC as necessary to minimize the impact of laboratory delays on EPA schedules. CSC also will immediately notify the Project Manager of any laboratory delays that are anticipated to impact EPA schedules.

C1.2 Product Review

Reviews of the sample preparation records and the results of the lipid homogeneity and rinsate testing will be performed by CSC. The results of those reviews will be documented in emails to the OST Project Manager.

C1.3 Quality Systems Audit

A quality system audit (QSA) is used to verify, by examination and evaluations of objective evidence, that applicable elements of the quality system are appropriate and have been developed, documented, and effectively implemented in accordance and in conjunction with specified requirements. The focus of these assessments is on the quality system processes – not on evaluating the quality of specific products or judging the quality of environmental data or the

performance of personnel or programs. The SHPD QA Coordinator may perform a QSA of the fillet tissue preparation portion of the 2013-2014 NRSA.

C1.4 Readiness Review

A readiness review of the sample preparation laboratory's capability to produce homogeneous tissue sample aliquots will begin with the kick-off meeting with the laboratory. This effort will include the initial demonstration of capabilities described in Appendix B. Routine processing of fish tissue samples will not begin until the laboratory has demonstrated acceptable performance in the initial demonstration of capabilities.

The results of the lipid homogeneity testing and rinsate analyses from the initial demonstration of capabilities will be examined by CSC data reviewers to determine if the laboratory met the QC acceptance criteria for the lipid homogeneity testing and the rinsate analyses. If problems are identified during these reviews, CSC will work with the laboratory, to the extent possible, to resolve the problem. If the problem cannot be resolved within the time frame required by EPA or within the scope of the laboratory's existing contract, CSC will notify the OST Project Manager immediately. Records of these reviews and any corrective actions are maintained by CSC. CSC staff will document their findings and recommendations concerning the readiness review as part of a written analytical QA report to EPA.

C1.5 Technical Systems Audit

The laboratory contract requires that the laboratory be prepared for and willing to undergo an on-site, or technical systems, audit of its facilities, equipment, staff, sample processing and rinsate analysis, training, record keeping, data validation, data management, and data reporting procedures. An audit will be conducted only if the results of the readiness reviews, data quality audits, and surveillance suggest serious or chronic laboratory problems that warrant on-site examinations and discussion with laboratory personnel.

If such an audit is determined to be necessary, a standardized audit checklist may be used to facilitate an audit walkthrough and document audit findings. Audit participants may include the OST Project Manager and/or the SHPD QA Coordinator (or a qualified EPA staff member designated by the OST QA Officer) and a CSC staff member experienced in conducting laboratory audits. One audit team member will be responsible for leading the audit and conducting a post-audit debriefing to convey significant findings to laboratory staff at the conclusion of the audit. Another audit team member will be responsible for gathering pre-audit documentation of problems that necessitated the audit, customizing the audit checklist as necessary to ensure that those problems are addressed during the audit, documenting audit findings on the audit checklist during the audit, and drafting a formal report of audit findings for review by EPA.

C2. Reports to Management

The sample preparation laboratory will provide CSC with a weekly status report that describes all of the fish samples processed during the previous week. CSC will review those reports for completeness and then forward them to the OST Project Manager.

D. DATA VALIDATION AND USABILITY

D1. Data Review, Verification, and Validation

The data review, verification, and validation aspects of the fillet tissue sample preparation effort are more limited than those that might be applied to typical chemical analysis efforts. The procedures described below apply primarily to the results of the lipid homogeneity and rinsate analyses.

D1.1 Data Review

All laboratory results and calculations will be reviewed by the Laboratory Manager prior to data submission. Any errors identified during this peer review will be returned to the analyst for correction prior to submission of the data package. Following correction of the errors, the Laboratory Manager will verify that the final package is complete and compliant with the contract, and will sign each data submission to certify that the package was reviewed and determined to be in compliance with the terms and conditions of the contract.

D1.2 Data Verification

The basic goal of data verification is to ensure that project participants know what data were produced, if they are complete, if they are contractually compliant, and the extent to which they meet the objectives of the study.

CSC staff will conduct reviews of the QC sample results for homogenized fish tissue samples prepared by Microbac. This will involve review of data for percent lipid measurements that serve as a surrogate for homogeneity testing and review of the results from rinsates of the sample processing equipment. The CSC Project Leader will verify the summary level results for these QC samples, determine if they meet the project objectives in this QAPP, and report the verification findings to OST.

D1.3 Data Validation

Data validation is the process of evaluating the quality of the results relative to their intended use. Data need not be “perfect” to be usable for a particular project, and the validation process is designed to identify data quality issues uncovered during the verification process that may affect the intended use. One goal of validation is to answer the “So what?” question with regard to any data quality issues.

As noted above, the data validation aspects of the fillet tissue sample preparation effort are more limited and will focus on the clarity and accuracy of the weekly sample processing reports.

D2. Verification and Validation Methods

D2.1 Verification Methods

In the first stage of the data verification process, CSC reviewers will perform a "Completeness Check" in which all elements in each laboratory submission will be evaluated to verify that results for all specified samples are provided, that data are reported in the correct format, and that all relevant information, such as preparation and analysis logs, are included in the data package. Corrective action procedures will be initiated if deficiencies are noted.

The second stage of the verification process will focus on an "Instrument Performance Check" in which the CSC data review chemists will verify that calibrations, calibration verifications, standards, and calibration blanks were analyzed at the appropriate frequency and met method or study performance specifications. If errors are noted at this stage, corrective action procedures will be initiated immediately.

Stage three of the verification process will focus on a "Laboratory Performance Check" in which CSC data review chemists will verify that the laboratory correctly performed the required analytical procedures and was able to demonstrate a high level of precision and accuracy. This stage includes evaluation of QC elements such as the laboratory control samples, method blanks, matrix spike samples and/or reference samples, where applicable. Corrective action procedures will be initiated with the laboratories to resolve any deficiencies identified.

D2.2 Validation Methods

CSC data review chemists will perform a data quality and usability assessment in which the overall quality of data is evaluated against the performance criteria. This assessment will strive to maximize use of data gathered in this study based on performance criteria established for this study. This will be accomplished by evaluating the overall quality of a particular data set rather than focusing on individual QC failures. Results of this assessment will be documented in a report after all of the results have been evaluated and forwarded to the OST Project Manager.

D3. Reconciliation with User Requirements

The QC results for lipids from the homogeneity testing and the rinsate analysis for each batch of fish tissue samples prepared will be assessed against the QC acceptance criteria. Although the sample preparation laboratory will be permitted to work "one batch ahead" of the delivery of the batch-specific QC results, CSC will track laboratory performance, notify the OST Project Manager of any issues, initiate corrective actions, and track progress by the sample preparation laboratory.

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Appendix A

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
AL	ALR9-0901	FW08AL012	34.95092	-87.04203	7	Elk River	Non-Urban
AL	ALR9-0902	FW08AL014	32.48594	-85.72031	5	Uphapee Creek	Non-Urban
AL	ALR9-0903	FW08AL015	31.08686	-87.08175	5	Murder Creek	Non-Urban
AL	ALR9-0904	FW08AL020	33.41281	-86.75190	5	Cahaba River	Urban
AL	ALR9-0905	FW08AL021	31.34381	-85.60881	5	Choctawhatchee River	Non-Urban
AL	ALR9-0906	FW08AL022	31.68740	-88.05213	5	Salitpa Creek	Non-Urban
AL	ALRM-1001		32.22869	-87.14828	7	Alabama River	Non-Urban
AR	ARR9-0901	FW08AR010	35.62598	-89.87933	10	Mississippi River	Non-Urban
AR	ARR9-0902	FW08AR012	34.35270	-91.10542	8	White River	Non-Urban
AR	ARR9-0903	FW08AR014	34.69500	-90.64588	7	Saint Francis River	Non-Urban
AR	ARR9-0904	FW08AR016	35.67817	-93.74409	5	Mulberry River	Non-Urban
AR	ARR9-0905	FW08AR017	33.55630	-92.02261	6	Saline River	Non-Urban
AR	ARR9-0906	FW08AR019	34.92888	-93.36068	5	Fourche Lafave River	Non-Urban
AR	ARR9-0907	FW08AR022	34.80142	-90.77003	5	Anguile River	Non-Urban
AR	ARR9-0908	FW08AR024	33.61703	-93.86001	6	Little River	Non-Urban
AR	ARR9-0909	FW08AR026	35.53264	-90.44201	6	Saint Francis River	Non-Urban
AR	ARRM-1001		35.60184	-89.90212	10	Mississippi River	Non-Urban
AZ	AZR9-0901	FW08AZ009	36.08766	-111.87061	8	Colorado River	Non-Urban
AZ	AZR9-0902	FW08AZ013	36.43346	-111.86409	8	Colorado River	Non-Urban
AZ	AZR9-0903	FW08AZ019	32.40882	-111.16063	6	Santa Cruz River	Non-Urban
AZ	AZR9-0904	FW08AZ022	33.29395	-109.49470	5	Eagle Creek	Non-Urban
AZ	AZR9-0913	FW08AZ062	33.47634	-114.60530	9	Colorado River	Non-Urban
AZ	AZRM-1001		33.66840	-114.53128	9	Colorado River	Non-Urban
CA	CAR9-0901	FW08CA020	41.31853	-123.52796	6	Klamath River	Non-Urban
CA	CAR9-0902	FW08CA022	34.35671	-119.01988	6	Santa Clara River	Urban
CA	CAR9-0903	FW08CA031	38.81627	-123.01119	5	Russian River	Urban
CA	CAR9-0904	FW08CA035	38.80836	-121.63521	6	Feather River	Non-Urban
CA	CAR9-0905	FW08CA038	34.42494	-118.55705	5	Santa Clara River	Urban
CA	CAR9-0906	FW08CA040	41.53926	-123.52673	6	Klamath River	Non-Urban
CA	CAR9-0907	FW08CA044	41.48963	-120.60459	6	Pit River	Non-Urban
CA	CAR9-0908	FW08CA056	41.45640	-123.93556	7	Klamath River	Non-Urban
CA	CAR9-0909	FW08CA059	40.00191	-121.26823	6	Feather River	Non-Urban
CA	CAR9-0910	FW08CA061	37.59638	-121.12876	6	Tuolumne River	Non-Urban
CA	CAR9-0911	FW08CA063	40.31844	-123.77101	7	Eel River	Non-Urban
CA	CAR9-0912	FW08CA067	38.57234	-121.35775	6	American River	Urban
CA	CAR9-0913	FW08CA068	41.84331	-122.89973	6	Klamath River	Non-Urban
CA	CARM-1001		33.85326	-117.78304	5	Santa Ana River	Urban
CO	COR9-0901	FW08CO021	39.98812	-108.77796	5	Douglas Creek	Non-Urban
CO	COR9-0902	FW08CO025	38.86531	-108.39814	6	Gunnison River	Non-Urban
CO	COR9-0903	FW08CO028	37.60462	-103.60597	5	Purgatoire River	Non-Urban
CO	COR9-0904	FW08CO032	40.94132	-102.34142	5	South Platte River	Non-Urban
CO	COR9-0905	FW08CO033	37.17608	-105.73105	6	Grande, Rio	Non-Urban
CO	COR9-0906	FW08CO036	40.39451	-103.47733	7	South Platte River	Non-Urban
CO	COR9-0907	FW08CO037	40.47797	-108.90822	7	Yampa River	Non-Urban
CO	COR9-0908	FW08CO046	39.65513	-107.06715	6	Colorado River	Non-Urban
CO	CORM-1001		39.18629	-108.90477	7	Colorado River	Non-Urban
CT	CTR9-0901	FW08CT005	41.89123	-72.66210	5	Farmington River	Urban
CT	CTR9-0902	FW08CT006	41.78270	-71.89588	5	Quinebaug River	Urban
CT	CTR9-0903	FW08CT007	41.54059	-72.55126	6	Connecticut River	Urban
CT	CTR9-0906	FW08CT016	41.84448	-72.63200	5	Farmington River	Urban
CT	CTRM-1001		41.48485	-72.50888	6	Connecticut River	Urban
DE	DER9-0901	FW08DE005	39.70013	-75.63339	5	White Clay Creek	Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
DE	DER9-0902	FW08DE009	39.83430	-75.57709	5	Brandywine Creek	Urban
DE	DER9-0903	FW08DE010	38.61817	-75.63092	5	Nanticoke River	Urban
FL	FLR9-0901	FW08FL005	30.35243	-84.68592	5	Ochlockonee River	Non-Urban
FL	FLR9-0902	FW08FL006	29.98459	-85.03299	8	Apalachicola River	Non-Urban
FL	FLR9-0903	FW08FL007	27.41502	-81.13117	5	Kissimmee River	Non-Urban
FL	FLRM-1001		30.21423	-85.11154	8	Apalachicola River	Non-Urban
GA	GAR9-0901	FW08GA006	30.70227	-83.03386	6	Alapaha River	Non-Urban
GA	GAR9-0902	FW08GA008	32.30967	-84.05752	5	Buck Creek	Non-Urban
GA	GAR9-0903	FW08GA009	33.05392	-81.82509	5	Brier Creek	Non-Urban
GA	GAR9-0904	FW08GA010	30.81591	-83.01665	6	Alapaha River	Non-Urban
GA	GAR9-0905	FW08GA012	32.14304	-83.38112	6	Ocmulgee River	Non-Urban
GA	GAR9-0906	FW08GA018	32.00825	-83.29546	6	Ocmulgee River	Non-Urban
GA	GAR9-0907	FW08GA020	31.15899	-85.07891	7	Chattahoochee River	Non-Urban
GA	GARM-1001		32.61633	-83.54926	6	Ocmulgee River	Non-Urban
IA	IAR9-0901	FW08IA019	42.79185	-96.60157	7	Big Sioux River	Non-Urban
IA	IAR9-0902	FW08IA021	41.00446	-91.66528	6	Skunk River	Non-Urban
IA	IAR9-0903	FW08IA022	42.20200	-90.33231	8	Mississippi River	Non-Urban
IA	IAR9-0904	FW08IA024	43.45106	-94.86716	6	Des Moines River	Non-Urban
IA	IAR9-0905	FW08IA029	42.24731	-92.32474	5	Wolf Creek	Non-Urban
IA	IAR9-0906	FW08IA034	43.10975	-91.17645	8	Mississippi River	Non-Urban
IA	IAR9-0907	FW08IA035	43.26844	-96.21303	5	Little Rock River	Non-Urban
IA	IAR9-0908	FW08IA037	40.87470	-91.04809	8	Mississippi River	Non-Urban
IA	IAR9-0909	FW08IA038	42.13068	-90.35650	6	Maquoketa River	Non-Urban
IA	IAR9-0914	FW08IA047	41.46760	-95.90931	5	Boyer River	Non-Urban
IA	IARM-1001		42.50707	-90.64463	8	Mississippi River	Urban
ID	IDR9-0901	FW08ID013	42.57566	-113.62921	7	Snake River	Non-Urban
ID	IDR9-0902	FW08ID014	46.13488	-115.95995	6	Middle Fork Clearwater River	Non-Urban
ID	IDR9-0903	FW08ID016	42.52532	-115.49077	5	Clover Creek	Non-Urban
ID	IDR9-0904	FW08ID017	45.36948	-114.28991	7	Salmon River	Non-Urban
ID	IDR9-0905	FW08ID019	47.69645	-116.91528	7	Spokane River	Urban
ID	IDR9-0906	FW08ID020	43.96347	-116.18915	6	Payette River	Non-Urban
ID	IDR9-0907	FW08ID021	44.83915	-114.78516	6	Middle Fork Salmon River	Non-Urban
ID	IDR9-0908	FW08ID023	46.66523	-115.54751	5	North Fork Clearwater River	Non-Urban
ID	IDR9-0909	FW08ID026	45.38528	-115.53329	7	Salmon River	Non-Urban
ID	IDR9-0910	FW08ID029	44.39607	-116.04608	6	North Fork Payette River	Non-Urban
ID	IDR9-0911	FW08ID032	42.66149	-114.66271	7	Snake River	Non-Urban
ID	IDR9-0912	FW08ID033	45.13323	-113.80082	6	Lemhi River	Non-Urban
ID	IDRM-1001		42.93930	-115.70144	7	Snake River	Non-Urban
IL	ILR9-0901	FW08IL009	39.20865	-90.59292	8	Illinois River	Non-Urban
IL	ILR9-0902	FW08IL011	41.48585	-89.84848	5	Green River	Non-Urban
IL	ILR9-0903	FW08IL012	37.00011	-89.26342	10	Mississippi River	Non-Urban
IL	ILR9-0904	FW08IL013	40.47669	-91.36704	8	Mississippi River	Non-Urban
IL	ILR9-0905	FW08IL017	40.78506	-90.13891	6	Fox Creek	Non-Urban
IL	ILR9-0906	FW08IL018	41.15196	-87.91418	6	Kankakee River	Urban
IL	ILR9-0907	FW08IL022	41.90002	-89.48215	7	Rock River	Urban
IL	ILR9-0908	FW08IL024	37.85242	-89.19183	5	Little Muddy River	Non-Urban
IL	ILRM-1001		42.47833	-89.05604	6	Rock River	Urban
IN	INR9-0901	FW08IN006	38.64279	-87.61438	7	Wabash River	Non-Urban
IN	INR9-0902	FW08IN008	38.83491	-86.52326	6	East Fork White River	Urban
IN	INR9-0903	FW08IN009	41.69465	-85.91740	5	Saint Joseph River	Urban
IN	INR9-0904	FW08IN010	38.45178	-87.59800	7	White River	Non-Urban
IN	INRM-1001		40.75459	-86.28108	5	Wabash River	Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
KS	KSR9-0901	FW08KS007	39.87204	-95.02724	9	Missouri River	Non-Urban
KS	KSR9-0902	FW08KS008	37.82360	-97.46279	7	Arkansas River	Non-Urban
KS	KSR9-0903	FW08KS009	38.12609	-98.07809	7	Arkansas River	Non-Urban
KS	KSR9-0904	FW08KS010	39.42728	-98.53949	5	South Fork Solomon River	Non-Urban
KS	KSR9-0905	FW08KS011	39.72624	-94.91122	9	Missouri River	Urban
KS	KSR9-0906	FW08KS015	37.39754	-95.67977	5	Fall River	Non-Urban
KS	KSR9-0907	FW08KS017	38.76631	-100.27414	6	Smoky Hill River	Non-Urban
KS	KSR9-0908	FW08KS018	39.25252	-96.32660	5	Rock Creek	Non-Urban
KS	KSR9-0909	FW08KS019	38.83876	-100.99193	5	Smoky Hill River	Non-Urban
KS	KSR9-0910	FW08KS022	39.49523	-97.23190	7	Republican River	Non-Urban
KS	KSR9-0911	FW08KS023	39.06027	-94.84195	8	Kansas River	Urban
KS	KSR9-0912	FW08KS024	37.33922	-97.25535	6	Ninnescah River	Non-Urban
KY	KYR9-0901	FW08KY013	37.78140	-88.03819	9	Ohio River	Non-Urban
KY	KYR9-0902	FW08KY014	37.62117	-83.49986	6	North Fork Kentucky River	Non-Urban
KY	KYR9-0903	FW08KY016	37.98150	-86.03399	8	Ohio River	Non-Urban
KY	KYR9-0904	FW08KY017	37.47040	-88.09642	9	Ohio River	Non-Urban
KY	KYR9-0905	FW08KY019	37.29116	-85.59289	5	Green River	Non-Urban
KY	KYR9-0906	FW08KY021	37.97943	-82.67112	6	Levisa Fork	Non-Urban
KY	KYR9-0907	FW08KY023	37.23007	-84.24396	5	Rockcastle River	Non-Urban
KY	KYRM-1001		37.33669	-87.13761	7	Green River	Urban
LA	LAR9-0901	FW08LA011	32.97480	-92.07644	7	Ouachita River	Non-Urban
LA	LAR9-0902	FW08LA013	31.62650	-92.90921	7	Red River	Non-Urban
LA	LAR9-0903	FW08LA014	32.78465	-91.95737	6	Bayou Bartholomew	Non-Urban
LA	LAR9-0904	FW08LA017	31.55119	-91.80545	7	Black River	Non-Urban
LA	LAR9-0905	FW08LA018	32.06618	-93.41412	7	Red River	Non-Urban
LA	LAR9-0906	FW08LA022	32.54806	-93.78100	6	Twelvemile Bayou	Urban
LA	LAR9-0915	FW08LA039	30.32848	-90.84382	6	Amite River	Urban
LA	LARM-1001		32.27010	-90.96074	10	Mississippi River	Non-Urban
MA	MAR9-0901	FW08MA002	41.96179	-70.91978	5	Taunton River	Non-Urban
MA	MAR9-0902	FW08MA003	42.70064	-71.21798	7	Merrimack River	Urban
MA	MAR9-0903	FW08MA005	42.57836	-72.56958	6	Connecticut River	Urban
MA	MARM-1001		42.65448	-72.46514	6	Connecticut River	Non-Urban
MD	MDR9-0901	FW08MD008	39.06637	-77.38957	7	Potomac River	Urban
MD	MDR9-0902	FW08MD009	39.59856	-77.88461	6	Potomac River	Urban
MD	MDR9-0905	FW08MD015	39.62421	-78.42927	6	Potomac River	Non-Urban
MD	MDRM-1003		39.44732	-78.97643	5	North Branch Potomac River	Urban
ME	MER9-0901	FW08ME013	47.13183	-67.89810	6	Saint John River	Non-Urban
ME	MER9-0902	FW08ME016	47.15428	-68.94424	6	Saint John River	Non-Urban
ME	MER9-0903	FW08ME017	45.87867	-68.62034	5	East Branch Penobscot River	Non-Urban
ME	MER9-0904	FW08ME018	44.73738	-67.54984	5	Machias River	Non-Urban
ME	MER9-0905	FW08ME019	43.49885	-70.46423	6	Saco River	Urban
ME	MER9-0906	FW08ME021	45.25733	-68.94966	5	Pleasant River	Non-Urban
ME	MER9-0907	FW08ME022	44.42155	-69.70560	6	Kennebec River	Non-Urban
ME	MER9-0908	FW08ME026	44.50109	-69.67614	6	Kennebec River	Urban
ME	MERM-1001		45.89867	-68.61411	5	East Branch Penobscot River	Non-Urban
MI	MIR9-0901	FW08MI019	43.05631	-85.59421	6	Grand River	Urban
MI	MIR9-0902	FW08MI020	42.55230	-82.58846	5	Saint Clair River	Non-Urban
MI	MIR9-0903	FW08MI023	42.06127	-86.42370	6	Saint Joseph River	Urban
MI	MIR9-0904	FW08MI024	43.34147	-83.62441	5	Cass River	Non-Urban
MI	MIR9-0905	FW08MI026	43.02234	-86.02397	6	Grand River	Non-Urban
MI	MIR9-0906	FW08MI028	43.31097	-83.96788	6	Flint River	Non-Urban
MI	MIR9-0907	FW08MI030	42.82316	-84.93878	6	Grand River	Non-Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
MI	MIR9-0908	FW08MI033	42.54167	-84.62803	5	Grand River	Non-Urban
MI	MIR9-0909	FW08MI034	44.64747	-84.09452	5	Au Sable River	Non-Urban
MI	MIR9-0910	FW08MI036	43.00436	-82.52504	5	Black River	Urban
MI	MIRM-1001		44.67449	-84.32747	5	Au Sable River	Non-Urban
MN	MNR9-0901	FW08MN013	44.13244	-91.72952	8	Mississippi River	Non-Urban
MN	MNR9-0902	FW08MN016	48.48513	-93.72216	5	Big Fork River	Non-Urban
MN	MNR9-0903	FW08MN017	43.71940	-95.04622	6	Des Moines River	Non-Urban
MN	MNR9-0904	FW08MN018	45.12479	-93.99624	5	North Fork Crow River	Non-Urban
MN	MNR9-0905	FW08MN019	45.29729	-93.57205	6	Mississippi River	Urban
MN	MNR9-0906	FW08MN022	46.98702	-92.81150	5	Saint Louis River	Non-Urban
MN	MNR9-0907	FW08MN024	44.79956	-93.53312	8	Minnesota River	Urban
MN	MNR9-0908	FW08MN031	45.56509	-92.79530	6	Saint Croix River	Non-Urban
MN	MNR9-0909	FW08MN032	48.70306	-94.33570	6	Rainy River	Non-Urban
MN	MNR9-0910	FW08MN033	44.85127	-93.98283	5	South Fork Crow River	Non-Urban
MN	MNR9-0911	FW08MN034	45.19240	-94.28959	5	North Fork Crow River	Non-Urban
MN	MNR9-0912	FW08MN035	45.23426	-93.49636	7	Mississippi River	Urban
MN	MNR9-0913	FW08MN036	44.80659	-93.01163	8	Mississippi River	Urban
MN	MNR9-0914	FW08MN037	43.90668	-94.06030	5	Maple River	Non-Urban
MN	MNR9-0915	FW08MN039	47.27310	-93.78416	5	Mississippi River	Non-Urban
MN	MNR9-0916	FW08MN042	46.94815	-92.43222	5	Cloquet River	Non-Urban
MN	MNR9-0917	FW08MN043	47.25508	-96.20332	5	Wild Rice River	Non-Urban
MN	MNR9-0918	FW08MN045	44.94299	-95.77757	7	Minnesota River	Non-Urban
MN	MNRM-1001		46.76493	-96.77698	6	Red River of the North	Non-Urban
MO	MOR9-0901	FW08KY097	36.53474	-89.46723	10	Mississippi River	Non-Urban
MO	MOR9-0902	FW08KY209	36.60870	-89.30583	10	Mississippi River	Non-Urban
MO	MOR9-0903	FW08MO009	38.96337	-90.41565	9	Mississippi River	Non-Urban
MO	MOR9-0904	FW08MO011	37.02754	-90.64282	6	Black River	Non-Urban
MO	MOR9-0905	FW08MO013	38.24012	-91.80405	6	Gasconade River	Non-Urban
MO	MOR9-0906	FW08MO014	39.17605	-90.71722	8	Mississippi River	Non-Urban
MO	MOR9-0907	FW08MO015	36.78477	-93.71443	5	Flat Creek	Non-Urban
MO	MOR9-0908	FW08MO017	37.53898	-92.36536	5	Gasconade River	Non-Urban
MO	MOR9-0909	FW08MO018	37.88325	-90.54442	5	Big River	Non-Urban
MO	MOR9-0910	FW08MO019	38.68744	-92.94992	5	Lamine River	Non-Urban
MO	MOR9-0911	FW08MO021	38.30297	-90.62706	6	Big River	Non-Urban
MO	MOR9-0912	FW08MO025	38.82912	-90.41661	9	Missouri River	Urban
MO	MORM-1001		36.45933	-89.46806	10	Mississippi River	Non-Urban
MS	MSR9-0901	FW08MS008	34.00166	-88.51833	6	Tombigbee River	Non-Urban
MS	MSR9-0902	FW08MS016	33.91374	-88.53107	7	Tombigbee River	Non-Urban
MS	MSR9-0903	FW08MS025	30.88339	-88.77355	7	Pascagoula River	Non-Urban
MS	MSR9-0904	FW08MS042	32.08681	-90.94759	6	Big Black River	Non-Urban
MS	MSR9-0905	FW08MS050	32.58246	-89.84870	7	Pearl River	Non-Urban
MS	MSR9-0906	FW08MS052	31.09621	-89.27796	5	Black Creek	Non-Urban
MS	MSR9-0907	FW08MS053	30.40424	-88.58716	7	Pascagoula River	Urban
MS	MSRM-1001		32.84498	-89.99038	6	Big Black River	Non-Urban
MT	MTR9-0901	FW08MT022	47.06636	-114.76985	7	Clark Fork	Non-Urban
MT	MTR9-0902	FW08MT024	48.07747	-114.01872	5	Swan River	Non-Urban
MT	MTR9-0903	FW08MT025	48.07661	-104.39125	8	Missouri River	Non-Urban
MT	MTR9-0904	FW08MT029	46.56604	-107.96573	6	Musselshell River	Non-Urban
MT	MTR9-0905	FW08MT031	47.41435	-111.49864	7	Missouri River	Non-Urban
MT	MTR9-0906	FW08MT032	46.35963	-105.81405	6	Tongue River	Non-Urban
MT	MTR9-0907	FW08MT033	48.34598	-107.58381	5	Beaver Creek	Non-Urban
MT	MTR9-0908	FW08MT035	44.97626	-112.99659	5	Medicine Lodge Creek	Non-Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
MT	MTR9-0909	FW08MT039	46.45775	-110.37131	5	South Fork Musselshell River	Non-Urban
MT	MTR9-0910	FW08MT041	46.86783	-104.99520	8	Yellowstone River	Non-Urban
MT	MTR9-0911	FW08MT042	47.90948	-113.87070	5	Swan River	Non-Urban
MT	MTR9-0912	FW08MT043	47.45161	-111.30128	7	Missouri River	Urban
MT	MTR9-0913	FW08MT045	47.01519	-108.16501	5	Box Elder Creek	Non-Urban
MT	MTR9-0914	FW08MT047	48.45767	-109.92638	5	Big Sandy Creek	Non-Urban
MT	MTR9-0915	FW08MT048	46.11059	-106.45039	5	Rosebud Creek	Non-Urban
MT	MTR9-0916	FW08MT049	48.00521	-105.90923	8	Missouri River	Non-Urban
MT	MTR9-0917	FW08MT050	45.59732	-109.31160	6	Stillwater River	Non-Urban
MT	MTR9-0918	FW08MT058	47.61666	-112.68106	5	Sun River	Non-Urban
MT	MTR9-0919	FW08MT061	48.14733	-107.54900	5	Beaver Creek	Non-Urban
MT	MTR9-0920	FW08MT062	45.90936	-111.56689	7	Jefferson River	Non-Urban
MT	MTR9-0921	FW08MT063	47.79247	-109.27680	7	Missouri River	Non-Urban
MT	MTRM-1001		48.36584	-108.15370	6	Milk River	Non-Urban
NC	NCR9-0901	FW08NC017	36.48171	-77.65994	7	Roanoke River	Non-Urban
NC	NCR9-0903	FW08NC024	36.21492	-80.96029	6	Yadkin River	Non-Urban
NC	NCR9-0905	FW08NC029	35.45800	-77.67500	5	Contentnea Creek	Non-Urban
NC	NCR9-0906	FW08NC032	35.99756	-80.41813	6	Yadkin River	Urban
NC	NCR9-0907	FW08NC034	36.15021	-76.73789	7	Chowan River	Non-Urban
NC	NCR9-0908	FW08NC035	34.92337	-78.79820	7	Cape Fear River	Non-Urban
NC	NCR9-0915	FW08NC050	36.47170	-76.94345	6	Chowan River	Non-Urban
NC	NCRM-1001		36.42778	-77.58416	7	Roanoke River	Urban
ND	NDR9-0901	FW08ND020	46.11629	-97.38506	5	Wild Rice River	Non-Urban
ND	NDR9-0902	FW08ND021	47.12958	-102.23419	5	Knife River	Non-Urban
ND	NDR9-0903	FW08ND022	47.16034	-102.04208	6	Knife River	Non-Urban
ND	NDR9-0904	FW08ND023	48.17576	-97.69986	5	Forest River	Non-Urban
ND	NDR9-0905	FW08ND024	46.22149	-101.50255	5	Cannonball River	Non-Urban
ND	NDR9-0906	FW08ND027	47.50223	-97.33886	6	Goose River	Non-Urban
ND	NDR9-0907	FW08ND028	46.79972	-101.10684	5	Sweetbriar Creek	Non-Urban
ND	NDR9-0908	FW08ND029	47.97871	-103.82529	9	Missouri River	Non-Urban
ND	NDR9-0909	FW08ND031	46.76208	-97.19334	5	Maple River	Non-Urban
ND	NDR9-0910	FW08ND034	45.95829	-103.12024	6	North Fork Grand River	Non-Urban
ND	NDR9-0918	FW08ND043	45.97889	-98.16758	5	James River	Non-Urban
ND	NDR9-0921	FW08ND049	47.28029	-101.17793	9	Missouri River	Non-Urban
ND	NDRM-1001		46.45899	-102.65684	5	Cannonball River	Non-Urban
NE	NER9-0901	FW08NE010	41.14421	-101.21225	5	South Platte River	Non-Urban
NE	NER9-0902	FW08NE013	42.44907	-102.99896	5	Niobrara River	Non-Urban
NE	NER9-0903	FW08NE014	40.24541	-99.70083	6	Republican River	Non-Urban
NE	NER9-0904	FW08NE015	40.79966	-98.43775	7	South Channel Platte River	Non-Urban
NE	NER9-0905	FW08NE016	41.15205	-96.54845	5	Wahoo Creek	Non-Urban
NE	NER9-0906	FW08NE017	41.25373	-103.61138	5	Lodgepole Creek	Non-Urban
NE	NER9-0907	FW08NE019	42.71175	-98.15501	6	Niobrara River	Non-Urban
NE	NER9-0908	FW08NE022	40.35743	-98.13044	5	Little Blue River	Non-Urban
NE	NER9-0909	FW08NE024	42.94804	-99.44767	5	Keya Paha River	Non-Urban
NE	NER9-0910	FW08NE026	42.43359	-103.69931	5	Niobrara River	Non-Urban
NE	NER9-0914	FW08NE036	41.93933	-96.14472	9	Missouri River	Non-Urban
NE	NERM-1001		40.01724	-95.33155	9	Missouri River	Non-Urban
NH	NHR9-0901	FW08NH005	44.24308	-72.04818	5	Connecticut River	Non-Urban
NH	NHR9-0902	FW08NH007	43.06807	-72.44870	6	Connecticut River	Urban
NH	NHR9-0903	FW08NH009	43.86581	-72.17822	5	Connecticut River	Non-Urban
NH	NHR9-0904	FW08NH010	43.19317	-71.52351	7	Merrimack River	Urban
NH	NHR9-0905	FW08NH011	43.35118	-72.39344	6	Connecticut River	Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
NH	NHRM-1001		44.86397	-71.54817	5	Connecticut River	Non-Urban
NJ	NJR9-0901	FW08NJ004	41.27242	-74.84022	6	Delaware River	Non-Urban
NJ	NJR9-0902	FW08NJ005	40.50890	-74.46615	6	Raritan River	Urban
NJ	NJRM-1001		40.84438	-73.95545	7	Hudson River	Urban
NM	NMR9-0901	FW08NM005	35.79088	-104.61169	5	Mora River	Non-Urban
NM	NMR9-0902	FW08NM008	34.75015	-106.74249	7	Grande, Rio	Urban
NM	NMR9-0903	FW08NM010	32.92507	-105.33746	5	Pe±asco, Rio	Non-Urban
NM	NMR9-0904	FW08NM019	33.30077	-108.12551	6	East Fork Gila River	Non-Urban
NM	NMR9-0905	FW08NM022	36.70793	-108.21145	6	San Juan River	Urban
NM	NMR9-0906	FW08NM023	34.00485	-104.31475	5	Pecos River	Non-Urban
NM	NMRM-1001		34.35425	-106.85316	8	Grande, Rio	Non-Urban
NV	NVR9-0901	FW08NV019	40.70241	-116.52352	7	Humboldt River	Non-Urban
NV	NVR9-0902	FW08NV020	35.07424	-114.60429	9	Colorado River	Urban
NV	NVR9-0903	FW08NV022	41.77180	-117.80605	6	Quinn River	Non-Urban
NV	NVR9-0904	FW08NV026	41.39736	-117.46467	6	Little Humboldt River	Non-Urban
NV	NVR9-0920	FW08NV069	41.88666	-114.68661	6	Salmon Falls Creek	Non-Urban
NV	NVRM-1001		36.73991	-114.20599	7	Virgin River	Non-Urban
NY	NYR9-0901	FW08NY017	42.42297	-75.63217	5	Chenango River	Non-Urban
NY	NYR9-0902	FW08NY019	42.14603	-77.05409	5	Chemung River	Urban
NY	NYR9-0903	FW08NY021	42.82852	-73.98933	6	Mohawk River	Urban
NY	NYR9-0904	FW08NY023	42.16144	-75.85678	6	Chenango River	Urban
NY	NYR9-0905	FW08NY025	43.24866	-73.74077	6	Hudson River	Non-Urban
NY	NYR9-0906	FW08NY027	44.25928	-75.76743	5	Indian River	Non-Urban
NY	NYR9-0907	FW08NY028	42.47413	-73.78702	7	Hudson River	Urban
NY	NYR9-0908	FW08NY030	42.08065	-78.42363	5	Olean Creek	Urban
NY	NYR9-0909	FW08NY032	42.06591	-78.46922	6	Allegheny River	Urban
NY	NYR9-0910	FW08NY034	43.13767	-76.29551	7	Seneca River	Urban
NY	NYR9-0911	FW08NY035	42.02896	-76.39831	6	Susquehanna River	Non-Urban
NY	NYR9-0912	FW08NY037	42.93558	-74.19445	6	Mohawk River	Urban
NY	NYR9-0913	FW08NY039	42.34775	-75.69644	5	Chenango River	Non-Urban
NY	NYR9-0914	FW08NY040	43.25553	-73.58640	6	Hudson River	Urban
NY	NYR9-0915	FW08NY042	42.85984	-77.84331	6	Genesee River	Non-Urban
NY	NYR9-0916	FW08NY044	42.05576	-73.93195	7	Hudson River	Urban
NY	NYRM-1001		42.01278	-75.77946	5	Susquehanna River	Urban
OH	OHR9-0901	FW08OH012	39.30982	-82.96430	5	Paint Creek	Urban
OH	OHR9-0902	FW08OH017	40.26612	-81.87411	7	Muskingum River	Urban
OH	OHR9-0903	FW08OH018	40.58128	-81.39514	6	Tuscarawas River	Non-Urban
OH	OHR9-0904	FW08OH019	39.13619	-84.34206	6	Little Miami River	Urban
OH	OHR9-0905	FW08OH021	39.46603	-81.48059	7	Muskingum River	Urban
OH	OHR9-0906	FW08OH023	38.82668	-83.01769	6	Scioto River	Non-Urban
OH	OHR9-0907	FW08OH024	41.02829	-83.21295	5	Sandusky River	Non-Urban
OH	OHR9-0908	FW08OH027	41.20854	-80.81059	5	Mahoning River	Urban
OH	OHRM-1001		41.23380	-84.59052	6	Maumee River	Non-Urban
OK	OKR9-0901	FW08OK017	35.92582	-99.51525	7	Canadian River	Non-Urban
OK	OKR9-0902	FW08OK018	36.95800	-97.42192	6	Chikaskia River	Non-Urban
OK	OKR9-0903	FW08OK019	33.86362	-97.00595	7	Red River	Non-Urban
OK	OKR9-0904	FW08OK022	35.39936	-95.79265	6	North Canadian River	Non-Urban
OK	OKR9-0905	FW08OK024	35.53000	-99.13021	6	Washita River	Non-Urban
OK	OKR9-0906	FW08OK025	36.05503	-98.12901	6	Cimarron River	Non-Urban
OK	OKR9-0907	FW08OK026	34.63573	-95.12159	5	Kiamichi River	Non-Urban
OK	OKR9-0908	FW08OK027	35.92491	-97.86391	6	Cimarron River	Non-Urban
OK	OKR9-0909	FW08OK028	34.59159	-99.02375	5	Otter Creek	Non-Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
OK	OKR9-0910	FW08OK031	36.69695	-101.67678	5	Beaver River	Non-Urban
OK	OKR9-0911	FW08OK032	34.22208	-96.70688	6	Washita River	Non-Urban
OK	OKR9-0912	FW08OK034	33.91222	-95.54936	7	Red River	Non-Urban
OK	OKRM-1001		33.63195	-94.57131	7	Red River	Non-Urban
OR	ORR9-0901	FW08OR011	43.99127	-123.66433	5	Siuslaw River	Non-Urban
OR	ORR9-0902	FW08OR012	45.39535	-122.14937	5	Sandy River	Non-Urban
OR	ORR9-0903	FW08OR014	44.16795	-122.24967	5	McKenzie River	Non-Urban
OR	ORR9-0904	FW08OR015	42.41324	-123.15797	5	Rogue River	Urban
OR	ORR9-0905	FW08OR016	44.49071	-122.81372	5	South Santiam River	Non-Urban
OR	ORR9-0906	FW08OR019	44.37292	-123.83635	5	Alsea River	Non-Urban
OR	ORR9-0907	FW08OR021	45.16978	-120.48228	6	John Day River	Non-Urban
OR	ORR9-0908	FW08OR022	43.31057	-123.21152	5	North Umpqua River	Non-Urban
OR	ORR9-0909	FW08OR025	45.57558	-116.48749	8	Snake River	Non-Urban
OR	ORR9-0910	FW08OR026	44.24753	-120.85947	6	Crooked River	Non-Urban
OR	ORR9-0911	FW08OR027	44.06547	-123.10635	6	Willamette River	Urban
OR	ORR9-0912	FW08OR028	45.48478	-122.95994	5	Tualatin River	Urban
OR	ORR9-0913	FW08OR030	42.46206	-121.46883	6	Sprague River	Non-Urban
OR	ORR9-0914	FW08OR033	43.77084	-118.04897	6	Malheur River	Non-Urban
OR	ORRM-1001		45.76530	-117.75988	6	Grande Ronde River	Non-Urban
PA	PAR9-0901	FW08PA015	41.41992	-78.74775	5	Clarion River	Urban
PA	PAR9-0902	FW08PA016	41.47516	-79.51793	6	Allegheny River	Non-Urban
PA	PAR9-0903	FW08PA017	41.92258	-77.12923	5	Tioga River	Non-Urban
PA	PAR9-0904	FW08PA019	40.49180	-76.94813	7	Susquehanna River	Urban
PA	PAR9-0905	FW08PA020	40.93107	-80.37396	6	Beaver River	Urban
PA	PAR9-0906	FW08PA021	41.96990	-76.51192	6	Susquehanna River	Urban
PA	PAR9-0907	FW08PA023	41.28871	-77.34123	5	Pine Creek	Non-Urban
PA	PAR9-0908	FW08PA024	41.02419	-80.16323	5	Slippery Rock Creek	Non-Urban
PA	PAR9-0909	FW08PA030	40.14318	-75.51026	6	Schuylkill River	Urban
PA	PAR9-0910	FW08PA035	40.88572	-76.80151	6	West Branch Susquehanna River	Urban
PA	PAR9-0911	FW08PA036	41.24333	-80.50937	6	Shenango River	Urban
PA	PARM-1001		40.76001	-79.54689	6	Allegheny River	Urban
RI	RIR9-0901	FW08RI003	41.88014	-71.38130	5	Seekonk River	Urban
RI	RIR9-0902	FW08RI004	41.39354	-71.84080	5	Pawcatuck River	Urban
SC	SCR9-0901	FW08SC002	33.88391	-78.78474	6	Waccamaw River	Non-Urban
SC	SCR9-0902	FW08SC003	33.90909	-79.44030	7	Great Pee Dee River	Non-Urban
SC	SCR9-0903	FW08SC004	34.12622	-80.65031	6	Wateree River	Non-Urban
SC	SCR9-0904	FW08SC005	34.57048	-81.77745	5	Enoree River	Non-Urban
SC	SCRM-1001		34.93019	-80.86840	6	Catawba River	Urban
SD	SDR9-0901	FW08SD023	45.65636	-100.85083	6	Grand River	Non-Urban
SD	SDR9-0902	FW08SD026	42.99859	-97.00442	5	Vermillion River	Non-Urban
SD	SDR9-0903	FW08SD027	45.72850	-101.98438	6	Grand River	Non-Urban
SD	SDR9-0904	FW08SD029	45.00144	-98.63766	5	South Fork Snake Creek	Non-Urban
SD	SDR9-0905	FW08SD031	44.04386	-101.45112	6	South Fork Bad River	Non-Urban
SD	SDR9-0906	FW08SD032	44.80320	-102.54405	6	Sulphur Creek	Non-Urban
SD	SDR9-0907	FW08SD034	43.42244	-103.99180	7	Cheyenne River	Non-Urban
SD	SDR9-0908	FW08SD035	44.59103	-101.44896	5	Plum Creek	Non-Urban
SD	SDR9-0909	FW08SD036	44.81715	-103.69388	5	Indian Creek	Non-Urban
SD	SDR9-0910	FW08SD038	43.81009	-100.89714	6	White River	Non-Urban
SD	SDR9-0911	FW08SD039	45.76411	-100.68313	5	Oak Creek	Non-Urban
SD	SDR9-0912	FW08SD040	45.25933	-100.91089	6	Moreau River	Non-Urban
SD	SDR9-0913	FW08SD042	42.85420	-97.28016	9	Missouri River	Non-Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
SD	SDR9-0926	FW08SD063	44.52040	-101.99408	7	Cheyenne River	Non-Urban
SD	SDRM-1001		45.15509	-102.75259	6	Moreau River	Non-Urban
TN	TNR9-0901	FW08TN010	36.60637	-85.50504	6	Cumberland River	Non-Urban
TN	TNR9-0902	FW08TN011	35.95145	-83.55066	7	French Broad River	Non-Urban
TN	TNR9-0903	FW08TN012	35.10401	-85.36090	8	Tennessee River	Urban
TN	TNR9-0904	FW08TN013	35.67241	-87.26333	6	Duck River	Non-Urban
TN	TNR9-0905	FW08TN015	36.12508	-83.18162	6	Nolichucky River	Non-Urban
TN	TNRM-1003		35.06895	-85.33960	8	Tennessee River	Urban
TX	TXR9-0901	FW08TX022	26.04502	-97.79641	8	Grande, Rio	Non-Urban
TX	TXR9-0902	FW08TX023	29.77151	-101.73182	8	Grande, Rio	Non-Urban
TX	TXR9-0903	FW08TX028	33.05566	-97.25306	5	Denton Creek	Non-Urban
TX	TXR9-0904	FW08TX030	25.84893	-97.43996	8	Grande, Rio	Urban
TX	TXR9-0905	FW08TX033	35.97241	-100.82439	7	Canadian River	Non-Urban
TX	TXR9-0906	FW08TX035	29.15721	-97.38375	5	Sandies Creek	Non-Urban
TX	TXR9-0907	FW08TX037	30.57973	-94.99791	6	Trinity River	Non-Urban
TX	TXR9-0908	FW08TX038	26.23520	-98.54719	8	Grande, Rio	Urban
TX	TXR9-0909	FW08TX042	28.30211	-98.05815	7	Nueces River	Non-Urban
TX	TXR9-0910	FW08TX043	29.23893	-98.45168	5	Medina River	Non-Urban
TX	TXR9-0911	FW08TX046	31.77034	-103.77929	7	Pecos River	Non-Urban
TX	TXR9-0912	FW08TX050	28.95042	-100.64937	8	Grande, Rio	Non-Urban
TX	TXR9-0913	FW08TX052	34.56295	-100.62735	5	Prairie Dog Town Fork Red River	Non-Urban
TX	TXR9-0914	FW08TX053	31.93453	-95.43336	5	Neches River	Non-Urban
TX	TXR9-0915	FW08TX055	31.76248	-100.14048	6	Colorado River	Non-Urban
TX	TXR9-0916	FW08TX057	30.88169	-93.57254	6	Sabine River	Non-Urban
TX	TXR9-0917	FW08TX058	28.72577	-99.81584	5	Nueces River	Non-Urban
TX	TXR9-0918	FW08TX059	31.55041	-97.09174	7	Brazos River	Urban
TX	TXRM-1001		29.02616	-103.31277	8	Grande, Rio	Non-Urban
UT	UTR9-0901	FW08UT014	37.22372	-109.20869	7	San Juan River	Non-Urban
UT	UTR9-0902	FW08UT016	41.59255	-111.14148	6	Bear River	Non-Urban
UT	UTR9-0903	FW08UT020	40.98295	-111.45065	5	Weber River	Non-Urban
UT	UTR9-0904	FW08UT021	37.74306	-112.09859	5	East Fork Sevier River	Non-Urban
UT	UTR9-0905	FW08UT022	39.08975	-109.10164	7	Colorado River	Non-Urban
UT	UTR9-0906	FW08UT023	38.35280	-109.75346	7	Colorado River	Non-Urban
UT	UTR9-0907	FW08UT026	39.30653	-110.40517	6	Price River	Non-Urban
UT	UTRM-1001		38.76150	-109.32372	7	Colorado River	Non-Urban
VA	VAR9-0901	FW08VA014	36.96530	-82.05180	5	Clinch River	Non-Urban
VA	VAR9-0902	FW08VA017	36.87645	-79.06775	5	Banister River	Non-Urban
VA	VAR9-0903	FW08VA018	37.31028	-80.68118	5	Walker Creek	Non-Urban
VA	VAR9-0904	FW08VA020	36.76203	-78.87142	5	Banister River	Non-Urban
VA	VAR9-0905	FW08VA022	38.30562	-78.90091	5	North River	Non-Urban
VA	VAR9-0906	FW08VA026	37.59328	-79.38321	6	James River	Non-Urban
VA	VAR9-0911	FW08VA038	37.12219	-79.35359	6	Roanoke River	Non-Urban
VA	VARO-1001		37.83120	-77.12220	5	Mattaponi River	Non-Urban
VT	VTR9-0901	FW08VT006	42.79337	-72.52477	6	Connecticut River	Non-Urban
VT	VTR9-0902	FW08VT009	43.79297	-72.67631	5	White River	Non-Urban
VT	VTR9-0903	FW08VT011	44.48913	-73.14832	5	Winooski River	Urban
WA	WAR9-0901	FW08WA015	48.95608	-119.69332	5	Similkameen River	Non-Urban
WA	WAR9-0902	FW08WA016	45.69861	-120.41753	9	Columbia River	Non-Urban
WA	WAR9-0903	FW08WA017	48.52373	-122.05344	6	Skagit River	Non-Urban
WA	WAR9-0904	FW08WA020	46.27617	-118.19248	5	Touchet River	Non-Urban
WA	WAR9-0905	FW08WA022	47.72892	-121.42756	5	South Fork Skykomish River	Non-Urban

List of 2013-2014 National Rivers and Streams Fish Tissue Study Sampling Locations[1]

State	Site ID 2013- 2014	Site ID 2008-2009[2]	Lat	Long	Stream Order	River Name	Urban/ Non-urban
WA	WAR9-0906	FW08WA028	47.50383	-119.29244	5	Trail Lake Coulee	Non-Urban
WA	WAR9-0907	FW08WA029	47.84358	-121.69460	5	Skykomish River	Urban
WA	WAR9-0908	FW08WA032	46.27117	-119.57047	7	Yakima River	Non-Urban
WA	WAR9-0909	FW08WA033	47.69212	-121.96626	5	Snoqualmie River	Non-Urban
WA	WARM-1001		47.18231	-120.90807	6	Yakima River	Non-Urban
WI	WIR9-0901	FW08WI021	44.68674	-92.69129	8	Mississippi River	Non-Urban
WI	WIR9-0902	FW08WI022	44.00670	-90.05381	5	Yellow River	Non-Urban
WI	WIR9-0903	FW08WI029	42.53137	-90.64118	8	Mississippi River	Urban
WI	WIR9-0904	FW08WI030	43.21793	-89.82451	6	Wisconsin River	Non-Urban
WI	WIR9-0905	FW08WI031	44.74817	-91.15140	5	Eau Claire River	Non-Urban
WI	WIR9-0906	FW08WI033	43.82745	-91.27193	8	Mississippi River	Urban
WI	WIR9-0907	FW08WI034	44.97808	-89.63045	5	Wisconsin River	Urban
WI	WIR9-0908	FW08WI036	45.07960	-88.62394	5	Wolf River	Non-Urban
WI	WIR9-0909	FW08WI039	46.41642	-90.74116	5	Marengo River	Non-Urban
WI	WIR9-0910	FW08WI040	45.66598	-91.17972	5	Chippewa River	Non-Urban
WI	WIR9-0911	FW08WI041	42.65018	-90.03286	5	Pecatonica River	Non-Urban
WI	WIR9-0912	FW08WI044	42.89687	-88.89542	6	Rock River	Urban
WI	WIR9-0913	FW08WI045	44.36113	-91.91075	5	Buffalo River	Non-Urban
WI	WIR9-0914	FW08WI046	43.56469	-89.65428	6	Wisconsin River	Non-Urban
WI	WIRM-1001		45.58439	-89.46554	5	Wisconsin River	Non-Urban
WV	WVR9-0901	FW08WV005	37.54136	-82.03361	5	Tug Fork	Non-Urban
WV	WVR9-0902	FW08WV006	39.13143	-81.34434	5	Hughes River	Non-Urban
WV	WVR9-0903	FW08WV007	39.91877	-80.79683	8	Ohio River	Non-Urban
WV	WVR9-0904	FW08WV008	38.58809	-80.89452	5	Elk River	Non-Urban
WV	WVRM-1001		39.24890	-77.81410	6	Shenandoah River	Urban
WY	WYR9-0901	FW08WY016	42.85202	-106.18585	7	North Platte River	Non-Urban
WY	WYR9-0902	FW08WY020	41.59625	-109.14876	6	Bitter Creek	Non-Urban
WY	WYR9-0903	FW08WY021	44.47897	-109.38303	5	North Fork Shoshone River	Non-Urban
WY	WYR9-0904	FW08WY022	43.41402	-106.28598	5	Salt Creek	Non-Urban
WY	WYR9-0905	FW08WY026	44.62542	-105.30171	5	Little Powder River	Non-Urban
WY	WYR9-0906	FW08WY029	44.26445	-107.90091	6	Nowood River	Non-Urban
WY	WYR9-0907	FW08WY030	43.34957	-104.29569	6	Lance Creek	Non-Urban
WY	WYR9-0908	FW08WY034	44.69584	-106.33826	5	Clear Creek	Non-Urban
WY	WYR9-0909	FW08WY038	43.96561	-106.17143	6	Powder River	Non-Urban
WY	WYR9-0910	FW08WY039	42.82884	-106.36679	7	North Platte River	Urban
WY	WYR9-0911	FW08WY040	41.96621	-110.00073	7	Green River	Non-Urban
WY	WYR9-0912	FW08WY042	44.38685	-104.67731	5	Inyan Kara Creek	Non-Urban
WY	WYR9-0913	FW08WY043	42.11494	-104.98519	5	Laramie River	Non-Urban
WY	WYRM-1002		43.44464	-104.51173	6	Cheyenne River	Non-Urban

[1] This list of sites is subject to change as the project proceeds. For example, access to some sites may not be granted by property owners. Other sites may not yield fish of suitable size or species. OST maintains the list of valid sites, and this QAPP will **not** be revised just to address changes in the list of sites.

[2] Empty cells in this column represent sites that were not selected for sampling in the 2008-2009 NRSA, but that are included in sample design for the 2013-2014 NRSA.

Appendix B

2013-2014 National Rivers and Streams Assessment Tissue Preparation, Homogenization, and Distribution Procedures

Note: This appendix contains the fish tissue preparation, homogenization, and distribution procedures developed by OST for the 2013-2014 NRSA. The information in this appendix formed the basis for the contractual SOW issued by CSC to the sample preparation laboratory. Therefore, the details of this appendix have **not** been revised to reflect that OST has prepared this QAPP. In addition, the acronyms, abbreviations, and units of measure listed in this appendix are not included in the list of acronyms at the front of the QAPP. Rather, they are spelled out on first usage in the appendix.

Appendix B

2013-2014 National Rivers and Streams Assessment
Tissue Preparation, Homogenization, and Distribution Procedures

I. PURPOSE

This document describes the procedures that the sample preparation laboratory will follow when preparing fish tissue samples for EPA's National Rivers and Streams Assessment (2013-2014 NRSA) under contract to CSC. Adherence to these procedures will ensure that fish tissue preparation activities are performed consistently across all study samples and in a manner consistent with previous EPA fish tissue studies. The effort is divided into four components:

- A kickoff meeting and workshop involving all study participants, including the sample preparation laboratory staff, EPA, CSC, and Tetra Tech (EPA's sampling contractor)
- An initial demonstration of capabilities, also referred to as the QA study
- Normal fish tissue processing and distribution procedures, including quality control steps
- Preparation and analyses of rinsate samples and blanks for mercury, selected polychlorinated biphenyls (PCBs), and selected polybrominated diphenyl ethers (PBDEs), and preparation of rinsate samples and blanks for perfluorinated compounds (PFCs) to be analyzed by a laboratory under a separate CSC purchase order.

Each of these components is described in detail below.

EPA will prepare a quality assurance project plan (QAPP) for this project which will include the details of fish tissue sample preparation processes described in this SOW, including the description of the analytical procedures and the QC acceptance criteria. After award, CSC will provide the laboratory with a copy of EPA's QAPP for the project.

II. KICKOFF MEETING AND WORKSHOP

Following award of a purchase order, CSC will schedule a kickoff meeting and workshop to be held at the sample preparation laboratory at a mutually agreed upon date and time. Staff from all study participants, including the sample preparation laboratory, EPA, CSC, and Tetra Tech, will meet at the sample preparation laboratory to review the overall 2013-2014 NRSA project goals, the roles of each participant, the fish sample preparation procedures, and the communication strategies necessary to ensure successful completion of the project. In conjunction with that meeting, CSC will provide whole fish samples that will be used during a hands-on workshop on the specific procedures for fish sample preparation. All the sample preparation laboratory staff involved in the preparation of fish samples must attend the kickoff meeting and workshop.

The kickoff meeting and workshop will be billable to the CSC subcontract as a fixed price line item.

III. INITIAL DEMONSTRATION OF CAPABILITIES

A routine aspect of any procedure for sample preparation or analysis is an initial demonstration of capabilities, or QA study. For the 2013-2014 NRSA project, the sample preparation laboratory will receive three whole large fish provided by Tetra Tech. Each of these fish will be treated as a separate project sample and will be prepared using the procedures detailed in Section IV (i.e., Steps 1 to 24). In between each fish, the sample preparation laboratory will prepare the entire series of equipment rinsate samples and blanks described in Section IV, Steps 32 and 33, but analyze only the rinsates and blanks for mercury, PCBs, and PBDEs (Steps 34 and 35, and Attachment 1). The sample preparation laboratory

will perform triplicate determinations of lipids on each test sample, as described in Step 31. The results of the QA study will be reported to CSC.

Note: The sample preparation laboratory will not be authorized to process actual project samples until CSC determines that the QA study results meet the project objectives, including the adequacy of the sample preparation laboratory's equipment cleaning and homogenization procedures.

The sample aliquots prepared from these QA study samples will be stored frozen at the sample preparation laboratory for possible future use by EPA, or until CSC authorizes their disposal. Each of the samples prepared for the QA study will be billable under the CSC subcontract at the cost for a normal project sample.

IV. FISH TISSUE PROCESSING AND DISTRIBUTION PROCEDURES

The procedures for processing and distributing 2013-2014 NRSA composite fish tissue samples are described below. The process description is organized into the following components, including the quality control (QC) procedures:

A. Sample Receipt and Storage
B. Sample Handling
C. Filleting and Homogenization Procedures, Including Removal of Plug Samples for Mercury Analysis
D. Aliquoting and Distribution Procedures
E. Equipment Cleaning between Composite Samples
F. Lipid Determination on Every Homogenized Composite Sample
G. Quality Control (QC) Procedures
H. Reporting Requirements
I. Shipping Samples

The individual steps in the overall process are presented as a series of numbered steps across the nine components listed above.

Note: The sample preparation laboratory may **not** process any fish tissue samples until directed by CSC to proceed. No samples collected from NRSA sampling sites may be processed until after the kickoff meeting and workshop and until CSC reviews the results of the initial demonstration of capabilities (QA study) described in Section III above.

Composite Sample Classifications

For the purposes of the 2013-2014 NRSA, EPA has classified each valid sample as a "routine" composite sample, or a "non-routine" composite sample, based on the following definitions:

- **Routine sample** – A routine composite sample consists of five individual adult fish of a single species that meet EPA's length requirements (i.e., length of the smallest specimen in the composite is at least 75% of the length of the largest individual). Fillets from both sides of all five fish will be removed (total of 10 fillets) and homogenized to prepare one composite fillet sample.

- **Non-routine sample** – A non-routine sample is any sample that does not meet the definition of a routine sample, including those that do not meet the 75% rule and those with fewer or greater than five fish. When non-routine samples are sent to the sample preparation laboratory, EPA and CSC will provide instructions for processing the non-routine samples. These instructions may include discarding some of the fish in the composite sample based on size before proceeding with filleting and homogenizing. In cases when fewer or more than five fish were collected, instructions may include processing some or all of those fish in the composite sample.

Each of the five fish in the routine samples must be filleted before homogenization. **For non-routine composites, only the designated specimens (identified by specimen number) will be filleted and homogenized.** For both types of samples, the specimens to be included in each composite must be scaled (i.e., scales removed) and both fillets from each specimen prepared as skin-on fillets (belly-flap included) to form the fillet composites.

Note: The classifications described above do not include samples that were collected from an incorrect sampling location, were an unnecessary duplicate sample, or contained an inappropriate fish species. EPA does not plan on using these "invalid" samples for the 2013-2014 NRSA, so it is imperative that the sample preparation laboratory not process any sample without specific instructions from CSC. Therefore, samples will be retained in frozen storage and processed only upon receipt of CSC-issued instructions. If the status of any composite sample in the instructions is not clear, contact CSC and wait for clarification.

IV.A Sample Receipt and Storage

Fish samples for the 2013-2014 NRSA are being collected by various organizations cooperating with EPA in this study, including State agencies, other Federal agencies, and contractors. Sample collection is expected to begin as early as May 2013, and continue through approximately November 2014, with the bulk of collection to occur between June and October of 2013 and June and October of 2014, respectively (i.e., a two-year sampling effort). Ultimately, EPA anticipates the collection of composite samples from up to 453 sites by the end of the collection effort in late 2014.

Samples will be shipped directly from the field sampling crews to the sample preparation laboratory for storage and processing. Therefore, the sample preparation laboratory must have sufficient freezer space to store **up to 150 unprocessed fish composite samples** (e.g., 150 5-fish composites) at a temperature of less than or equal to -20 °C from the time of receipt until completion of sample processing and sufficient freezer space to store **homogenized tissue aliquots from up to 100 processed samples** (e.g., up to 900 jars) prior to distribution. CSC will provide as much advance notice of sample shipments from the field crews as possible, but we anticipate that some shipments may arrive before we can notify the laboratory. CSC also will provide the laboratory with a list of all of the valid sites from which samples are being collected.

1. Although samples will be shipped frozen, on dry ice, they must be inspected promptly on receipt. As samples are received, the sample custodian must:

    - Check that each shipping container has arrived undamaged and verify that samples are still frozen and in good condition.
    - Check the temperature of one of the samples in the cooler using a thermometer that reads to at least -20 ºC, or an infra-red (IR) temperature "gun" and record the reading.
    - Verify that all associated paperwork is complete, legible, and accurate.
    - Compare the information on the label on each individual fish specimen to the sample tracking form for each composite and verify that each specimen was included in the shipment and is properly wrapped and labeled.
    - Notify CSC of the fact that samples were received and of any discrepancies in the paperwork identified above.
    - Check that the samples were collected from sites on the list of valid whole-fish tissue sampling locations (uniquely designated by the site identification number) provided by CSC, and notify CSC immediately if samples have been received from sites not on that list.
    - Transfer the samples to the freezer for long-term storage.

2. Notify CSC immediately about any problems encountered upon receipt of samples. Problems involving sample integrity, conformity, or inconsistencies for fish tissue samples should be reported to CSC in writing (e.g., by email) as soon as possible following sample receipt and inspection.

   Following sample processing, the sample preparation laboratory must store sample aliquots frozen to less than or equal -20 °C until they are distributed to the laboratories performing analyses under separate CSC purchase orders (see Sec IV.I).

IV.B Sample Handling

The whole fish collected for the 2013-2014 NRSA must remain frozen at less than or equal to -20 ºC until the sample processing laboratory receives composite-specific processing instructions from CSC. Samples to be processed must be retrieved from the freezer, with their associated paperwork, and allowed to partially thaw before they can be processed.

3. CSC will send sample processing instructions to the laboratory via email. The instructions consist of an Excel spreadsheet file that details the site and sample identifiers for fish that EPA has determined are routine valid five-fish composites, or non-routine composites to be prepared. At a minimum, the Excel file will list the following fields for each individual fish specimen in a given composite sample:

   - Site ID
   - Date of collection
   - Sample ID (XXXXXX.YY, where YY usually ranges from 1 to 5 specimens in the composite, but can range up to 10)
   - Common name for the fish species
   - Measured length of each specimen in mm
   - Relative length order of the specimens in the composite (e.g., "1" for the longest specimen, etc.)
   - Composite type (predator or bottom dweller)
   - Composite classification (Routine, Non-Routine, or Invalid)
   - Deviation (e.g., why it is not routine or not valid)
   - Instructions (sample-specific details about which fish to process), including which two specimens to be used for plug sample collection

   Samples shipped to the laboratory that EPA identifies as "invalid" are to be held in the freezer until CSC provides instructions for their disposition or disposal.

4. When retrieving samples from the freezer, the sample custodian must:

   - Verify that all associated paperwork stored with the samples is complete, legible, and accurate.
   - Compare the information on the label on each individual fish specimen to the processing instructions and notify CSC of any discrepancies between the sample labels and the Excel file of instructions. Problems involving sample paperwork, sample integrity, or custody inconsistencies for all fish tissue samples should be reported to CSC in writing (e.g., by email) as soon as possible following sample retrieval and inspection. **Do not proceed with sample processing until discrepancies are resolved.**

     Note: The hardcopy paperwork generated by the field samplers and stored with the samples does *not* contain all of the information in the Excel instruction files. Therefore, lack of information on hardcopy field paperwork regarding the composite type, composite classification, or deviation is *not* a discrepancy that must be reported.

IV.C. Filleting and Homogenization Procedures, Including Plug Sampling for Mercury Analysis

As part of the overall 2013-2014 NRSA, mercury analyses will be performed on two types of samples: aliquots of the homogenized composite fillet samples and plugs removed from two fish in each composite with an 8-mm disposable biopsy tool (Acuderm brand Acu-Punch or equivalent). The sample processing instructions sent by CSC will include instructions to collect one plug sample each from two specific fish specimens in the sample composite, using the procedure described in Steps 11 - 16. Prior to collecting the plug sample for mercury, there are a number of steps that must be taken before preparing both types of samples.

5. Prior to preparing any samples, thoroughly clean utensils and cutting boards using the following series of procedures:

   - Wash with a detergent solution (phosphate- and scent-free) and warm tap water
   - Rinse three times with warm tap water
   - Rinse three times with DI water
   - Rinse with acetone
   - Rinse three times with DI water
   - Rinse with (not soak in) 5% nitric acid
   - Rinse three times with DI water

   To control contamination, separate sets of utensils and cutting boards must be used for scaling fish and for filleting fish.

   Note: The biopsy punches provided by EPA for collecting the plug samples are to be used as received and are **not** subjected to the cleaning procedures above.

6. Put on powder-free nitrile gloves before unpacking individual fish specimens for plug sampling (as directed) and for filleting and tissue homogenization. As samples are unpacked and unwrapped, inspect each fish carefully to verify that it has not been damaged during collection or shipment. If damage (e.g., tearing the skin or puncturing the gut) is observed, document it in the laboratory project log sheet and notify CSC before proceeding further.

7. The sample collection personnel measured the total length of each fish specimen in the field and recorded those lengths on the sample tracking form. However, the label applied to each wrapped specimen does not include the length information, and it may be difficult to reproduce the field measurements of fish length when the specimens are still partially frozen.

   Therefore, begin processing the specimens by laying them out in order by specimen number (the portion of the sample ID after the decimal point) and allowing them to partially thaw to the point that each specimen can be laid relatively flat. Using the length data on the sample tracking form (or the relative length order data in the fish sample processing instructions spreadsheet), confirm that the specimen ID for the longest specimen recorded on the tracking form is the same as the specimen ID on the label of the longest specimen. Repeat this relative length comparison for each of the other specimen IDs to ensure that the length orders based on the recorded lengths in the sample tracking form are consistent with the specimen IDs on the individual fish labels. This check is important for confirming that the field crews attached the correct label to each fish in the composite sample.

   If discrepancies are observed, document them in the laboratory project log sheet and notify CSC before proceeding further.

8. Weigh each fish to the nearest gram (wet weight) prior to any sample processing. Enter weight information for each individual fish into a laboratory project log sheet. Individual specimen weights eventually will be transferred to spreadsheets for submission to CSC.

9. Rinse each fish with deionized water as a precautionary measure to treat for possible contamination from sample handling in the field. Use HDPE wash bottles for rinsing fish and for cleaning homogenization equipment and utensils. Do **NOT** use Teflon® wash bottles for these procedures, because PFCs are among the target analytes for this study.

10. Before beginning the scaling process for the first fish in the composite, put on new powder-free nitrile gloves. (Gloves must be changed *between* composites, but the same gloves may be used for all fish *within* a given composite.) Fish with scales must be scaled (and any adhering slime should be removed) prior to filleting. ***Begin with the two fish specimens designated by EPA for plug sampling.*** Scale the first designated fish by laying it flat on a clean glass cutting board and scraping from the tail to the head using a stainless steel scaler or the blade-edge of a clean stainless steel knife.

11. Turn the first scaled fish specimen designated by EPA for the plug sample so that the left side is facing up. Insert a new 8-mm biopsy punch into the fish through the tissue in the dorsal (upper) portion of the specimen between the dorsal fin and the lateral line, avoiding areas where the punch may contact the viscera (internal organs). Insert the punch with a slight twisting motion, cutting the skin and muscle tissue. Once the punch is inserted to its full depth, use a slight bending or tilting motion of the punch to break off the end of the sample.

12. Remove the biopsy punch, taking care to ensure that the sample remains in the punch.

13. Place a laboratory pipette bulb on the end of the biopsy punch and squeeze the bulb quickly, blowing the tissue sample into a tared clean 20-mL scintillation vial (supplied by EPA).

14. Repeat Steps 10 through 13 with the second fish specimen designated by EPA for plug sampling. The same biopsy punch used for the first specimen is used for the second specimen.

15. After transferring the second plug to the tared vial, weigh the tared vial containing the two plugs and determine the combined weight of the plugs by difference. Label the vial with the Site ID, and the two Specimen IDs (XXXXXX.YY and XXXXXX.ZZ, where YY and ZZ are the specimen numbers of the fish designated by EPA for the plug samples), the total weight of the plugs, and the date the sample was processed.

Note: The two punch samples should yield at least 0.5 to 0.7 grams of fish tissue for mercury analysis.

16. Transfer the vial to the freezer within 30 minutes. (The vial may be stored in a small cooler in the sample processing area on water ice or dry ice while the remainder of the composite sample is processed.)

17. Continue scaling all the other fish in the sample composite as described in Step 10 above. Filleting can proceed after all scales have been removed from the skin and a separate clean cutting board and fillet knife are prepared or available.

18. Place each fish on a clean glass cutting board in preparation for the filleting process. Note that filleting should be conducted under the supervision of an experienced fisheries biologist, if possible. Ideally, fish should be filleted while ice crystals are still present in the muscle tissue. Fish should be thawed only to the point where it becomes possible to make an incision into the flesh. Remove both fillets (lateral muscle tissue with skin attached) from each fish specimen using clean, high-quality stainless steel knives. Include the belly flap (ventral muscle and skin) with each fillet. Care must be taken to avoid contaminating fillet tissues with material released from inadvertent puncture of internal organs. In the event that an internal organ is punctured, rinse the fillet with deionized water immediately after filleting and make a note on the laboratory project log sheet that a puncture has

occurred. Bones still present in the tissue after filleting should be carefully removed using the tip of the fillet knife or a clean pair of forceps.

19. Samples should be homogenized partially frozen for ease of grinding. Composite the fillets using the "batch" method, in which all of the fillets from the individual specimens that comprise the sample are homogenized together, regardless of each individual specimen's proportion to one another (as opposed to the "individual" method, in which equal weights of tissue from each specimen are added together).

20. Process each sample using a size-appropriate homogenization apparatus (e.g., automatic grinder or high-speed blender). Entire fillets (with skin and belly flap) from both sides of each fish must be homogenized, and the entire homogenized volume of all fish fillets from the composite will be used to prepare the composite. Mix the tissues thoroughly until they are completely homogenized as evidenced by a final composite sample that consists of a fine paste of uniform color and texture. Chunks of skin or tissue will hinder extraction and digestion and, therefore, are NOT acceptable. Grinding of tissue may be easier when tissues are partially frozen. Chilling the grinder briefly with a few small pieces or pellets of dry ice may also keep the tissue from sticking to the equipment. Pellets of dry ice also may be added to the tissue as it enters the grinder.

21. Grind the sample a second time, using the same grinding equipment. This second grinding should proceed more quickly. The grinding equipment does not need to be cleaned between the first and second grinding of the sample. The final sample must consist of a fine paste of uniform color and texture. If there are obvious differences in color or texture, grind the entire sample a third time.

22. Measure the collective weight of the homogenized fillet tissue from each composite to the nearest gram (wet weight) after processing and record the total homogenized tissue weight of each composite on a laboratory project log sheet. The collective weight of the homogenized tissue from each sample will be transferred to spreadsheets for submission to CSC. At least 536 g of homogenized tissue will be needed to fill all of the containers in Table 1 below with their minimum acceptable masses. **If a sample does not yield at least 536 g of homogenized tissue, contact CSC via email immediately and await instructions.** As appropriate, place any less-than-536-g homogenized samples in the freezer while waiting for instructions, which are likely to involve preparing fewer archive aliquots.

23. After the final (second or third) grinding, clean the **grinding equipment and all other sample preparation equipment** using the procedures described in Step 29.

24. Once in every batch of 20 samples, verify the continued absence of equipment contamination and uniformity of homogenization using the procedures described in Steps 32 to 37.

IV.D. Aliquoting and Distribution Procedures

25. The sample preparation laboratory will prepare one bulk homogenate tissue aliquot per fish composite sample and use it to fill the pre-cleaned sample containers specified for each type of sample listed in Table 1, following the procedures described in Step 26. **Except as noted in Table 1, all containers will be provided by the sample preparation laboratory.** Documentation of their cleanliness provided by the vendor (i.e., certificates of analysis) must be retained by the sample preparation laboratory and provided to CSC on request. The target masses listed in Table 1 are designed to provide enough tissue for multiple analyses of each sample and analyte type, including tissue for QC purposes, as needed. The sample preparation laboratory should not exceed those target masses when filling the containers. The order of the containers and target masses in Table 1 are important and are designed to ensure that adequate tissue is available for all analyses, as well as archiving.

Table 1. 2013-2014 NRSA Initial Tissue Sample Aliquot Requirements

Analysis	Target Mass	Container Type	Destination
Mercury, plug	0.5 - 0.7 g	20-mL glass scintillation vial (provided by EPA)	TBD
Mercury, fillet	5 - 10 g	50-mL HDPE straight-sided jar with **foil-lined lid**, or conical HDPE tube with snap top	TBD
PFCs	60 - 65 g	100-mL HDPE straight-sided jar with **foil-lined lid**, or conical HDPE tube with snap top. ***PTFE lid liners not allowed.***	TBD
PBDEs	30 - 35 g	125-mL straight-sided amber or clear glass jar with PTFE-lined lid	TBD
PCBs	30 - 35 g	125-mL straight-sided amber or clear glass jar with PTFE-lined lid	TBD
Lipids	10 - 15 g	Laboratory's choice, as this aliquot will be used in-house to determine the lipid content of the sample	In-house
Bulk Archive 1	250 - 260 g	500-mL straight-sided amber or clear glass jar with **foil-lined lid**	CSC Sample Repository
Small Archive 1	50 - 60 g	125-mL straight-sided amber or clear glass jar with **foil-lined lid**	CSC Sample Repository
Small Archive 2	50 - 60 g	125-mL straight-sided amber or clear glass jar with **foil-lined lid**	CSC Sample Repository
Bulk Archive 2	All remaining mass up to 260 g	500-mL straight-sided amber or clear glass jar with **foil-lined lid**	CSC Sample Repository
Total (to the nearest gram)*	536 - 801 g	*Assumes at least 50 g of tissue is available for Bulk Archive 2*	

* In the event that insufficient fish tissue mass exists to prepare the required number of aliquots, contact CSC for instructions, per Step 22.

26. Prepare the sample aliquots for **mercury, PFCs, PBDEs, and PCBs**. Weigh an appropriate clean sample container (Table 1) to the nearest 0.5 g and record the weight. Transfer sufficient aliquots of ground sample to the container to achieve the target mass for that container in Table 1, weigh the container again, record the weight, and determine the weight of the aliquot to the nearest 0.5 g by difference. **The sample preparation laboratory must use foil-lined lids for jars containing the tissue aliquots for PFC analysis and the archived tissue samples, as specified in Table 1.**

    Note: The archive sample jars are not filled until after sufficient volume for lipids determination has been collected, as described in Step 27. For the sample used for homogeneity testing, the archive jars are not filled until triple the lipid mass is collected (see Step 36).

    When filling jars, leave sufficient space at the top of each jar to allow for expansion of the tissue as it freezes. *In no case should jars be filled beyond 80% capacity, as this may result in breakage on freezing.* Wipe off the outside of the jars to remove any tissue residue or moisture. Fill out a label for each container using a waterproof marker. Include the following information (at a minimum) on each label:

    - site identification number,
    - sample identification number,
    - analysis type (e.g., mercury, PFCs, PBDEs, etc.),
    - aliquot weight (to the nearest 0.5 gram),
    - preparation batch ID, and
    - preparation date (e.g., mm/dd/yyyy)

(Other information may be included on the label at the laboratory's discretion, provided that CSC is given an explanation of each additional field.)

Affix the label to the container with clear wide tape. Place each container inside one heavy-weight food-grade self-sealing plastic freezer bag to avoid sample loss due to breakage. Freeze the tissue aliquots at -20 ºC, and maintain samples in the freezer until directed by CSC to ship them to the analytical laboratories. (CSC will not issue such instructions until equipment rinsate and homogeneity tests described in Steps 31 to 37 have been completed, reported, evaluated, and determined to be acceptable.)

27. After filling all of the containers for the aliquots for mercury, PFCs, PBDEs, and PCBs, remove 10 to 15 g of homogenized tissue to be used by the sample preparation laboratory to determine the lipid content of each sample. Place this aliquot in a clean glass or plastic container of suitable size and label it with the site ID and sample number. Transfer the lipid aliquot to the appropriate staff performing the lipid determinations described in Steps 31, 36, and 37.

28. The archive sample jars are not filled until after sufficient volume for lipids determination has been collected. Once the aliquots for mercury, PFCs, PBDEs, PCBs, and lipids have been collected, the remaining tissue mass is used to create at five archive samples. Begin by transferring 250 - 260 g of tissue to the first bulk archive sample container, thus ensuring that at least one large volume (bulk) aliquot is archived. Continue by transferring one 50 - 60 g aliquot to each of the two small archive containers. Ideally, sufficient homogenized fillet tissue mass will remain to produce a second bulk archive container. Therefore, transfer 250 - 260 g of tissue to the second bulk archive sample container. However, if less than 250 g of tissue is available, transfer all of the remaining homogenized tissue to the second bulk archive container. Seal and label the containers as described in Step 26 for the other aliquots.

    Note: Step 22 requires that the laboratory contact CSC whenever a sample does not yield at least 536 g of tissue. CSC will provide direction to the laboratory regarding samples yielding less than 536 g of tissue that must be followed at this point in the procedure.

    Any tissue that remains after filling the second bulk archive jar may be discarded.

IV.E. Equipment Cleaning between Composite Samples

29. All of the homogenization equipment must be thoroughly cleaned between each composite sample. Once all of the fillets from the individual specimens in a given composite sample have been homogenized, disassemble the homogenization equipment (i.e., blender, grinder, or other device) and thoroughly **clean all surfaces and parts** that contact the sample. Similarly, **clean all knives, cutting boards, and other utensils used**. At a minimum:

    - Wash with a detergent solution (phosphate- and scent-free) and warm tap water
    - Rinse three times with warm tap water
    - Rinse three times with deionized (DI) water
    - Rinse with acetone
    - Rinse three times with DI water
    - Rinse with (not soak in) 5% nitric acid
    - Rinse three times with DI water
    - Allow the components to air dry

30. Reassemble the homogenization equipment and proceed with homogenization of the next sample in the batch (e.g., begin with Step 6 above).

IV.F. Lipid Determination on Every Homogenized Composite Sample

The procedures for determining the lipid content of every fillet composite are described in Step 31 below. (Additional lipid determinations are required for one sample in every preparation batch, as described in Steps 36 and 37.)

31. Use the 5 to 10 g of homogenized tissue collected in Step 27 to determine the lipid content of the sample. Extract the aliquot using SW-846 Method 9071B. Determine the lipid content of that aliquot and record it in units of percent (i.e., grams of lipid per gram of tissue x 100), and provide the results to CSC by email, as described in Section IV.H. These results may be used by the laboratories conducting the other analyses to lipid-normalize their results.

IV.G. Quality Control (QC) Procedures

The project-specific QC procedures include preparation and testing of equipment rinsate samples and homogeneity testing, using lipids as a surrogate. The QC procedures are performed in two distinct phases: (1) as part of an initial demonstration of capabilities after the kickoff meeting and workshop with EPA, and (2) during normal operations.

Initial demonstration of capabilities: After the kickoff meeting and workshop, the sample preparation laboratory staff will prepare three test fish samples provided by Tetra Tech. Each test sample will consist of a single large fish which will be processed separately. Each of these test samples will be carried through the entire sample preparation and aliquoting procedures separately. The resulting sample aliquots will not be distributed to other laboratories at this time, but stored frozen. In between processing each individual fish sample, the sample preparation laboratory staff will clean all of the sample preparation equipment as described in Step 29 above. After each cleaning, the sample preparation laboratory staff will prepare the entire series of equipment rinsates and solvent blanks described in Step 32 below.

The sample preparation laboratory also will collect three lipid aliquots from each sample prepared during the initial demonstration and use them for triplicate determinations of lipids, as described in Step 36 below.

The results of the analyses of the rinsates and the homogeneity testing (three sets each) will be submitted to CSC for review. The sample preparation laboratory may **not** begin 2013-2014 NRSA sample preparation until CSC and EPA determine that the sample preparation laboratory has successfully demonstrated proficiency in meeting QC requirements for equipment cleaning and tissue homogenization.

Normal Operations: During normal sample preparation efforts, the sample preparation laboratory will prepare one set of rinsate samples and will conduct one set of triplicate lipid determinations per batch of 20 composite fish samples, as described in Steps 32 to 37, below. The batch-specific rinsate and homogeneity results will be reviewed by CSC and EPA. The sample preparation laboratory may continue to process up to one additional batch of 20 samples (based on sample preparation instructions provided by CSC) during that review process. However, the sample preparation laboratory may **not** continue beyond that next batch of samples until receiving notification from CSC that review of the prior batch rinsate and homogeneity test results is complete and the results were deemed satisfactory.

Thus, continued sample processing is dependent on both the quality of the sample preparation laboratory's efforts and on the timeliness of their delivery of QC results.

Rinsate and Blank Sample Production

32. Prior to reassembling the homogenization equipment (Step 30) between each of the samples processed during the initial demonstration of capabilities, and once per batch during normal operations, prepare three rinsate samples, as follows:

    - Prepare a **hexane rinsate sample** by pouring a 100-mL portion of pesticide-grade hexane over all parts of homogenization equipment, including the cutting boards and knives, and collect it in a clean glass container. Place an additional 100-mL aliquot of clean hexane in a similar glass container for use as a solvent blank. Allow the solvent to evaporate from the equipment. This rinsate and solvent blank will be analyzed for selected PCBs and PBDEs. Label, store, and analyze the PCB/PBDE rinsate and blank as described in Step 34.

    - Once the hexane has evaporated, prepare the **first DI water rinsate** using 250 mL of DI water. Collect the DI water rinsate in a clean glass or HDPE container. Place a second aliquot of DI water in a separate similar clean container for use as a blank. Acidify these two samples to pH < 2 with nitric acid. These rinsate and blank samples will be analyzed for mercury as described in Step 35.

    - Prepare the **second DI water rinsate** using an additional 250 mL of DI water. Collect this rinsate in a clean glass container **with a non-PTFE lid liner**. Place a second aliquot of DI water in a separate similar clean glass container for use as a blank. This rinsate and blank will be analyzed for PFCs by a laboratory to be determined later, thus the non-PTFE lid liners are essential. CSC will provide the sample preparation laboratory with the PFC laboratory name and shipping information as soon as it is available. Label and store these PFC rinsates and blanks as described in Step 33.

    Note: In order to minimize the number of project samples that might be affected by cross contamination, collect the normal rinsate samples on the first day that samples in a batch of 20 are processed. Ideally, the laboratory will vary the point at which the rinsates are collected on that first day over the course of the project (e.g., between the 1st and 2nd samples for one batch, the 2nd and 3rd samples for another batch, etc.).

33. Label each container as either “rinsate - [insert name of solvent]” or “blank - [insert name of solvent],” and include the date it was prepared (mm/dd/yyyy), the analysis type (Hg, PFCs, PCBs/PBDEs), and the preparation batch identifier. Store the rinsates and blanks cold (<6 ºC).

Rinsate Analyses

34. As part of the initial demonstration of capabilities, the sample preparation laboratory will analyze three sets of hexane rinsate and blank samples for PCBs/PBDEs (e.g., one set prepared after each tissue sample prepared during the initial demonstration process) using EPA Methods 1668A and 1614, respectively. Those methods will require concentration of the hexane to a final volume of 0.5 mL, and analysis by GC and high resolution mass spectrometry, in order to identify the PCB/PBDE congeners of interest. During normal operations, the sample preparation laboratory will analyze one set of the hexane rinsate and blank samples per batch. (The PCB analyses will be conducted by Cape Fear Analytical and the PBDE analyses by Vista Analytical Laboratories, both under contract to Microbac.)

35. As part of the initial demonstration of capabilities, the sample preparation laboratory will analyze three sets of DI water rinsate and blank samples for mercury using EPA Method 245.1, a cold-vapor atomic absorption procedure (e.g., one set prepared after each tissue sample prepared during the initial demonstration process). During normal operations, the sample preparation laboratory will analyze one set of the DI water rinsate and blank samples per batch for mercury.

Corrective Actions for Rinsates

CSC will evaluate the rinsate results based on the mass of each analyte detected, and assuming that all of the apparent contamination could be transferred to a nominal 536-g mass of homogenized tissue. Results for mercury or any PCBs/PBDEs above the anticipated reporting limits for these analytes in tissue samples may be cause for corrective actions by the sample preparation laboratory. Such corrective actions may include revisions to the sample preparation laboratory's equipment cleaning procedures, followed by a successful demonstration of the revised cleaning procedures through preparation and analysis of additional rinsate samples.

Lipid Determination to Confirm Homogeneity

36. For each of the samples processed during the initial demonstration of capabilities, and for one sample in every batch of 20 composite samples prepared during normal operations, the sample preparation laboratory will conduct triplicate analyses of the lipid content of samples to confirm that the samples are homogeneous.

    As with the collection of rinsate samples, the homogeneity testing must be performed on the first day on which samples in a batch of 20 are processed. However, the sample chosen for homogeneity testing must be one that yields enough tissue mass to support the added mass needed for triplicate lipid aliquots (15 to 30 g). Therefore, unless otherwise directed by CSC for a particular batch of samples, the sample preparation laboratory will select one sample processed on the first day of every batch that will provide well over 536 g of total tissue mass.

    From that sample, remove three 5- to 10-g aliquots of tissue before filling the archive sample containers. Place these three aliquots in clean glass or plastic containers of suitable size and label each with the site ID, sample number, and an aliquot identifier of the laboratory's choice. Transfer the lipid aliquot to the appropriate staff performing the lipid determination.

37. From the lipid results, calculate the mean lipid content (in percent), the standard deviation (SD), and the relative standard deviation (RSD) using the formulae below, or the corresponding functions in Excel.

$$\text{mean } \%\text{lipids} = \frac{\sum_{i=1}^{3} (\%\text{lipids})_i}{3}$$

$$\text{SD} = \sqrt{\frac{\sum_{i=1}^{3} (\%\text{lipids}_i - \text{mean lipids})^2}{2}}$$

$$\text{RSD} = \frac{\text{SD}}{\text{mean}}$$

    If the RSD of the triplicate results is less than or equal to 15%, then the homogenization effort is judged to be sufficient for all samples in that preparation batch. For this sample analyzed in triplicate, the mean lipid content will be the value reported for that sample, following the requirements described in Step 31.

Corrective Actions for Homogeneity

If the RSD is greater than 15%, then corrective action is required for all samples in that preparation batch. Corrective actions will be determined by CSC in direct consultation with the laboratory and EPA, but the default corrective action consists of regrinding all of the aliquots from each composite sample in the affected batch until the RSD criterion is met.

This may entail retrieving all sample aliquots (see Table 1) from the freezer, allowing them to partially thaw, and homogenizing them again, beginning at Step 20. In these instances, all of the equipment cleaning procedures will be repeated between each composite sample, new lipids results will be determined for each composite, and a new homogenization QC determination (triplicate lipids on one sample per batch) will be performed. New sample containers will be required for any rehomogenized samples.

IV.H. Reporting Requirements

38. The sample preparation laboratory will prepare a weekly progress report to document the status of fish preparation activities and forward the report electronically to CSC. The format of the weekly progress report will be as an Excel spreadsheet. For each fillet composite processed or plug sample collected during that period, include at least the following information in the report:

    - site identification number,
    - sample identification number,
    - specimen numbers of the fish homogenized for the fillet composite,
    - specimen numbers of the fish from which the plug samples were collected,
    - common name for the fish species (provided to the laboratory in the instructions from EPA),
    - field-determined lengths and lab-determined weights of individual specimens that were filleted and homogenized,
    - field-determined lengths and lab-determined weights of individual specimens from which the plug samples were collected,
    - total composite sample (i.e., homogenate) weight (to the nearest gram),
    - total plug sample weight (to the nearest 0.1 gram),
    - analysis type (e.g., mercury, PFCs, PCBs, PBDEs, archive sample, etc.),
    - aliquot weight (to the nearest 0.5 gram),
    - preparation batch ID,
    - preparation date (e.g., mm/dd/yyyy),
    - QC sample identifiers associated with the batch of fillet composite samples,
    - lipid results for each fillet composite sample, and
    - airbill numbers for all sample shipments that week (these may include samples prepared during previous weeks), even though this information was transmitted to CSC at the time of shipment.

    (Much of the sample-specific information above will be provided to the sample preparation laboratory electronically in the sample processing instructions from CSC.)

    The weekly report will be due by COB Monday, or as agreed to in writing by CSC after consultation with the laboratory in the cases of holidays, and will document sample preparation progress for the previous week.

    In addition, the laboratory must report the results of the rinsate analyses for mercury, PCBs, and PBDEs and the triplicate lipid results associated with the sample batch. Those results **must** be reported to CSC as soon after the analyses as practical to facilitate CSC's timely review and to minimize delays in receiving instructions to process future batches.

Note: As specified in the QC section of this document, the sample preparation laboratory may **not** continue beyond the next batch of samples until receiving notification from CSC that review of the prior batch rinsate and homogeneity test results is complete and the results were deemed satisfactory.

IV.I. Shipping Samples

39. **No samples may be shipped until CSC and EPA have reviewed the sample homogeneity testing and rinsate results.** CSC will notify the sample preparation laboratory by email when specific samples may be shipped, and to whom.

    When shipping batches of pre-frozen fillet tissue aliquots, keep the individual containers bagged in the food-grade plastic freezer bags. Place these bags in a cooler with adequate space for the tissue containers, packing materials, and dry ice. (CSC may provide suitable coolers from existing stocks.) Secure each of the tissue containers with packing materials (e.g., bubble wrap or foam) before adding the dry ice. Place a modest layer of newspaper on top of the containers before adding the dry ice, as this can prevent cracking the lids. A single "section" of the local newspaper will usually suffice.

    The amount of dry ice required for shipping will depend on the number of fillet tissue samples in the cooler and the time of year. It should be an adequate supply to keep the tissue samples frozen for 48 hours (i.e., a minimum of 25 pounds of dry ice per cooler for up to 10 pounds of fillet tissue samples).

    Shipments of plug samples for mercury analyses may use smaller coolers and other forms of packing materials (e.g., foam blocks with pre-drilled holes) appropriate for the scintillation vials, but must be shipped on dry ice as well.

    Record the samples contained in the cooler on a shipping form provided by CSC and place the form in a plastic bag taped to the inside lid of the cooler. (CSC will provide separate forms for plug samples and homogenized fillet samples.) Secure the outside of the cooler with sealing tape, address it to the sample recipient identified by CSC, and attach a dry ice (dangerous goods) label. Ship the cooler via an overnight express carrier on a date that will allow delivery of the cooler to the analytical laboratory on a normal business day (e.g., **no Saturday deliveries and no deliveries on U.S. Federal holidays without express permission from CSC**). Provide the air bill number for each shipment to CSC via email on the day that the shipment occurs. **CSC will provide the sample preparation laboratory with a third-party FedEx account to which each shipment will be billed.**

CSC Contact Information

Primary CSC Contact	**Alternative CSC Contact**
Harry McCarty	Lynn Walters
6361 Walker Lane	6361 Walker Lane
Alexandria, VA 22310	Alexandria, VA 22310
703-461-2392	703-461-2060
hmccarty@csc.com	lwalters3@csc.com

V. Deliverables

Item	Deliverable Description	Mechanism	Schedule
1	Kickoff meeting and workshop	--	July 16, 2013
2	Initial demonstration of capabilities	--	Begin within 3 days of receipt of test samples from Tetra Tech

Item	Deliverable Description	Mechanism	Schedule
3	Results of the initial demonstration, to include three sets of rinsate and solvent blank results for mercury and PBDEs, plus three sets of triplicate lipid determinations	Email	Close of Business (COB) of the day after results are generated
4	Confirmation of receipt of sample processing instructions, identifying any specific sample discrepancies	Email	COB on the day of receipt
5	Notification of samples that do not yield at least 536 g of homogenized fillet tissue	Email/phone	Immediately upon discovery during sample preparation
6	First completed batch of 20 homogenized fillet samples and 20 plug samples, ready for shipment at CSC's direction (i.e., sample turnaround time)	--	**21 calendar days** from receipt of sample processing instructions from CSC
7	Each subsequent batch of 20 homogenized fillet samples and 20 plug samples	--	**14 calendar days** from completion of the previous batch, or **14 days** from receipt of sample processing instructions from CSC, whichever is longer
8	Mercury and PCB/PBDE results for rinsates and solvent blanks and lipid RSD results	Email/phone	COB of the day after results are generated
9	Weekly status report	Email	COB Monday of each week
10	Homogenized sample shipments	FedEx overnight	Within **3 working days** of receipt of shipping information from CSC
11	Shipping information (airbills, shipping forms, etc.) for tissue or rinsate samples	Email	COB on day samples ship to other labs
12	Copies of all bench sheets, sample preparation records, and other project records	Hard copy or PDF	As directed by CSC after the completion of the project

ATTACHMENT 1
ANALYSES OF RINSATES AND BLANKS FOR MERCURY AND PCBs/PBDEs

This attachment describes the analyses of rinsate samples and blanks generated during the composite fish sample preparation process. The results of those analyses are important in demonstrating that the sample preparation laboratory's equipment cleaning procedures are effective at preventing cross-contamination between fish tissue samples.

A. EQUIPMENT AND MATERIALS:

- Mercury analyzer suitable for aqueous samples using cold-vapor atomic absorption (CVAA) instruments compatible with EPA Method 245. Must be capable of achieving an MDL of approximately 1 µg/L.
- Gas chromatograph with a high resolution mass spectrometric detector (GC/HRMS) suitable for analysis of PCB and PBDE congeners via EPA Methods 1668A and 1614.
- Solvent concentration equipment suitable for reducing hexane rinsates to final volumes of 0.5 to 10 mL.
- A PCB standard solution containing at least the following PCB congeners: **52, 66, 105, 118, 141, 146, 170, 174, 177, and 187**, to be used to establish retention times and perform calibration of the GC/HRMS. (Additional congeners can be included by the laboratory. These congeners represent those that EPA has found frequently, at relatively high concentrations, in other fish tissue studies.)
- A PBDE standard solution containing at least the following PBDE congeners: **47, 49, 66, 99, 100, 153, 154, and 155**, to be used to establish retention times and perform calibration of the GC/HRMS. (Additional congeners can be included by the laboratory. These congeners represent those that EPA has found frequently, at relatively high concentrations, in other fish tissue studies.)
- Assorted glassware, syringes, etc.

B. RINSATE AND BLANK ANALYSES

During the initial demonstration of capabilities, the laboratory will prepare three sets of rinsate samples, i.e., one set after each fish prepared as part of that demonstration. Each set of rinsate samples will include:

- Two de-ionized water (DI) rinsate samples and two DI water blanks sample for analysis of mercury and for analysis of PFCs.
- One hexane rinsate sample and one hexane blank sample for analysis of PCBs and PBDEs (e.g., one rinsate may be analyzed for both groups of contaminants).

During normal sample preparation efforts, the laboratory will prepare rinsates at a frequency of one set for each batch of 20 fish tissue samples prepared. Up to 25 sets of rinsates are anticipated.

The laboratory will digest and analyze the mercury rinsates and blanks by CVAA. The laboratory will concentrate the PCB/PBDE rinsates and blanks to a final volume of 1 mL and analyze the concentrated samples by GC/HRMS. For each analysis, the laboratory will determine the mass of each analyte (mercury, PCB congener, or PBDE congener) in the total volume of each rinsate or blank sample, rather than the concentration of each analyte.

The laboratory will either perform a method detection limit (MDL) study for mercury in aqueous samples, or use existing aqueous MDL data for the CVAA instrument employed. The laboratory must be able to achieve an MDL of approximately 1 µg/L. Mercury results will be reported down to the mass equivalent to the mass at the method detection limit (MDL) for aqueous samples.

Because the PCB/PBDE rinsates are not aqueous samples that are extracted, a traditional MDL study for aqueous samples does not apply. Therefore, the laboratory must perform an instrument detection limit (IDL) study before beginning any rinsate analyses. The IDL study will consist of analyzing 7 low-level standards containing the PCBs and PBDEs listed above, determining the standard deviation of results for each PCB and PBDE across all 7 analyses, and multiplying the standard deviation times 3.143, the Student's t-value for 7 replicates. The laboratory must achieve an IDL on the order of 0.5 ng/mL, for a 1-mL final volume.

PCB congeners and PBDE congeners will be identified based on the requirements of EPA Methods 1668A and 1614, respectively. PCB and PBDE results in the rinsates and blanks will be reported down to the mass equivalent to the IDL.

The rinsates for PFCs will not be analyzed by the laboratory, but will be held by the sample preparation laboratory.

C. QUALITY CONTROL

The quality control (QC) procedures required for the rinsate analyses include:

- MDL or IDL studies, as described above
- Instrument calibration (see Methods 245.1, 1668A, and 1614 for procedures and acceptance criteria)
- Instrument blanks for mercury, PCB, and PBDE analyses
- Calibration verification (once per analysis batch) for mercury, PCB, and PBDE analyses
- Laboratory control sample (LCS) once per analysis batch, for mercury only

The MDL and IDL results will be reviewed by CSC as soon as they become available, and the laboratory will not be authorized to prepare additional fish tissue samples until that review is complete and the results are acceptable.

The matrix for the mercury rinsates is reagent water, which should not adversely affect method performance. Therefore, matrix spike samples are not required for mercury.

Because the PCB/PBDE rinsates do not involve extraction of an environmental matrix, matrix spike samples are not applicable. Likewise, laboratory control samples are not applicable to PCBs and PBDEs.

The instrument blanks for mercury, PCBs, and PBDEs take the place of a traditional method blank that would be extracted along with environmental samples.

D. DELIVERABLES

Summary data from the rinsate analyses are to be delivered to CSC in an Excel file. That file must contain the following information, at a minimum:

- Batch ID - to be established by the laboratory, but a simple approach would be to number or letter each sample batch (e.g., A to H, or 1 to 8). The batch ID for the rinsates prepared during the initial demonstration results may be reported as "QA study."
- Sample ID - as described in the instructions for preparing the rinsates
- Lab sample ID - unique internal identifier used by the laboratory, if any
- Prep date - Date (MM/DD/YYYY) on which the rinsate or solvent blank was prepared
- Analysis type - "Mercury," "PCB, "or "PBDE" (or "PCB/PBDE" if both types of analytes are analyzed together)
- Analysis date - Date (MM/DD/YYYY) on which the rinsate or solvent blank was analyzed
- Analyte name - PCB and PBDE congeners may be abbreviated as PCB-066, PBDE-047, etc.

- Mass of analyte found - in micrograms for mercury, and either micrograms or nanograms for the PCBs and PBDEs, provided that the reporting units for PCBs and PBDEs are consistent throughout the effort
- Lab qualifiers - as needed to describe any analytical concerns. A complete list of the qualifiers and their meanings must be included with each data submission (e.g., in a separate tab on the Excel file).
- Reporting limit for each analyte - in the same mass units used for the results
- Instrument calibration data - Submit as a separate tab in the Excel file. Must include results for the initial calibrations for mercury, PCBs, and PBDEs, as well as any relevant calibration verifications associated with the analyses. Include calibration equations (e.g., regressions) and metrics (e.g., correlation coefficient or calibration factor).

Separate Excel files may be provided for each type of analysis (mercury, PCBs, and PBDEs), at the laboratory's discretion. Raw data supporting each analysis (e.g., chromatograms or instrument printouts) must be retained by the laboratory and made available to CSC when requested, at no additional cost. If requested, raw data may be submitted in hard copy, or as a PDF file.

APPENDIX E

GLEC Fish Information Summary Tables

Table 1. **Fish Tissue Processing Field Data**
Boulder, Thomson, and Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Reservoir	Description	# of Individuals	Field Weight 1	Field Weight 2	Field Weight 3	Field Weight 4	Field Weight 5	Total Mass (mg)	Perform Sex ID?	Perform Otolith?	SexID 1	SexID 2	SexID 3	SexID 4	SexID 5	Processed Tissue Shipped to Test America
5044		Boulder	Black Crappie A	6						116	No	No						11/7/16
5035		Boulder	Rock Bass A	9						368	No	No						11/7/16
5033		Boulder	Shiners A	Many						152	No	No						11/7/16
5032		Boulder	Shiners B	Many						152	No	No						11/7/16
5045		Boulder	Shiners C	Many						163	No	No						11/7/16
5041	Dupe	Boulder	Walleye A	3	664	578	577			1,819	1,2,3	1,2,3	M	M	M			11/7/16
5042		Boulder	Walleye B	3	131	212	81			424	1,2,3	1,2,3	M	M	M			11/7/16
5043		Boulder	Walleye C	5	58	84	83	72	73	370	1,2,3,4,5	1,2,3,4,5	IND	IND	IND	IND	IND	11/7/16
5027	MSD	Boulder	White Sucker A	3	221	1,007	619			1,847	1,2,3	1,2,3	IND	M	M			11/7/16
5029		Boulder	White Sucker B	3	622	1,230	1,150			3,002	1,2,3	1,2,3	M	M	M			11/7/16
5028		Boulder	White Sucker C	3	1,779	1,285	1,326			4,390	1,2,3	1,2,3	F	F	F			11/7/16
5031		Boulder	Yellow Perch A	13						378	No	No						11/7/16
5030		Boulder	Yellow Perch B	13						311	No	No						11/7/16
5034		Boulder	Yellow Perch C	12						304	No	No						11/7/16
5006		Thomson	North Pike A	3	275	178	186			639	1,2,3	1,2,3	F	F	F			11/14/16
5009		Thomson	Rock Bass A	3	50	58	142			250	1,2,3	1,2,3	M	F	M			11/14/16
5010		Thomson	Rock Bass B	8						150	No	No						11/7/16
5003		Thomson	Small Mouth Bass A	10						394	No	No						11/14/16
5036		Thomson	Small Mouth Bass B	3	763	768	714			2,245	1,2,3	1,2,3	F	M	F			11/14/16
5004		Thomson	Small Mouth Bass C	3	1,090	1,012	936			3,038	1,2,3	1,2,3	F	M	M			11/14/16
5038		Thomson	Small Mouth Bass D	9						358	No	No						11/7/16
5007	MSD	Thomson	Walleye A	3	261	360	311			932	1,2,3	1,2,3	M	M	M			11/7/16
5011	Dupe	Thomson	White Sucker A	3	1,204	1,144	1,064			3,412	1,2,3	1,2,3	F	F	F			11/14/16
5015		Thomson	White Sucker B	3	965	820	923			2,708	1,2,3	1,2,3	F	F	F			11/14/16
5014		Thomson	White Sucker C	3	1,070	618	633			2,321	1,2,3	1,2,3	F	F	F			11/14/16
5005	Dupe	Thomson	Yellow Perch A	3	268	352	239			859	1,2,3	1,2,3	F	F	F			11/14/16
5008		Thomson	Yellow Perch B	4	210	211	188	172		781	1,2,3,4	1,2,3,4	F	M	M	F		11/14/16
5019	Dupe	Scanlon	Northern Pike A	2	340	487				827	1,2	1,2	F	F				11/14/16
5024		Scanlon	Shiners A	Many						61	No	No						11/15/16
5001		Scanlon	Small Mouth Bass A	3	547	627	547			1,721	1,2,3	1,2,3	M	F	M			11/14/16
5002	Dupe	Scanlon	Small Mouth Bass B	3	473	587	278			1,338	1,2,3	1,2,3	F	M	F			11/15/16
5021		Scanlon	Small Mouth Bass C	3	213	371	274			858	1,2,3	1,2,3	F	F	M			11/14/16
5023		Scanlon	Walleye A	3	237	215	168			620	1,2,3	1,2,3	M	M	M			11/14/16
5022		Scanlon	White Sucker A	3	1,015	736	792			2,543	1,2,3	1,2,3	F	F	F			11/14/16
5017		Scanlon	White Sucker B	3	844	952	324			2,120	1,2,3	1,2,3	F	F	M			11/14/16
5016		Scanlon	White Sucker C	3	791	781	817			2,389	1,2,3	1,2,3	F	F	F			11/14/16
5025	MSD	Scanlon	Yellow Perch A	4	166	136	124	74		500	1,2,3,4	1,2,3,4	F	F	F	M		11/15/16
5020		Scanlon	Yellow Perch B	3	98	76	141			315	1,2,3	1,2,3	M	M	F			11/15/16
5018		Scanlon	Yellow Perch C	19						432	No	No						11/14/16

For greyed-out samples, see small species Tables 5, 6, and 7 for individual mass and length
Dupe - Send two distinct samples from the same homoginization to Test America for analysis with separate IDs
MSD - Send twice as much sample from the same homoginization to Test America for Test America laboratory Quality Assurance/Quality Control requirements

Table 2. **Fish Tissue Processing Laboratory Data**
Scanlon Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Description	# of Individuals	Metric	1	2	3	4	SexID 1	SexID 2	SexID 3	SexID 4	Average	+10% of Average	-10% of Average	Samples all within 10% of Average
5019	Dupe	Northern Pike A	2	Length (mm)	415	459			F	F			437	481	393	Yes
5019	Dupe	Northern Pike A	2	Mass (g)	343	481			F	F			412	453	371	No
5024		Shiners A	Many													
5001		Small Mouth Bass A	3	Length (mm)	343	338	341		M	F	M		341	375	307	Yes
5001		Small Mouth Bass B	3	Mass (g)	552	631	555		M	F	M		579	637	521	Yes
5002	Dupe	Small Mouth Bass B	3	Length (mm)	321	345	272		F	M	F		313	344	281	No
5002	Dupe	Small Mouth Bass C	3	Mass (g)	481	594	282		F	M	F		452	498	407	No
5021		Small Mouth Bass C	3	Length (mm)	250	289	265		F	F	M		268	295	241	Yes
5021		SMB C	3	Mass (g)	218	379	279		F	F	M		292	321	263	No
5023		Walleye A	3	Length (mm)	307	290	276		M	M	M		291	320	262	Yes
5023		Walleye A	3	Mass (g)	241	216	172		M	M	M		210	231	189	No
5022		White Sucker A	3	Length (mm)	436	389	395		F	F	F		407	447	366	Yes
5022		White Sucker A	3	Mass (g)	1,016	736	796		F	F	F		849	934	764	No
5017		White Sucker B	3	Length (mm)	420	432	306		F	F	M		386	425	347	No
5017		White Sucker B	3	Mass (g)	826	939	320		F	F	M		695	765	626	No
5016		White Sucker C	3	Length (mm)	415	410	414		F	F	F		413	454	372	Yes
5016		White Sucker C	3	Mass (g)	785	743	799		F	F	F		776	853	698	Yes
5025	MSD	Yellow Perch A	4	Length (mm)	232	219	214	187	F	F	F	M	213	234	192	No
5025	MSD	Yellow Perch A	4	Mass (g)	170	137	125	75	F	F	F	M	127	139	114	No
5020		Yellow Perch B	3	Length (mm)	201	186	219		M	M	F		202	222	182	Yes
5020		Yellow Perch B	3	Mass (g)	100	87	142		M	M	F		110	121	99	No
5018		Yellow Perch C	19													

For greyed-out samples, see small species Table 5 for individual mass and length
Dupe - Send two distinct samples from the same homoginization to Test America for analysis with separate IDs
MSD - Send twice as much sample from the same homoginization to Test America for Test America laboratory Quality Assurance/Quality Control requirements

Table 3. Fish Tissue Processing Laboratory Data
Thomson Reservoir
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Description	# of Individuals	Metric	1	2	3	4	SexID 1	SexID 2	SexID 3	SexID 4	Average	+10% of Average	-10% of Average	Samples all within 10% of Average
5006		North Pike A	3	Length (mm)	371	347	339		F	F	F		352	388	317	Yes
5006		North Pike A	3	Mass (g)	278	179	183		F	F	F		213	235	192	No
5009		Rock Bass A	3	Length (mm)	136	145	192		M	F	M		158	173	142	No
5009		Rock Bass A	3	Mass (g)	50	58	132		M	F	M		80	88	72	No
5010		Rock Bass B	8													
5003		Small Mouth Bass A	10													
5036		Small Mouth Bass B	3	Length (mm)	366	311	363		F	M	F		347	381	312	No
5036		Small Mouth Bass B	3	Mass (g)	763	768	714		F	M	F		748	823	674	Yes
5004		Small Mouth Bass C	3	Length (mm)	393	348	393		F	M	M		378	416	340	Yes
5004		Small Mouth Bass C	3	Mass (g)	1,083	1,001	924		F	M	M		1,003	1,103	902	Yes
5038		Small Mouth Bass D	9													
5007	MSD	Walleye A	3	Length (mm)	317	332	330		M	M	M		326	359	294	Yes
5007	MSD	Walleye A	3	Mass (g)	261	360	309		M	M	M		310	341	279	No
5011	Dupe	White Sucker A	3	Length (mm)	468	468	480		F	F	F		472	519	425	Yes
5011	Dupe	White Sucker A	3	Mass (g)	1,023	1,108	1,169		F	F	F		1,100	1,210	990	Yes
5015		White Sucker B	3	Length (mm)	419	412	438		F	F	F		423	465	381	Yes
5015		White Sucker B	3	Mass (g)	949	794	892		F	F	F		878	966	791	Yes
5014		White Sucker C	3	Length (mm)	431	404	392		F	F	F		409	450	368	Yes
5014		White Sucker C	3	Mass (g)	1,030	591	620		F	F	F		747	822	672	No
5005	Dupe	Yellow Perch A	3	Length (mm)	271	276	259		F	F	F		269	296	242	Yes
5005	Dupe	Yellow Perch A	3	Mass (g)	267	345	238		F	F	F		283	312	255	No
5008		Yellow Perch B	4	Length (mm)	216	215	228	230	F	M	M	F	222	244	200	Yes
5008		Yellow Perch B	4	Mass (g)	138	138	190	168	F	M	M	F	159	174	143	No

For greyed-out samples, see small species Table 6 for individual mass and length
Dupe - Send two distinct samples from the same homoginization to Test America for analysis with separate IDs
MSD - Send twice as much sample from the same homoginization to Test America for Test America laboratory Quality Assurance/Quality Control requirements

Table 4. **Fish Tissue Processing Laboratory Data**
Boulder Reservoirs
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	QC	Description	# of Individuals	Metric	1	2	3	4	5	SexID 1	SexID 2	SexID 3	SexID 4	SexID 5	Average	+10% of Average	-10% of Average	Samples all within 10% of Average
5044		Black Crappie A	6															
5035		Rock Bass A	9															
5033		Shiners A	Many															
5032		Shiners B	Many															
5045		Shiners C	Many															
5041	Dupe	Walleye A	3	Length (mm)	418	394	393			M	M	M			402	442	362	Yes
5041	Dupe	Walleye A	3	Mass (g)	671	599	591			M	M	M			620	682	558	Yes
5042		Walleye B	3	Length (mm)	248	288	219			M	M	M			252	277	227	No
5042		Walleye B	3	Mass (g)	127	213	79			M	M	M			140	154	126	No
5043		Walleye C	5	Length (mm)	204	213	220	200	211	IND	IND	IND	IND	IND	210	231	189	Yes
5043		Walleye C	5	Mass (g)	56	83	82	72	73	IND	IND	IND	IND	IND	73	81	66	No
5027	MSD	White Sucker A	3	Length (mm)	272	399	347			IND	M	M			339	373	305	No
5027	MSD	White Sucker A	3	Mass (g)	221	1,013	615			IND	M	M			616	678	555	No
5029		White Sucker B	3	Length (mm)	351	464	455			M	M	M			423	466	381	No
5029		White Sucker B	3	Mass (g)	616	1,232	1,147			M	M	M			998	1,098	899	No
5028		White Sucker C	3	Length (mm)	502	468	457			F	F	F			476	523	428	Yes
5028		White Sucker C	3	Mass (g)	1,884	1,368	1,326			F	F	F			1,526	1,679	1,373	No
5031		Yellow Perch A	13															
5030		Yellow Perch B	13															
5034		Yellow Perch C	12															

For greyed-out samples, see small species Table 7 for individual mass and length
Dupe - Send two distinct samples from the same homoginization to Test America for analysis with separate IDs
MSD - Send twice as much sample from the same homoginization to Test America for Test America laboratory Quality Assurance/Quality Control requirements

Table 5. Fish Tissue Processing Laboratory Data (continued)
Scanlon Reservoirs
Duluth, Minnesota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Species	Individual	Mass (g)	Length (mm)
5024	many	Shiners A	Min	<1	15
			Max	33	120
5018	19	Yellow Perch C	1	132	216
			2	82	178
			3	43	153
			4	25	132
			5	20	118
			6	16	111
			7	15	109
			8	14	109
			9	18	116
			10	14	103
			11	12	103
			12	12	103
			13	10	97
			14	3	68
			15	2	66
			16	3	65
			17	3	70
			18	3	70
			19	2	59
			Average	23	108
			+10% Average	25	118
			-10% Average	20	97
		Samples all within 10% of Average		No	No

Table 6. Fish Tissue Processing Laboratory Data (continued)
Thomson Reservoir
Duluth, Minnesota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Species	Individual	Mass (g)	Length (mm)
5010	8	Rock Bass B	1	20	97
			2	14	91
			3	19	97
			4	19	101
			5	23	107
			6	22	104
			7	18	95
			8	18	95
			Average	19	98
			+10% Average	21	108
			-10% Average	17	89
		Samples all within 10% of Average		No	Yes
5003	10	Small Mouth Bass A	1	30	132
			2	30	135
			3	29	133
			4	35	142
			5	39	141
			6	41	138
			7	40	139
			8	47	151
			9	38	140
			10	64	161
			Average	39	141
			+10% Average	43	155
			-10% Average	35	127
		Samples all within 10% of Average		No	No
5038	9	Small Mouth Bass D	1	57	156
			2	55	155
			3	63	169
			4	50	149
			5	39	141
			6	35	133
			7	28	124
			8	19	110
			9	16	100
			Average	40	137
			+10% Average	44	151
			-10% Average	36	124
		Samples all within 10% of Average		No	No

Table 7. Fish Tissue Processing Laboratory Data (continued)
Boulder
Duluth, Minnesota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Species	Individual	Mass (g)	Length (mm)
5044	6	Black Crappie A	1	18	95
			2	14	86
			3	18	94
			4	21	99
			5	26	103
			6	25	104
			Average	20	97
			+10% Average	22	107
			-10% Average	18	87
		Samples all within 10% of Average		No	No
5035	9	Rock Bass A	1	29	108
			2	36	117
			3	23	102
			4	20	96
			5	22	101
			6	28	110
			7	24	103
			8	18	94
			9	19	100
			Average	24	103
			+10% Average	27	114
			-10% Average	22	93
		Samples all within 10% of Average		No	No
5033	many	Shiners A	Min	<1	18
			Max	24	125
5032	many	Shiners B	Min	<1	39
			Max	12	110
5045	many	Shiners C	Min	<1	9
			Max	32	101
5031	13	Yellow Perch A	1	77	181
			2	64	172
			3	48	156
			4	65	169
			5	16	109
			6	14	107
			7	15	111
			8	14	108
			9	15	106
			10	17	116
			11	15	111
			12	10	97
			13	12	103
			Average	29	127
			+10% Average	32	139
			-10% Average	26	114
		Samples all within 10% of Average		No	No

Table 7. Fish Tissue Processing Laboratory Data (continued)
Boulder
Duluth, Minnessota
U.S. Army Corps of Engineers
GLEC Project Number: 5148

GLEC ID	# of Individuals	Species	Individual	Mass (g)	Length (mm)
5030	13	Yellow Perch B	1	12	102
			2	46	162
			3	12	107
			4	35	143
			5	18	110
			6	11	96
			7	12	104
			8	11	100
			9	17	109
			10	12	99
			11	14	104
			12	41	147
			13	67	176
			Average	24	120
			+10% Average	26	132
			-10% Average	21	108
		Samples all within 10% of Average		No	No
5034	12	Yellow Perch C	1	11	99
			2	12	103
			3	12	104
			4	13	105
			5	14	108
			6	11	99
			7	13	103
			8	12	103
			9	45	154
			10	59	168
			11	47	146
			12	59	171
			Average	26	122
			+10% Average	28	134
			-10% Average	23	110
		Samples all within 10% of Average		No	No

APPENDIX F

EPA and MCPA Macroinvertebrate COCs and Instructions

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

N = 11 jars

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company:	Report To:	Attention:	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address:	Copy To: praymaker@baywest.com	Company Name:	Facility_Code: St Louis River Sed	
	nmcdonald@baywest.com	Address:	Facility_ID:	COC# SLR-GLEC-3
Email To:	Purchase Order No.:	Lab Quote Reference:	Subfacility_code:	
Phone:	Project Name: SLR Sediment AOCs	Lab Project Manager: Mailee Garton		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number:			

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins/furans (1613B)	Lipids	Mercury (7471B)	Methyl Mercury (1630)	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0 15	SO	G	3/12/15	1204														
1	BW16SR-001	BW16SR-001-M ✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
2	BW16SR-002	BW16SR-002-M ✓✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
3	BW16SR-002	BW16SR-002-D ✓✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
4	BW16SR-102	BW16SR-102-D	TS	G	9/19/16-10/6/16											X	X			Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
5	BW16SR-003	BW16SR-003-M ✓✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
6	BW16SR-103	BW16SR-103-M	TS	G	9/19/16 10/6/16													X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
7	BW16SR-005	BW16SR-005-D ✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
8	BW16SR-005	BW16SR-005-C ✓	TS	G	9/19/16-10/6/16											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
✓ 9	EPA16BRHD	EPA16 BRHDMCRS	TS	C	extra jars 4 homogenized tissue composite											X	X	X	X	Priority Order: 1 Dioxins/Lipids, 2 Hg, 3 MeHg
10	~~EPA16SR-HD~~	~~EPA16SR-HD-MCRS~~	TS	C	RLR													X	X	Priority Order: 1 Hg, 1 MeHg
11	~~EPA16-TR-HD~~	~~EPA16-TR-HD-MCRS~~	TS	C														X	X	Priority Order: 1 Hg, 1 MeHg
10	~~EPA16-TR-HD~~	~~EPA16-TR-HD-C~~	TS	C														X	X	Priority Order: 1 Hg, 1 MeHg
✓ 11	BW16SR-003	BW16-SR-003-D	TS	G	9/18/16-10/6/16											X	X	X	X	

BW16SR-102 is to be collected from BW16SR-002 as a DUP and BW16SR-103M is to be collected from BW16SR005M as a DUP.

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
	Amber A/BayWest	11/09/16	1041							

Sample name extension definitions:
M = Mayfly
D = Dragonfly
C = Crayfish
MCRS = Macrobenthos

SAMPLER NAME AND SIGNATURE	
PRINT Name of SAMPLER:	
SIGNATURE of SAMPLER:	DATE Signed (MM/DD/YY):

N = 5 of 19 jar

GLEC
Great Lakes Environmental Center

GREAT LAKES ENVIRONMENTAL CENTER, INC. (GLEC)

CHAIN OF CUSTODY RECORD

(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company: EPA Mid-Continent Ecol. Div.

Report Results To: Joel Hoffman

Address: 6201 Congdon Blvd, Duluth MN 55804

Phone: 218 529 5420 E-mail: hoffman.joel@epa.gov

Section II.

Project Name: St. Louis AOC

Project Number:

P.O.#:

Sampled by: initials
☐ GLEC ☑ Client EPA/MPCA

Section III. Sample Information at Collection

Section IV.

#	LAB USE GLEC ID#	Sample Identification	Sample Information Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers Type	Size	No.	Requested Processing/Analysis
1	16-BR-	HD-003	10-11-16	11:00	HD	Comp.	on ice			1	Dioxin/Hg 2.9 g
2	16-BR-	HD-001	10-11-16	12:05	HD	Comp.	on ice			1	" " 2.3 g
3	16-BR-	HD-002	10-11-16	11:55	HD	Comp.	on ice			1	" " 10.2 g
4	16-BR-	HD-004	10-11-16	10:40	HD	Comp.	on ice			1	" " 3.9 g
5	16-BR-	HD-005	10-11-16	10:10	HD	Comp	on ice			1	" " 5.1 g
6											

Client/Sampler Notes:

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Joel Hoffman EPA Duluth	11/15/16	12:00 PM	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY

Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice

Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT; W = SURFACE WATE; E = EFFLUENT; GW = GROUNDWATER; SL = SLUDGE; AO = AQUATIC ORGANISM

N = 8 of 19 jars

GLEC
Great Lakes Environmental Center

GREAT LAKES ENVIRONMENTAL CENTER, INC. (GLEC)

CHAIN OF CUSTODY RECORD
(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company: EPA Mid Continent Eco. Div
Report Results To: Jim LAZORCHAK
Address: 6201 Congdon BLVD, Duluth MN 55804
Phone: 513 569 7076 E-mail: lazorchak.Jim@EPA.GOV

Section II.

Project Name: ST Louis AOC
Project Number:
P.O.#:
Sampled by: initials ☐ GLEC ____ ☒ Client ____

Section III. Sample Information at Collection

Section IV.

#	LAB USE GLEC ID#	Sample Identification	Sample Information: Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers: Type	Size	No.	Requested Processing/Analysis
1	16-TR	HD-016 1of2	10/12/16		HD	"	ice			1	Dioxin/Furan 2.4g
2	16-TR	HD-016 2of2	"		HD	"	"	Craft Fish		1	" " 2.4g
3	16-TR	HD-017	"		"	"	"			1	" " 0.1g
4	16-TR	HD-013 1/2	"		"	"	"			1	" " 0.68gm
5	16-TR	HD-013 2/2	"		"	"	"	Craftfish		1	" " 1.2gm
6	16-TR	HD-008 1/2	"		"	"	"			1	" " 1.6 gm
	Client/Sampler Notes: 16_TR-008 2/2		"		"	"	"	Craftfish		1	" " 6.6 gm
	✓16-TR-018		"		"	"	"			1	" " 1.1 gm

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Joe HOFFMAN EPA Duluth	11/10/16	12:00pm	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY
Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice
Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT; W = SURFACE WATE; E = EFFLUENT; GW = GROUNDWATER; SL = SLUDGE; AO = AQUATIC ORGANISM

V = 6 of 19 jar

GLEC
Great Lakes Environmental Center

GREAT LAKES ENVIRONMENTAL CENTER, INC. (GLEC)

CHAIN OF CUSTODY RECORD
(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company:

Report Results To: Joel Hoffman/Jim Lazor

Address: 6201 Congdon Blvd, Duluth, MN 55804

Phone: 218-529-5420 E-mail: Hoffman.Joel@epa.gov

Section II.

Project Name: St Louis River

Project Number:

P.O.#:

Sampled by: initials ☐ GLEC JL ☐ Client EPA/MPCA

Section III. Sample Information at Collection

Section IV.

#	LAB USE GLEC ID#	Sample Identification	Sample Information Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers Type	Size	No.	Requested Processing/Analysis
1	16-SR	HD-007	10-13-16	9:30	HD	"	ice			1/1	Dioxin / Hg 0.7g
2	16-SR	HD-003	"	10:10	"	"	"			1/1	" " 1.4g
3	16-SR	HD-001	"	9:55	"	"	"			1/1	" " 1.0g
4	16-SR	HD-004	"	10:50	"	"	"			1/1	" " 0.5g
5	16SR	HD-005	"	10:35	"	"	"			1/2	" " 0.9g
6	16-SR	HD-005 composite	"	"	"	"	"			2/2	" " 0.4g

Client/Sampler Notes:

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Joel Hoffman EPA Duluth	11/10/16	12:30PM	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY

Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice

Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT; W = SURFACE WATE; E = EFFLUENT; GW = GROUNDWATER; SL = SLUDGE; AO = AQUATIC ORGANISM

SLR team,

I know last week there were a few emails going back and forth but wanted to make sure everyone was in agreement with the macroinvertebrates (and to make sure I understand). I have attached Mariah's recent spreadsheet and mine for reference. From my understanding of the discussions so far this is what I have:

1. 3 Mayfly samples from Scanlon (BW16SR-001-M to 003-M) plus a duplicate (BW16SR103-M) for %lipids, dioxin, MeHg, Total Hg
2. 2 Dragonfly samples from Scanlon (BW16SR-002 and 005) plus a duplicate (BW16SR-102) for %lipids, dioxin, MeHg, Total Hg
3. 1 Crawfish sample from Scanlon (BW16SR-005-C) for %lipids, dioxin, MeHg, Total Hg
4. 1 Macrobenthos from Boulder (EPA16-BR-HD-M) by combining all Boulder Reservoir jars for % lipids, dioxin, and total Hg
5. 1 Thomson crawfish (EPA16TR-HD-C) combined and ran for total Hg, and % lipids
6. 7 Lumbriculus: (4 from Thomson), (2 from Scanlon) and (1 from Boulder) for %lipids, dioxin, MeHg, Total Hg

Based on the summary above, please see the following questions:

1. According to my spreadsheet (macroninvertcollectionsummary), there are 4.5 g from EPA Scanlon Macrobenthos and 5.8 g EPA Thomson Macrobenthos. Is it possible and does anyone have any objections to at least running these samples for % lipids, Total Hg and/or MeHg?

2. I do not have it written in my notes, I apologize, but did the group come to the agreement that no snails should be analyzed?

3. If the group has finalized the testing numbers, Mariah, how many samples does that leave us with to plan for spring?

Meaghan Kern
Great Lakes National Program Office
U.S. Environmental Protection Agency
77 W. Jackson Blvd.
Chicago, IL 60604
Phone: (312) 353-5784

calcs-fish tissue mod October sampling.xlsx

Macroinvertebratecollectionsummar...716 (1).xlsx

	Baywest				
Species biomas	**Mayfly**	**Snail**	**Dragonfly**	**Crawfish**	**Alderfly**
Scanlon					
BW16SR-001	39.36	33.8			
BW16SR-002	40.43		51.72		
BW16SR-003	51.67				
BW16SR-004	NONE				
BW16SR-005		88	48.4	37	
Thomson	NONE				
Boulder					
BW16BR-001					0.9
BW16BR-002					2.4
BW16BR-003					

Will be analyzed for Hg, Total Hg, % lipids, and dioxin
7 Lumbriculus samples for Hg, Total Hg, % lipids, and dioxin

	EPA-ORD	
	Macros (g)	**crawfish (g)**
Scanlon		
BW16SR-HD-001	1.0	
BW16SR-HD-007	0.7	
BW16SR-HD-003	1.4	
BW16SR-HD-004	0.5	
BW16SR-HD-005	0.9	0.4
	4.5	
Thomson		
EPA16-TR-HD-016	2.4	2.4
EPA16-TR-HD-017	0.1	
EPA16-TR-HD-013	0.6	1.2
EPA16-TR-HD-008	1.6	6.6
EPA16-TR-HD-018	1.1	
	5.8	
Boulder		
EPA16-BR-HD-001	2.3	
EPA16-BR-HD-002	6.2	
EPA16-BR-HD-003	2.9	
EPA16-BR-HD-004	3.9	
EPA16-BR-HD-005	5.1	

combined for % lipids, total Hg, and dioxin
combining for % lipids, and total Hg?

CHAIN-OF-CUSTODY / Analytical Request Document

"The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately."

N = 11 jars

Section A Required Client Information	Section B Required Project Information	Section C Invoice Information	Section D EQuIS Information	
Company:	Report To:	Attention:	Facility_Name: St Louis River Sediment Areas of Concern	Page: 1 of 1
Address:	Copy To: mmcdonald@baywest.com	Company Name:	Facility_Code: St Louis River Sed	
	mmcdonald@baywest.com	Address:	Facility_ID:	COC#: SLR-GLEC-3
Email To:	Purchase Order No.:	Lab Quote Reference:	SubFacility_code:	
Phone:	Project Name: SLR Sediment AOCs	Lab Project Manager: Malee Garton		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number:			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WS; Tissue AR; Other TS; OT

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (Sys_Sample_Code)	MATRIX CODE	SAMPLE TYPE (G GRAB C COMP)	Collection: DATE	Collection: Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins/furans (1613B)	Lipids	Mercury (7471B)	Methyl Mercury (1630)	Comments
Ex.	BW16MLW-005-0-0 TS	BW16MLW-005-0-0 TS	SO	C	9/18/16	1200														
1	BW16SR-001	BW16SR-001-M	TS	G	9/19/16	10:40										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
2	BW16SR-002	BW16SR-002-M	TS	G	9/19/16	10:15										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
3	BW16SR-002	BW16SR-002-D	TS	G	9/19/16	10:40										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
4	BW16SR-102	BW16SR-102-D	TS	G	9/19/16	10:40										x	x			Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
5	BW16SR-003	BW16SR-003-M	TS	G	9/19/16	10:10										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
6	BW16SR-103	BW16SR-103-M	TS	G	9/19/16	10:10												x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
7	BW16SR-005	BW16SR-005-D	TS	G	9/19/16	10:40										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
8	BW16SR-005	BW16SR-005-C	TS	G	9/18/16	10:40										x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
✓ 9	EPA16BRHD	EPA16 BRHDMCRS	TS	C																
9	~~EPA16-BR-HD~~	~~EPA16-BR-HD-MCRS~~	TS	C												x	x	x	x	Priority Order 1 Dioxins/Lipids, 2 Hg, 3 MeHg
10	~~EPA16SR-HD~~	~~EPA16SR-HD-MCRS~~	TS	C														x	x	Priority Order 1 Hg, 1 MeHg
11	~~EPA16-TR-HD~~	~~EPA16-TR-HD-MCRS~~	TS	C														x	x	Priority Order 1 Hg, 1 MeHg
10	~~EPA16-TR-HD~~	~~EPA16-TR-HD-C~~	TS	C														x	x	Priority Order 1 Hg, 1 MeHg
✓ 11	BW16SR-003	BW16-SR-003-D	TS	G	9/18/16	11:40										x	x	x	x	

BW16SR-102 is to be collected from BW16SR-003 as a DUP and BW16SR-003M is to be collected from BW16SR003M as a DUP.

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS
Samples Matrix extension definitions: S Mayfly; D Dragonfly; C Crayfish; MCRS Macrosentinels	Amber A/BayWest	11/09/16	1041				

SAMPLER NAME AND SIGNATURE		
PRINT Name of SAMPLER:		
SIGNATURE of SAMPLER:	DATE Signed (MM/DD/YY):	

Temp (°C) | Received on Ice (Y/N) | Custody Sealed Cooler (Y/N) | Samples Intact (Y/N)

V = 6 of 19 jar

GLEC
Great Lakes Environmental Center

GREAT LAKES ENVIRONMENTAL CENTER, INC. (GLEC)
CHAIN OF CUSTODY RECORD
(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company:

Report Results To: Joel Hoffman/Jim Lazor

Address: 6201 Cunningham Blvd, Duluth, MN 55804

Phone: 218-529-5420 E-mail: Hoffman.Joe@epa.gov

Section II.

Project Name: St Louis River

Project Number:

P.O.#:

Sampled by: initials ☐ GLEC JL ☐ Client EPA/MPCA

Section III. Sample Information at Collection

#	LAB USE GLEC ID#	Sample Identification	Sample Information Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers Type	Size	No.	Section IV. Requested Processing/Analysis
1	16-SR	HD-007	10-13-16	9:35	HD	"	ICE			1/1	Dioxin / Hg 0.7g
2	16-SR	HD-003	"	10:10	"	"	"			1/1	" " 1.4g
3	16-SR	HD-001	"	9:55	"	"	"			1/1	" " 1.0g
4	16-SR	HD-004	"	10:20	"	"	"			1/1	" " 0.5g
5	16-SR	HD-005	"	10:35	"	"	"			1/2	" " 0.9g
6	16-SR	HD-005 sunfish	"	"	"	"	"			2/2	" " 0.4g

Client/Sampler Notes:

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Joel Hoffman EPA Duluth	11/15/16	12:00 P.M.	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY
Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice
Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT W = SURFACE WATE E = EFFLUENT GW = GROUNDWATER SL = SLUDGE AO = AQUATIC ORGANISM

GLEC Lab Composite all samples into one sample BW16SR-HD: ___ M
Test America analysis order % Lipids Total Mercury, Methyl mercury

N = 8 of 19 jars

GLEC

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CHAIN OF CUSTODY RECORD
(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company: EPA Mid Continent Ecol. Div
Report Results To: JIM LAZORCHAK
Address: 6201 Congdon BLVD, Duluth MN 55804
Phone: 513 569 7076 E-mail: lazorchak.Jim@EPA.gov

Section II.

Project Name: ST Louis AOC
Project Number:
P.O.#:
Sampled by: initials ☐ GLEC ____ X Client ____

Section III. Sample Information at Collection

#	LAB USE GLEC ID#	Sample Identification	Sample Information Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers Type	Size	No.	Section IV. Requested Processing/Analysis
✓ 1	16-TR	HD-016 jar 1	10/6/16		HD	"	ice			1	Dioxin/Furan 2.4g
✓ 2	16-TR	HD-016 2 jar 2	"		HD	"	"	Glass Fish		1	" " 2.4g
✓ 3	16-TR	HD-017	"		"	"	"			1	" " 0.1g
✓ 4	16-TR	HD-013 1/2	"		"	"	"			1	" " 0.69m
✓ 5	16-TR	HD-013 2/2	"		"	"	"	Glass Fish		1	" " 1.2 gm
✓ 6	16-TR	HD-008 1/2	"		"	"	"			1	" " 1.16 gm
✓	Client/Sampler Notes: 16-TR-008 2/2		"		"	"	"	Glass Fish		1	" " 6.6 gm
	✓16-TR-018		"		"	"	"			1	" " 1.1 gm

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Joe Hoffman EPA Duluth	11/10/16	12:30pm	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY
Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice
Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT; W = SURFACE WATE; E = EFFLUENT; GW = GROUNDWATER; SL = SLUDGE; AQ = AQUATIC ORGANISM

GLEC Lab Composite ~~2~~ samples into one Sample EPA 16 TR HD ___ M
1, 3, 4, 8 TA analysis order % Lipids + total mercury
GLEC Lab Composite 2, 5, 7 into one Sample EPA 16 TR HD ___ C

N = 8 of 19 jars

GLEC
Great Lakes Environmental Center

GREAT LAKES ENVIRONMENTAL CENTER, INC. (GLEC)
CHAIN OF CUSTODY RECORD
(Complete and include a minimum of one per cooler)

739 Hastings Street
Traverse City, MI 49686

www.glec.com
Phone 231-941-2230
Fax 231-941-2240

Section I.

Submitting Company: EPA Mid Continent Eco. Div
Report Results To: JIM LAZORCHAK
Address: 6201 Congdon BLVD, Duluth, MN 55804
Phone: 513 569 7076 E-mail: lazorchak.jim@EPA.gov

Section II.

Project Name: ST Louis AOC
Project Number:
P.O.#:
Sampled by: initials ☐ GLEC ____ ☒ Client ____

Section III. Sample Information at Collection

#	LAB USE GLEC ID#	Sample Identification	Sample Information: Date	Time	Matrix	Grab or Composite	Preservative	Sample Containers: Type	Size	No.	Section IV. Requested Processing/Analysis
1	16-TR	HD-016 jar 2	10/12/16		HD	"	ice			1	Dioxin/Furan 2.4g
2	16-TR	HD-016 jar 2	"		HD	"	"	Ziploc		1	" " 2.4g
3	16-TR	HD-017	"		"	"	"			1	" " 0.1g
4	16-TR	HD-013 1/2	"		"	"	"			1	" " 0.68gm
5	16-TR	HD-013 2/2	"		"	"	"	Ziplock		1	" " 1.2gm
6	16-TR	HD-008 1/2	"		"	"	"			1	" " 1.16gm
	Client/Sampler Notes: 16_TR-008 2/2		"		"	"	"	Ziplock		1	" " 6.6gm
		16-TR-018	"		"	"	"			1	" " 1.1gm

RELEASED BY / ORGANIZATION	DATE	TIME	RECEIVED BY / ORGANIZATION	DATE	TIME
Print Name & Organization: Jim Lazorchak EPA Duluth	11/15/16	12:00 pm	Print Name & Organization		
Signature			Signature		
Print Name & Organization			Print Name & Organization		
Signature			Signature		

FOR LAB USE ONLY
Temperature of Samples: _____ °C ☐ Received on Wet Ice ☐ Received on Dry Ice
Notes/Anomalies/Discrepancies: _____

MATRIX CODES: S = SEDIMENT; W = SURFACE WATE; E = EFFLUENT; GW = GROUNDWATER; SL = SLUDGE; AO = AQUATIC ORGANISM

GLEC Lab Composite ~~all~~ samples into one Sample EPA 16 TR HD ___M
1, 3, 4, 8 TM analysis order % Lipids - total mercury
GLEC Lab composite 2, 5, 7, 6 into one Sample EPA 16 TR HD ___C

APPENDIX G

Scanlon Reservoir Fish Samples Analytical Results Summary Table

Sample ID		MN16+SR-NP-A	MN16+SR-NP-A	MN16+SR-GSH-A	MN16+SR-SMB-A	MN16+SR-SMB-B	MN16+SR-SMB-C	MN16+SR-WAL-A	MN16+SR-WS-A	MN16+SR-WS-B	MN16+SR-WS-C	MN16+SR+YP-A	MN16+SR+YP-B	MN16+SR+YP-C
Fish		Northern Pike	Northern Pike	Shiner Mix	Smallmouth Bass	Smallmouth Bass	Smallmouth Bass	Walleye	White Sucker	White Sucker	White Sucker	Yellow Perch	Yellow Perch	Yellow Perch
GLEC Lab ID		5019	5019	5024	5001	5002	5021	5023	5022	5017	5016	5025	5020	5018
Weight Homoginized	mg	827	827	61	1721	1338	858	620	2543	2120	2389	500	315	432
Weights within 10% of Average		No	No	NA	Yes	No	No	No	No	No	Yes	No	No	No
Lengths within 10% of Average		Yes	Yes	NA	Yes	No	Yes	Yes	Yes	No	Yes	Yes	Yes	No
Test America Lab ID		180-60837-14	180-60837-15	180-60852-1	180-60837-17	180-60852-2	180-60837-18	180-60837-16	180-60837-19	180-60837-20	180-60837-13	180-60852-3	180-60852-4	180-60837-21
Total Mercury	mg/kg	**0.12 J**	**0.13**	**0.054 J**	**0.22**	**0.2**	**0.071 J**	**0.12 J**	**0.075 J**	**0.067 J**	**0.083 J**	**0.086 J**	**0.079 J**	**0.092 J**
Methyl Mercury	µg/kg	**100**	**110**	**41**	**230**	**170**	**110**	**110**	**96**	**58**	**95**	**89**	**98**	**74**
% Lipids	%	**0.14**	**0.16**	**0.55**	**1.3**	**1.1**	**1.1**	**0.37**	**3.1**	**1.3**	**1.8**	**0.72**	**0.56**	**0.40**
1998 WHO FISH TEQ ND=EDL		**0.32**	**0.26**	**0.18**	**0.85**	**0.78**	**0.58**	**0.37**	**1.0**	**0.48**	**0.68**	**0.28**	**0.52**	**0.45**
2005 WHO HUMAN TEQ ND=0		**0.30**	**0.22**	**0.10**	**0.81**	**0.73**	**0.56**	**0.32**	**1.0**	**0.47**	**0.68**	**0.25**	**0.51**	**0.45**
2,3,7,8-TCDD	pg/g	**0.058 J**	**0.084 Q J**	**0.051 Q J**	**0.24 Q J**	**0.27 Q J**	**0.15 Q J**	**0.078 Q J**	**0.11 Q J**	**0.13 J**	**0.20 Q J**	0.030 U	**0.12 Q J**	**0.081 Q J**
Total TCDD	pg/g	**0.078 Q J**	**0.084 Q J**	**0.051 Q J**	**0.24 Q J**	**0.27 Q J**	**0.15 Q J**	**0.14 Q J**	**0.27 Q J**	**0.18 Q J**	**0.20 Q J**	0.030 U	**0.15 Q J**	**0.081 Q J**
1,2,3,7,8-PeCDD	pg/g	**0.18 Q B J**	**0.095 Q B J**	0.035 U	**0.40 Q B J**	**0.25 B Q J**	**0.25 Q B J**	**0.21 B J**	**0.41 Q B J**	**0.23 Q B J**	**0.28 Q B J**	**0.12 B J**	**0.16 Q B J**	**0.23 Q B J**
Total PeCDD	pg/g	**0.18 Q B J**	**0.095 Q B J**	0.035 U	**0.40 Q B J**	**0.25 B Q J**	**0.25 Q B J**	**0.21 B J**	**1.0 J Q B**	**0.49 Q J B**	**0.28 Q B J**	**0.18 Q J B**	**0.16 Q B J**	**0.23 Q B J**
1,2,3,4,7,8-HxCDD	pg/g	0.027 U	0.042 U	0.045 U	0.035 U	0.026 U	0.031 U	0.035 U	**0.16 Q J**	0.042 U	0.051 U	0.052 U	0.031 U	0.038 U
1,2,3,6,7,8-HxCDD	pg/g	**0.11 J**	0.040 U	0.047 U	**0.22 Q J**	**0.20 J**	**0.20 J**	0.037 U	**0.63 J**	**0.22 Q J**	**0.61 J**	**0.38 J**	**0.35 Q J**	**0.37 J**
1,2,3,7,8,9-HxCDD	pg/g	0.025 U	0.038 U	0.043 U	0.031 U	0.025 U	0.030 U	0.034 U	0.030 U	0.041 U	0.049 U	**0.19 J**	0.029 U	**0.13 Q J**
Total HxCDD	pg/g	**0.11 J**	0.040 U	0.045 U	**0.22 Q J**	**0.20 J**	**0.20 J**	0.035 U	**1.4 J Q**	**0.22 Q J**	**1.2 Q J**	**0.58 J**	**0.35 Q J**	**0.50 Q J**
1,2,3,4,6,7,8-HpCDD	pg/g	0.039 U	0.045 U	**0.24 Q B J**	0.058 U	0.042 U	0.056 U	**0.17 Q B J**	**0.75 Q B J**	**0.72 B J**	**0.67 Q B J**	**0.69 B J**	**0.59 Q B J**	**0.82 B J**
Total HpCDD	pg/g	**0.11 Q J**	0.045 U	**0.24 Q B J**	0.058 U	0.042 U	0.056 U	**0.17 Q B J**	**1.2 Q J B**	**1.2 Q J B**	**1.1 Q J B**	**0.69 B J**	**0.59 Q B J**	**0.82 B J**
OCDD	pg/g	**0.57 B J**	**0.73 Q B J**	**0.97 B J**	**0.75 Q B J**	**0.95 B J**	**1.0 B J**	**1.0 Q B J**	**3.6 Q B J**	**3.6 B J**	**3.1 B J**	**1.7 B J**	**2.2 B J**	**2.4 B J**
2,3,7,8-TCDF	pg/g	**0.16 Q J**	**0.098 Q J**	**0.15 Q J**	**0.33 Q J**	**0.18 Q J**	**0.32 J**	0.043 U	**1.5 Q**	**0.46 J**	**0.44 Q J**	**0.085 Q J**	**0.18 Q J**	**0.15 J**
Total TCDF	pg/g	**14 Q**	**13 Q**	**5.3 Q**	**31 Q**	**44 Q**	**48 Q**	**8.4 Q**	**75 Q**	**6.8 Q**	**2.3 Q**	**7.3 Q**	**37 Q**	**26 Q**
1,2,3,7,8-PeCDF	pg/g	0.028 U	0.031 U	0.037 U	0.042 U	0.036 U	0.041 U	0.032 U	0.047 U	0.035 U	0.027 U	0.049 U	0.042 U	0.040 U
2,3,4,7,8-PeCDF	pg/g	0.023 U	0.030 U	0.034 U	**0.22 Q J**	**0.27 J**	0.040 U	0.031 U	**0.37 J**	0.035 U	**0.19 J**	0.046 U	**0.15 Q J**	0.038 U
Total PeCDF	pg/g	**1.3 J Q**	**1.4 J Q**	**0.26 Q J**	**4.2 Q J**	**20 Q**	**8.8 Q**	**1.2 Q J**	**14 Q**	**0.88 Q J**	**0.58 Q J**	**4.0 J Q**	**6.2 J Q**	**3.4 J Q**
1,2,3,4,7,8-HxCDF	pg/g	0.026 U	0.033 U	**0.30 Q J**	0.035 U	0.029 U	0.039 U	0.046 U	**0.2 Q J**	0.056 U	**0.18 Q J**	**0.17 J**	**0.22 Q J**	0.036 U
1,2,3,6,7,8-HxCDF	pg/g	**0.34 Q B J**	**0.26 Q B J**	0.063 U	**0.47 Q B J**	**0.92 Q B J**	**1.1 Q B J**	**0.26 Q B J**	**1.2 Q B J**	**0.22 Q B J**	0.052 U	**0.34 Q B J**	**1.0 Q B J**	**0.59 Q B J**
2,3,4,6,7,8-HxCDF	pg/g	0.024 U	0.031 U	0.045 U	0.035 U	0.028 U	0.042 U	0.032 U	0.037 U	0.041 U	0.029 U	0.050 U	0.039 U	0.039 U
1,2,3,7,8,9-HxCDF	pg/g	0.030 U	0.040 U	0.053 U	0.047 U	0.033 U	0.051 U	0.043 U	0.048 U	0.052 U	0.035 U	0.069 U	0.049 U	0.051 U
Total HxCDF	pg/g	**1.3 J Q B**	**1.0 Q J B**	**0.64 Q J**	**4.2 J Q B**	**6.3 Q J B**	**7.7 Q J B**	**1.8 J Q B**	**16 Q B**	**2.5 J Q B**	**3.1 J Q**	**4.8 J Q B**	**8.4 J Q B**	**5.6 J Q B**
1,2,3,4,6,7,8-HpCDF	pg/g	**0.08 Q B J**	0.028 U	**0.17 Q B J**	**0.21 Q B J**	**0.18 Q B J**	0.035 U	**0.33 Q B J**	**1.4 B J**	**1.1 Q B J**	**1.5 B J**	**0.86 Q B J**	**0.60 B J**	**0.89 B J**
1,2,3,4,7,8,9-HpCDF	pg/g	**0.056 Q B J**	0.039 U	0.038 U	0.043 U	0.031 U	0.043 U	0.039 U	0.044 U	0.038 U	0.033 U	0.062 U	0.042 U	0.043 U
Total HpCDF	pg/g	**0.14 Q B J**	0.032 U	**0.17 Q B J**	**0.21 Q B J**	**0.18 Q B J**	0.039 U	**0.58 Q J B**	**2.2 Q J B**	**1.9 J Q B**	**2.6 Q J B**	**1.1 Q J B**	**0.77 J B**	**1.2 Q J B**
OCDF	pg/g	**0.15 B J**	**0.049 Q B J**	**0.28 Q B J**	**0.22 B J**	**0.12 Q B J**	0.024 U	**0.16 B J**	**0.37 B J**	**0.31 Q B J**	**0.38 B J**	**0.38 B J**	**0.28 B J**	**0.19 Q B J**

*Results are on an as-received (wet-weight) basis, and have not been corrected for dry weight or % lipids.

B - The analyte is present in the associated method blank at a detectable level.
J - The reported result is an estimata
Q - Estimated maximum possible concentration.
U - Not detected
TEQ calculated with non-detect values (U) being 0

APPENDIX H

Thomson Reservoir Fish Samples Analytical Results Summary Table

Sample ID		MN16+TR-NP-A	MN16+TR-RB-A	MN16+TR-RB-B	MN16+TR-SMB-A	MN16+TR-SMB-B	MN16+TR-SMB-C	MN16+TR-SMB-D	MN16+TR-WAL-A	MN16+TR-WS-A	MN16+TR-WS-A DUP	MN16+TR-WS-B	MN16+TR-WS-C	MN16+TR+YP-A	MN16+TR+YP-A Dup	MN16+TR+YP-B
Fish		**Northern Pike**	**Rock Bass**	**Rock Bass**	**Smallmouth Bass**	**Smallmouth Bass**	**Smallmouth Bass**	**Smallmouth Bass**	**Walleye**	**White Sucker**	**White Sucker**	**White Sucker**	**White Sucker**	**Yellow Perch**	**Yellow Perch**	**Yellow Perch**
GLEC Lab ID		5006	5009	5010	5003	5036	5004	5038	5007	5011	5011	5015	5014	5005	5005	5008
Weight Homoginized	mg	639	250	150	394	2245	3038	3358	932	3412	3412	2708	2321	859	859	781
Weights within 10% of Average		No	No	No	No	Yes	Yes	No	No	Yes	Yes	No	Yes	No	No	No
Lengths within 10% of Average		Yes	No	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Test America Lab ID		180-60837-7	180-60837-3	180-60593-16	180-60837-4	180-60837-5	180-60837-6	180-60593-17	180-60593-15	180-60837-11	180-60837-12	180-60837-1	180-60837-2	180-60837-8	180-60837-9	180-60837-10
Total Mercury	mg/kg	**0.066 J**	**0.11 J**	**0.049 J B**	**0.078 J**	**0.17**	**0.22**	**0.1 J B**	**0.17 B**	**0.12 J**	**0.12 J**	**0.086 J**	**0.1 J**	**0.085 J**	**0.083 J**	**0.053 J**
Methyl Mercury	µg/kg	**78**	**92**	**83**	**70**	**140**	**220**	**99**	**200**	**110**	**110**	**94**	**110**	**74**	**73**	**49**
% Lipids	%	**0.071 J**	**0.48**	**0.96**	**0.73**	**1.3**	**1.1**	**1.3**	**0.6**	**3.1**	**3.6**	**2.7**	**2.2**	**1.8**	**2.2**	**1.1**
1998 WHO FISH TEQ ND=EDL		**0.28**	**0.31**	**0.25**	**0.26**	**0.56**	**0.77**	**0.24**	**0.42**	**1.1**	**0.84**	**0.36**	**0.73**	**0.46**	**0.48**	**0.74**
2005 WHO HUMAN TEQ ND=0		**0.27**	**0.28**	**0.029**	**0.18**	**0.50**	**0.65**	**0.016**	**0.32**	**1.1**	**0.82**	**0.35**	**0.69**	**0.46**	**0.48**	**0.77**
2,3,7,8-TCDD	pg/g	**0.074 Q J**	**0.056 Q J**	0.039 U	0.046 U	**0.11 Q J**	**0.14 Q J**	0.038 U	0.043 U	**0.26 Q J**	**0.19 Q J**	**0.051 Q J**	**0.20 Q J**	**0.045 Q J**	**0.083 Q J**	**0.14 Q J**
Total TCDD	pg/g	**0.074 Q J**	**0.056 Q J**	0.039 U	**0.15 Q J B**	**0.11 Q J**	**0.26 B Q J**	0.038 U	0.043 U	**0.54 Q J**	**0.49 Q J**	**0.051 Q J**	**0.32 Q J**	**0.12 Q J B**	**0.083 Q J**	**0.14 Q J**
1,2,3,7,8-PeCDD	pg/g	**0.084 Q J**	**0.17 Q J**	0.034 U	**0.063 Q J**	**0.30 Q J**	**0.29 Q J**	0.050 U	**0.18 Q J**	**0.41 Q J**	**0.27 Q B J**	**0.18 Q J**	**0.20 Q J**	**0.14 Q J**	**0.19 J**	**0.35 J**
Total PeCDD	pg/g	**0.084 Q J**	**0.17 Q J**	0.034 U	**0.063 Q J**	**0.30 Q J**	**0.29 Q J**	0.050 U	**0.18 Q J**	**0.73 Q J**	**0.72 Q J B**	**0.45 Q J**	**0.20 Q J**	**0.18 Q J**	**0.19 J**	**0.35 J**
1,2,3,4,7,8-HxCDD	pg/g	0.026 U	0.025 U	0.097 U	0.025 U	0.031 U	0.037 U	0.083 U	0.070 U	**0.11 Q J**	**0.11 Q J**	0.049 U	0.048 U	0.031 U	0.032 U	0.028 U
1,2,3,6,7,8-HxCDD	pg/g	**0.11 Q J**	**0.063 Q J**	0.110 U	**0.12 J**	0.035 U	**0.31 J**	0.084 U	**0.26 J**	**0.55 Q J**	**0.50 J**	**0.23 Q J**	**0.41 J**	**0.42 Q J**	**0.36 Q J**	**0.40 Q J**
1,2,3,7,8,9-HxCDD	pg/g	0.026 U	0.024 U	0.096 U	0.023 U	0.031 U	0.037 U	0.078 U	0.067 U	0.027 U	0.031 U	0.047 U	0.047 U	**0.12 J**	**0.12 Q J**	**0.18 J**
Total HxCDD	pg/g	**0.11 Q J**	**0.063 Q J**	0.100 U	**0.12 J**	0.032 U	**0.31 J**	0.082 U	**0.26 J**	**0.92 Q J**	**0.61 Q J**	**0.23 Q J**	**0.41 J**	**0.54 J Q**	**0.49 Q J**	**0.58 J Q**
1,2,3,4,6,7,8-HpCDD	pg/g	**0.19 Q J**	**0.11 Q J**	0.110 U	0.061 U	0.068 U	0.062 U	0.120 U	0.098 U	**0.60 Q J**	**0.59 Q B J**	**0.23 Q J**	**0.43 Q J**	**1.3 J**	**1.2 J**	**1.1 J**
Total HpCDD	pg/g	**0.19 Q J**	**0.19 Q J**	0.110 U	**0.20 Q J**	0.068 U	0.062 U	0.120 U	0.098 U	**0.76 J Q**	**0.76 Q J B**	**0.52 Q J**	**0.68 Q J**	**1.3 J**	**1.3 Q J**	**1.4 J**
OCDD	pg/g	**1.1 B J**	**0.42 Q B J**	**1.3 B J**	**2.2 Q B J**	**0.44 Q B J**	**1.1 B J**	**0.76 Q B J**	**0.92 B J**	**2.1 B J**	**1.3 Q B J**	**1.7 B J**	**1.9 B J**	**2.8 B J**	**2.7 Q B J**	**3.1 B J**
2,3,7,8-TCDF	pg/g	**0.28 Q J**	**0.12 J**	**0.24 J**	**0.15 J**	**0.061 Q J**	**0.24 J**	0.054 U	**0.082 Q J**	**0.96 J**	**0.94 J**	**0.62 J**	**0.61 Q J**	**0.17 Q J**	**0.15 Q J**	**0.42 J**
Total TCDF	pg/g	**13 Q**	**13 Q**	**0.54 Q J**	**15 Q**	**5.4 Q**	**5.3 Q**	**0.64 J Q**	**25 Q**	**40 Q**	**38 Q**	**6.2 Q**	**6.5 Q**	**20 Q**	**2.6 Q**	**21 Q**
1,2,3,7,8-PeCDF	pg/g	0.027 U	0.037 U	0.038 U	0.039 U	0.032 U	**0.17 Q J**	0.044 U	0.053 U	0.035 U	**0.12 Q J**	0.048 U	0.063 U	0.041 U	**0.062 Q J**	0.032 U
2,3,4,7,8-PeCDF	pg/g	0.025 U	0.035 U	0.037 U	**0.14 Q J**	**0.16 Q J**	**0.54 J**	**0.052 Q J**	0.054 U	**0.36 J**	**0.34 J**	0.047 U	**0.36 J**	**0.17 Q J**	**0.18 J**	**0.15 Q J**
Total PeCDF	pg/g	**3.5 J Q**	**1.6 J Q**	0.037 U	**3.7 Q J**	**0.96 Q J**	**1.7 Q J**	**0.15 Q J**	**6.2 J Q**	**5.2 J Q**	**5.4 Q J**	**0.27 Q J**	**1.7 Q J**	**3.9 J Q**	**0.55 Q J**	**4.4 J Q**
1,2,3,4,7,8-HxCDF	pg/g	0.030 U	0.033 U	0.350 U	0.038 U	0.034 U	0.066 U	0.290 U	0.097 U	**0.18 Q J**	**0.15 Q J**	0.059 U	**0.39 Q J**	0.045 U	0.048 U	**0.19 Q J**
1,2,3,6,7,8-HxCDF	pg/g	**0.71 Q J**	**0.31 Q J**	0.460 U	**0.48 Q J**	**0.34 J**	0.070 U	0.330 U	**0.98 B J**	**0.89 Q J**	**0.69 Q B J**	**0.21 Q J**	**0.34 Q J**	**1.3 Q J**	**0.50 J**	**0.92 Q J**
2,3,4,6,7,8-HxCDF	pg/g	0.025 U	0.034 U	0.050 U	0.042 U	0.035 U	0.043 U	0.057 U	0.057 U	0.028 U	0.026 U	0.044 U	0.050 U	0.043 U	**0.093 Q J**	0.031 U
1,2,3,7,8,9-HxCDF	pg/g	0.029 U	0.042 U	0.063 U	0.052 U	0.048 U	0.055 U	0.067 U	0.075 U	0.034 U	0.035 U	0.046 U	0.066 U	0.053 U	0.036 U	0.040 U
Total HxCDF	pg/g	**4.5 Q J**	**2.3 J Q**	**0.69 Q J**	**4.2 Q J**	**1.5 J Q**	**1.4 Q J**	0.100 U	**6.2 J Q B**	**8.3 Q J**	**6.8 Q J B**	**2.0 Q J**	**3.4 J Q**	**9.1 J Q**	**4.4 J Q**	**8.2 Q J**
1,2,3,4,6,7,8-HpCDF	pg/g	0.031 U	**0.23 Q B J**	**0.44 Q B J**	0.037 U	0.020 U	0.075 U	0.170 U	**0.27 Q B J**	**0.74 B J**	**0.68 B J**	**0.58 B J**	**0.47 Q B J**	**1.2 Q B J**	**1.2 Q B J**	**1.1 B J**
1,2,3,4,7,8,9-HpCDF	pg/g	0.034 U	0.043 U	0.051 U	0.048 U	0.029 U	0.062 U	0.054 U	0.086 U	0.031 U	0.035 U	0.053 U	0.048 U	0.056 U	0.045 U	0.029 U
Total HpCDF	pg/g	0.032 U	**0.23 Q B J**	**0.44 Q B J**	0.042 U	0.024 U	0.068 U	0.080 U	**0.27 Q B J**	**1.1 Q J B**	**1.0 Q J B**	**0.93 Q J B**	**0.84 Q J B**	**1.4 Q J B**	**1.4 Q J B**	**1.4 Q J B**
OCDF	pg/g	**0.039 J**	**0.037 Q J**	**0.12 Q B J**	0.0093 U	**0.031 Q J**	**0.032 Q J**	0.045 U	**0.25 Q B J**	**0.20 J**	**0.14 B J**	**0.13 Q J**	**0.32 J**	**0.065 J**	**0.08 Q J**	**0.17 Q J**

*Results are on an as-received (wet-weight) basis, and have not been corrected for dry weight or % lipids.

B - The analyte is present in the associated method blank at a detectable level.
J - The reported result is an estimata
Q - Estimated maximum possible concentration.
U - Not detected
TEQ calculated with non-detect values (U) being ND=0

APPENDIX I

Boulder Lake Reservoir Fish Samples Analytical Results Summary Table

APPENDIX I
BOULDER LAKE (BACKGROUND) RESERVOIR FISH SAMPLES
ANALYTICAL RESULTS SUMMARY TABLE

Sample ID		MN16+BR+BLC-A	MN16+BR+RB-A	MN16+BR+GSH-A	MN16+BR+GSH-B	MN16+BR+GSH-C	MN16+BR+WAL-A	MN16+BR+WAL-A	MN16+BR+WAL-B	MN16+BR+WAL-C	MN16+BR+WS-A	MN16+BR+WS-B	MN16+BR+WS-C	MN16+BR+YP-A	MN16+BR+YP-B	MN16+BR+YP-C
Fish		Black Crappie	Rock Bass	Shiner Mix	Shiner Mix	Shiner Mix	Walleye	Walleye	Walleye	Walleye	White Sucker	White Sucker	White Sucker	Yellow Perch	Yellow Perch	Yellow Perch
Lab ID		5044	5035	5033	5032	5045	5041	5041	5042	5043	5027	5029	5028	5031	5030	5034
Weight Homoginized	mg	116	368	152	152	163	1819	424	370	370	1847	3002	4390	378	311	304
Weights within 10% of Average		No	No	NA	NA	NA	Yes	Yes	No	Yes	No	No	No	No	No	No
Lengths within 10% of Average		No	No	NA	NA	NA	Yes	Yes	No	Yes	No	No	Yes	No	No	No
Test America Lab ID		180-60593-2	180-60593-1	180-60593-6	180-60593-7	180-60593-8	180-60593-9	180-60593-20	180-60593-14	180-60593-10	180-60593-12	180-60593-11	180-60593-13	180-60593-3	180-60593-4	180-60593-5
Methyl Mercury	µg/kg	**53**	**76**	**62**	**65**	**62**	**140**	**140**	**120**	**130**	**57**	**81**	**110**	**56**	**54**	**65**
Total Mercury	mg/kg	**0.068 J B**	**0.077 J B**	**0.064 J B**	**0.071 J B**	**0.068 J B**	**0.13 J B**	**0.12 J B**	**0.098 J B**	**0.11 J B**	**0.056 J B**	**0.071 J B**	**0.051 J B**	**0.073 J B**	**0.068 J B**	**0.077 J B**
% Lipids	%	**1.0**	**1.2**	**1.8**	**1.5**	**2.0**	**2.0**	**2.0**	**0.28**	**0.27**	**2.2**	**2.5**	**3.5**	**0.52**	**0.27**	**0.45**
1998 WHO FISH TEQ ND=EDL		**0.10**	**0.13**	**0.11**	**0.13**	**0.084**	**0.2**	**0.20**	**0.11**	**0.11**	**0.22**	**0.22**	**0.14**	**0.14**	**0.14**	**0.098**
2005 WHO HUMAN TEQ ND=0		**0.00015**	**0.0002**	**0.00086**	**0.018**	**0.00012**	**0.17**	**0.028**	**0.0045**	**0.000075**	**0.16**	**0.11**	**0.018**	**0.035**	**0.00016**	**0.000066**
2,3,7,8-TCDD	pg/g	0.026 U	0.037 U	0.031 U	0.037 U	0.016 U	**0.046 J**	0.033 U	0.032 U	0.027 U	0.028 U	0.029 U	0.033 U	0.024 U	0.038 U	0.027 U
Total TCDD	pg/g	0.026 U	0.037 U	0.031 U	0.037 U	**0.04 Q B J**	**0.091 J**	**0.047 J**	0.032 U	0.027 U	**0.036 Q J**	0.029 U	**0.068 Q J**	0.024 U	**0.048 Q J**	0.027 U
1,2,3,7,8-PeCDD	pg/g	0.022 U	0.026 U	0.023 U	0.029 U	0.023 U	**0.086 Q J**	0.049 U	0.029 U	0.025 U	**0.12 Q J**	**0.11 Q J**	0.037 U	0.029 U	0.032 U	0.024 U
Total PeCDD	pg/g	0.022 U	0.026 U	0.023 U	**0.11 Q J**	0.023 U	**0.086 Q J**	0.049 U	**0.093 Q J**	0.025 U	**0.33 J Q**	**0.30 Q J**	**0.89 Q J**	0.029 U	0.032 U	0.024 U
1,2,3,4,7,8-HxCDD	pg/g	0.034 U	0.042 U	0.040 U	0.038 U	0.029 U	0.035 U	0.059 U	0.037 U	0.034 U	0.033 U	0.040 U	0.034 U	0.034 U	0.049 U	0.032 U
1,2,3,6,7,8-HxCDD	pg/g	0.031 U	0.042 U	0.043 U	0.037 U	0.030 U	0.038 U	0.067 U	0.039 U	0.035 U	**0.099 Q J**	0.042 U	0.034 U	0.033 U	0.051 U	0.032 U
1,2,3,7,8,9-HxCDD	pg/g	0.030 U	0.039 U	0.039 U	**0.15 J**	0.028 U	0.034 U	0.058 U	0.035 U	0.032 U	0.030 U	0.038 U	0.032 U	0.031 U	0.047 U	0.030 U
Total HxCDD	pg/g	0.032 U	0.041 U	0.040 U	**0.15 J**	0.029 U	0.035 U	0.061 U	**1.2 J**	0.034 U	**0.099 Q J**	0.040 U	0.033 U	0.033 U	0.049 U	0.031 U
1,2,3,4,6,7,8-HpCDD	pg/g	0.051 U	0.056 U	0.060 U	0.049 U	0.046 U	**0.47 J**	0.081 U	0.045 U	0.048 U	0.055 U	0.057 U	0.051 U	**0.091 Q J**	0.064 U	0.040 U
Total HpCDD	pg/g	0.051 U	**0.18 Q J**	0.060 U	0.049 U	**0.068 Q J**	**0.73 Q J**	0.081 U	0.045 U	0.048 U	0.055 U	0.057 U	0.051 U	**0.091 Q J**	0.064 U	0.040 U
OCDD	pg/g	**0.5 B J**	**0.68 B J**	**1.1 Q B J**	**0.66 B J**	**0.36 Q B J**	**3.9 B J**	**0.29 Q B J**	**0.71 B J**	**0.25 Q B J**	**0.39 B J**	**0.49 B J**	**0.38 B J**	**0.40 Q B J**	**0.55 Q B J**	**0.22 Q B J**
2,3,7,8-TCDF	pg/g	0.057 U	0.067 U	0.049 U	0.061 U	0.040 U	0.079 U	0.110 U	0.039 U	0.050 U	**0.12 Q J**	0.072 U	0.067 U	0.053 U	0.072 U	0.044 U
Total TCDF	pg/g	**9.0 Q**	**8.1 Q**	**9.5 Q**	**9.3 Q**	**9.4 Q**	**90 Q**	**34 Q**	**13 Q**	**11 Q**	**25 Q**	**31 Q**	**42 Q**	**20 Q**	**14 Q**	**10 Q**
1,2,3,7,8-PeCDF	pg/g	0.041 U	0.043 U	0.041 U	0.034 U	0.033 U	0.048 U	0.060 U	0.025 U	0.041 U	0.034 U	0.038 U	0.035 U	0.041 U	0.056 U	0.032 U
2,3,4,7,8-PeCDF	pg/g	0.035 U	0.040 U	0.038 U	0.033 U	0.031 U	0.048 U	0.060 U	0.025 U	0.035 U	0.031 U	0.034 U	0.032 U	**0.071 Q J**	0.050 U	0.029 U
Total PeCDF	pg/g	**0.54 Q J**	**1.5 J**	**0.67 Q J**	**1.4 Q J**	**0.49 Q J**	**6.6 J Q**	**6.4 J Q**	**0.67 Q J**	**0.89 Q J**	**4.7 Q J**	**2.1 J**	**3.0 J Q**	**1.4 Q J**	**0.92 Q J**	**0.45 Q J**
1,2,3,4,7,8-HxCDF	pg/g	0.033 U	0.042 U	0.032 U	0.034 U	0.024 U	0.036 U	0.084 U	**0.043 Q B J**	0.034 U	0.030 U	0.036 U	0.030 U	0.031 U	0.039 U	0.028 U
1,2,3,6,7,8-HxCDF	pg/g	0.033 U	0.039 U	0.033 U	0.035 U	0.023 U	**0.25 Q J**	**0.28 Q B J**	0.025 U	0.034 U	**0.13 Q J**	0.037 U	**0.18 Q J**	**0.13 Q J**	0.039 U	0.026 U
2,3,4,6,7,8-HxCDF	pg/g	0.032 U	0.033 U	0.033 U	0.035 U	0.026 U	0.038 U	0.059 U	0.027 U	0.035 U	**0.052 Q J**	0.040 U	0.032 U	0.033 U	0.040 U	0.027 U
1,2,3,7,8,9-HxCDF	pg/g	0.045 U	0.045 U	0.041 U	0.048 U	0.033 U	0.050 U	0.072 U	0.033 U	0.047 U	0.045 U	0.051 U	0.041 U	0.043 U	0.056 U	0.039 U
Total HxCDF	pg/g	0.035 U	0.039 U	**0.12 Q J**	0.037 U	0.026 U	**1.3 Q J**	**2.7 J Q B**	**0.043 Q B J**	**0.15 Q J**	**0.46 Q J**	**0.19 Q J**	**0.64 Q J**	**0.24 Q J**	0.042 U	0.029 U
1,2,3,4,6,7,8-HpCDF	pg/g	0.017 U	0.020 U	**0.053 Q B J**	**0.12 B J**	0.013 U	**0.3 B J**	0.059 U	0.014 U	0.022 U	**0.055 Q B J**	0.018 U	0.022 U	0.019 U	0.022 U	0.015 U
1,2,3,4,7,8,9-HpCDF	pg/g	0.024 U	0.031 U	0.028 U	**0.15 Q J**	0.020 U	0.044 U	0.053 U	0.021 U	0.034 U	0.035 U	0.030 U	0.037 U	0.026 U	0.036 U	0.022 U
Total HpCDF	pg/g	0.020 U	0.024 U	**0.053 Q B J**	**0.27 B J Q**	0.016 U	**0.3 B J**	0.055 U	0.017 U	0.027 U	**0.055 Q B J**	0.023 U	0.027 U	0.022 U	0.027 U	0.018 U
OCDF	pg/g	0.021 U	0.031 U	0.021 U	**0.27 Q B J**	**0.043 Q B J**	**0.62 B J**	**0.072 Q B J**	0.026 U	0.027 U	0.026 U	**0.11 Q B J**	**0.10 B J**	**0.089 Q B J**	0.026 U	0.019 U

*Results are on an as-received (wet-weight) basis, and have not been corrected for dry weight or % lipids.

B - The analyte is present in the associated method blank at a detectable level.
J - The reported result is an estimata
Q - Estimated maximum possible concentration.
U - Not detected
TEQ calculated with non-detect values (U) being ND=0

APPENDIX J

Test America Lab Report

APPENDIX K

Test America Lab Report

APPENDIX L

Test America Lab Report

APPENDIX M

Macroinvertebrate Analytical Results Summary Table

Reservoir		**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Scanlon**	**Boulder Lake**	**Scanlon**	**Thomson**	**Thomson**
Sample ID		BW16SR-001M	BW16SR-002M	BW16SR-003M	BW16SR-103M	BW16SR-002D	BW16SR-102D	BW16SR-003D	BW16SR-005D	BW16SR-005C	EPA16-BR-HD-001-MCRS	EPA16-SR-HD-001-MCRS	EPA16-TR-HD-001-MCRS	EPA16-TR-HD-001-C
TA Laboratory ID		180-61461-1	180-61461-2	180-61461-5	180-61461-6	180-61461-3	180-61461-4	180-61461-7	180-61461-8	180-61461-9	180-61461-10	180-61461-11	180-61461-12	180-61461-13
Organism		Mayfly	Mayfly	Mayfly	Mayfly	Dragonfly	Dragonfly	Dragonfly	Dragonfly	Crawfish	Macro	Macro	Macro	Crawfish
Methyl Mercury	µg/kg	**3.1**	**3.3**	**4.5**	**3.6**	**23**	NA	NA	**25**	**18**	**4.3**	**4.5**	**2.7**	**34**
Mercury	mg/kg	0.034 U	0.031 U	0.036 U	0.033 U	0.029 U	NA	NA	0.030 U	0.030 U	0.032 U	0.037 U	0.036 U	**0.036 J**
Percent Lipids	%	**0.72**	**0.56**	**0.66**	NA	**0.68**	**0.81**	**0.69**	**0.78**	**0.39**	**0.72**	NA	NA	NA
1998 WHO FISH TEQ ND=EDL		**1.5**	**0.51**	**1.3**	NA	**0.47**	**0.58**	**0.92**	**0.44**	**0.59**	**0.30**	NA	NA	NA
2005 WHO HUMAN TEQ ND=0		**1.7**	**0.41**	**1.5**	NA	**0.47**	**0.28**	**0.84**	**0.41**	**0.63**	**0.016**	NA	NA	NA
2,3,7,8-TCDD	pg/g	**0.15 J**	0.055 U	0.074 U	NA	0.036 U	0.100 U	0.094 U	0.050 U	**0.037 Q J**	0.091 U	NA	NA	NA
Total TCDD	pg/g	**1.4 Q B J**	**0.61 Q B J**	**0.78 Q B J**	NA	**0.7 Q B J**	**3.3 Q B**	**1.2 Q J**	**0.53 Q B J**	**1.2 B J Q**	0.091 U	NA	NA	NA
1,2,3,7,8-PeCDD	pg/g	**0.44 J**	0.064 U	0.082 U	NA	0.045 U	0.099 U	0.110 U	0.058 U	0.098 U	0.051 U	NA	NA	NA
Total PeCDD	pg/g	**3.0 Q J**	**0.93 J Q**	**1.8 Q J**	NA	**1.2 J Q**	**0.35 Q J**	**1.8 Q J**	**0.76 Q J**	**4.3 J Q**	0.051 U	NA	NA	NA
1,2,3,4,7,8-HxCDD	pg/g	**0.35 Q J**	0.066 U	0.1 U	NA	0.058 U	0.130 U	0.190 U	0.071 U	0.093 U	0.110 U	NA	NA	NA
1,2,3,6,7,8-HxCDD	pg/g	**1.5 J**	**0.49 J**	**2.0 Q J**	NA	**0.53 Q J**	0.130 U	**1.4 J**	**0.80 J**	**1.1 J**	0.120 U	NA	NA	NA
1,2,3,7,8,9-HxCDD	pg/g	**1.1 J**	0.062 U	**1.1 J**	NA	**0.29 J**	0.120 U	**0.69 Q J**	**0.33 J**	**0.72 J**	0.110 U	NA	NA	NA
Total HxCDD	pg/g	**17 Q**	**4.1 J**	**16 Q**	NA	**3.7 Q J**	**3.3 Q J**	**11 J Q**	**4.9 J Q**	**9.7**	0.110 U	NA	NA	NA
1,2,3,4,6,7,8-HpCDD	pg/g	**15**	**4.7 J**	**15**	NA	**5.8**	**4.4 J**	**13**	**5.9**	**5.2**	**0.34 Q J**	NA	NA	NA
Total HpCDD	pg/g	**39**	**12**	**30**	NA	**11 Q**	**9.0 Q J**	**27**	**12**	**11**	**0.34 Q J**	NA	NA	NA
OCDD	pg/g	**150 B**	**47 B**	**94 B**	NA	**44 B**	**35 Q B**	**87 B**	**40 B**	**33 B**	**4.0 Q B J**	NA	NA	NA
2,3,7,8-TCDF	pg/g	**0.58 Q J**	0.069 U	**0.34 Q J**	NA	**0.2 Q J**	0.086 U	0.110 U	0.050 U	**0.38 Q J**	0.087 U	NA	NA	NA
Total TCDF	pg/g	**7.2 Q**	**3.6 Q**	**7.7 Q**	NA	**1.6 J Q**	**0.96 Q J**	**2.5 Q J**	**1.5 Q**	**5.9 Q**	**2.0 Q J**	NA	NA	NA
1,2,3,7,8-PeCDF	pg/g	0.066 U	0.055 U	0.083 U	NA	0.051 U	0.093 U	0.120 U	0.054 U	0.079 U	0.088 U	NA	NA	NA
2,3,4,7,8-PeCDF	pg/g	**0.26 J**	0.050 U	0.082 U	NA	0.045 U	0.082 U	0.110 U	0.051 U	0.073 U	0.078 U	NA	NA	NA
Total PeCDF	pg/g	**4.3 J Q**	**1.6 Q J**	**6.5 J Q**	NA	**2.3 J Q**	**1.1 Q J**	**1.7 Q J**	**2.3 J Q**	**6.9 Q J**	0.082 U	NA	NA	NA
1,2,3,4,7,8-HxCDF	pg/g	**0.76 J**	**0.34 Q J**	**1.5 J**	NA	**0.45 J**	0.100 U	0.130 U	**0.26 Q J**	**0.53 J**	0.110 U	NA	NA	NA
1,2,3,6,7,8-HxCDF	pg/g	**1.5 Q J**	**0.92 Q J**	**1.6 J**	NA	**0.66 Q J**	**0.80 Q J**	**1.1 J**	**0.48 J**	**0.52 Q J**	0.096 U	NA	NA	NA
2,3,4,6,7,8-HxCDF	pg/g	**0.22 Q J**	0.062 U	**0.48 J**	NA	0.058 U	0.110 U	0.130 U	0.062 U	**0.25 Q J**	0.098 U	NA	NA	NA
1,2,3,7,8,9-HxCDF	pg/g	0.079 U	0.079 U	0.12 U	NA	0.076 U	0.140 U	0.160 U	0.081 U	0.098 U	0.120 U	NA	NA	NA
Total HxCDF	pg/g	**26 Q**	**16 Q**	**80 Q**	NA	**14 Q**	**12 J Q**	**24**	**13 Q**	**26 Q**	0.100 U	NA	NA	NA
1,2,3,4,6,7,8-HpCDF	pg/g	**24 B**	**17 B**	**64 B**	NA	**18 B**	**14 B**	**36 B**	**15 B**	**18 B**	**1.1 Q B J**	NA	NA	NA
1,2,3,4,7,8,9-HpCDF	pg/g	**0.37 Q J**	0.073 U	**0.65 Q J**	NA	0.078 U	0.140 U	0.160 U	0.097 U	0.10 U	0.170 U	NA	NA	NA
Total HpCDF	pg/g	**45 B Q**	**33 B**	**120 Q B**	NA	**31 B**	**24 B**	**64 B**	**27 B**	**28 B**	**1.9 Q J B**	NA	NA	NA
OCDF	pg/g	**8.7 B J**	**4.3 B J**	**17 B**	NA	**6.4 B J**	**4.5 Q B J**	**12 B J**	**4.9 J B**	**5.6 B J**	**0.33 Q B J**	NA	NA	NA

*Results are on an as-received (wet-weight) basis, and have not been corrected for dry weight or % lipids.
NA - Sample not analyzed due to client request or lack of organisms
B - The analyte is present in the associated method blank at a detectable level.
J - The reported result is an estimate
Q - Estimated maximum possible concentration.
U - Not detected
TEQ calculated with non-detect values (U) being 0

APPENDIX N

Test America Lab Report

APPENDIX O

Lumbriculus Variegatus Analytical Results Summary Table

DULUTH RESERVOIRS (SCANLON, THOMSON, AND BOULDER)
DULUTH MINNESOTA 2016 TISSUE ANALYSIS

APPENDIX O
LUMBRICULUS VARIEGATUS SAMPLES
ANALYTICAL RESULTS SUMMARY TABLE

Reservoir			**Boulder**	**Scanlon**	**Scanlon**	**Thomson**	**Thomson**	**Thomson**	**Thomson**
		BACKGROUND DAY 0	BW16BLR -001	BW16SR -004	BW16SR -016	BW16TR -008	BW16TR -013	BW16TR -017	BW16TR -018
GLEC Lab ID			11097	11095	11096	11101	11100	11098	11099
Test America Lab ID		180-62135-8	180-62135-1	180-62135-2	180-62135-3	180-62135-7	180-62135-6	180-62135-4	180-62135-5
Methyl Mercury	µg/kg	**0.088**	**0.15**	**0.24**	**0.32**	**0.19**	**0.22**	**0.23**	**0.25**
Mercury	mg/kg	0.038 U	0.038 U	0.036 U	0.037 U	0.038 U	0.038 U	0.033 U	0.030 U
Percent Lipids	%	**1.2**	**0.63**	**0.71**	**0.74**	**0.74**	**0.68**	**0.61**	**0.62**
1998 WHO FISH TEQ ND=EDL		**0.19**	**0.17**	**1.1**	**4.0**	**0.33**	**0.39**	**0.50**	**0.46**
2005 WHO HUMAN TEQ ND=0		**0.013**	**0.00024**	**1.3**	**4.3**	**0.10**	**0.14**	**0.33**	**0.25**
1,2,3,4,6,7,8-HpCDD		0.1 U	0.082 U	**8.1**	**15**	**0.76 Q J**	**1.7 Q J**	**4.1 J**	**1.6 Q J**
1,2,3,4,6,7,8-HpCDF		0.16 U	0.044 U	**14 B**	**86 B**	**3.3 Q B J**	**2.6 B J**	**12 B**	**12 B**
1,2,3,4,7,8-HxCDD		0.057 U	0.051 U	0.087 U	**0.44 Q J**	0.094 U	0.073 U	0.073 U	0.065 U
1,2,3,4,7,8-HxCDF		0.057 U	0.066 U	**0.66 J**	**2.6 J**	0.06 U	0.063 U	0.075 U	0.065 U
1,2,3,4,7,8,9-HpCDF		0.19 U	0.053 U	0.1 U	**0.7 Q J**	0.09 U	0.074 U	0.1 U	0.094 U
1,2,3,6,7,8-HxCDD		0.056 U	0.056 U	**1.3 Q J**	**3.6 J**	**0.39 J**	0.08 U	**0.41 Q J**	**0.29 Q J**
1,2,3,6,7,8-HxCDF		0.052 U	0.059 U	**1.1 Q J**	**4.5 Q J**	**0.23 Q J**	0.39 Q J	**0.88 Q J**	**0.53 Q J**
1,2,3,7,8-PeCDD		0.055 U	0.044 U	0.082 U	**1.3 Q J**	0.076 U	0.08 U	0.075 U	0.092 U
1,2,3,7,8-PeCDF		0.054 U	0.06 U	0.077 U	0.094 U	0.064 U	0.066 U	0.079 U	0.059 U
1,2,3,7,8,9-HxCDD		0.053 U	0.05 U	**0.36 Q J**	**1.6** J	0.087 U	0.071 U	0.069 U	0.059 U
1,2,3,7,8,9-HxCDF		0.063 U	0.069 U	0.083 U	0.12 U	0.082 U	0.077 U	0.095 U	0.086 U
2,3,4,6,7,8-HxCDF		0.052 U	0.058 U	0.065 U	**0.4 Q J**	0.064 U	0.062 U	0.076 U	0.069 U
2,3,4,7,8-PeCDF		0.047 U	0.051 U	0.071 U	**0.72 J**	0.062 U	0.06 U	0.07 U	0.055 U
2,3,7,8-TCDD		0.046 U	0.043 U	**0.47 Q J**	**0.35 Q J**	0.085 U	0.12 U	0.083 U	0.083 U
2,3,7,8-TCDF		**0.13 Q J**	0.077 U	**2.8**	**0.96 J**	0.09 U	**0.56 Q J**	**0.33 Q J**	**0.3 J**
OCDD		**0.6 B J**	**0.68 B J**	**52 B**	**110 B**	**5.2 Q B J**	**13 B**	**30 B**	**13 Q B**
OCDF		**0.12 B J**	**0.12 Q B J**	**4.5 B J**	**25 B**	**0.88 B J**	**1.2 B J**	**3.9 B J**	**2.6 B J**
Total HpCDD		0.1 U	0.082 U	**16**	**32**	**2.2 J Q**	**3.9 J Q**	**9.3**	**3.7 Q J**
Total HpCDF		0.17 U	0.048 U	**29 B**	**160 B Q**	**7 J Q B**	**5.6 J B**	**24 B**	**23 B**
Total HxCDD		0.055 U	0.052 U	**6.4 J Q**	**21 Q**	**1.8 J**	**1.3 J Q**	**3 J Q**	**2.4 J Q**
Total HxCDF		0.055 U	0.063 U	**16 Q**	**65 Q**	**3.5 Q J**	**4.1 J Q**	**11 Q**	**9.6 Q**
Total PeCDD		0.055 U	0.044 U	**1.8 Q J**	**8.5 J Q**	**0.84 Q J**	0.08 U	**0.74 Q J**	0.092 U
Total PeCDF		0.05 U	**0.31 Q J**	**5.9 J Q**	**14 J Q**	**1 Q J**	**1.1 J Q**	**1.4 Q J**	**1 Q J**
Total TCDD		0.046 U	0.043 U	**2.2 B J Q**	**4.5 B Q**	**0.95 B J Q**	**1.4 Q B J**	**1.4 Q B J**	**1.1 Q B J**
Total TCDF		**2.8 Q**	**4.8 Q**	**20 Q**	**23 Q**	**5.6 Q**	**6.2 Q**	**5.9 Q**	**8 Q**

*Results are on an as-received (wet-weight) basis, and have not been corrected for dry weight or % lipids.

B - The analyte is present in the associated method blank at a detectable level.
J - The reported result is an estimata
Q - Estimated maximum possible concentration.
U - Not detected
TEQ calculated with non-detect values (U) being 0

APPENDIX P

Test America Lab Report

APPENDIX Q

Test America Lab Report

APPENDIX R

Test America Lab Report

APPENDIX S

Test America Lab Report

Appendix C

Disposal Documentation

VEOLIA
ENVIRONMENTAL SERVICES

WASTESTREAM INFORMATION PROFILE

☐ Recertification

Disposal Code

Veolia ES LOCATION
☐ Invoice Address
☐ Manifest from – blank if direct

ADDRESS _____ CITY _____ ST ___

Veolia ES TSDF requested ____ Technology requested ____ Generator No.____ **Generator EPA ID No.** MND982612368

1. **Generator Name** MPCA-Duluth **Generator State No.** ____
Address 525 South Lake Ave, Suite **State Wastestream No.**
City Duluth **State** MN **Country** USA **ZIP** 55802
NAICS (SIC) Code ____ ____ ____ **Source** ____ **Origin** ____ **Form** ____ **System Type**

2. **Waste Name** SLR Sediment **Lab or Waste Area**____

3. **Process Generating Waste** Investigation river sediment sampling

4. **Shipping Name** Non DOT, Non RCRA Hazardous Waste
Hazard Class ____ **UN/NA No.**____ **PG**__ **RQ amt**____**lb**

RQ Desc:	1.	2.
DOT Desc:	1.	2.

5. **Waste Codes** ____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____ ____

Wastewater ☐ **Non Wastewater** ☐ **Sub Category**

6. **Physical and chemical properties** *(check all that apply)*

pH
a ☐ < 2
b ☐ 2 - 5
c ☒ 5 - 9
d ☐ 9 - 12.5
e ☐ > 12.5
____ exact

Specific Gravity
a ☐ < .8
b ☐ .8 - 1.0
c ☐ 1.0
d ☐ 1.0 - 1.2
e ☒ > 1.2
____ exact

Flash Point (F)
a ☐ < 80
b ☐ 80 - 100
c ☐ 101 - 140
d ☐ 141 - 200
e ☐ > 200
f ☒ no flash ____exact

Solids
____ % suspended
____ % settleable
____ % dissolved
____ % ash
____ water solubility
____ BTU/lb

Free Liquid Range 0% to 0% %

Physical State
s ☒ solid
m ☐ semi-solid
l ☐ liquid
p ☐ pumpable semi-solid
f ☐ flowable powder
g ☐ gas
a ☐ aerosol
r ☐ pressurized liquid
d ☐ debris per 40 CFR 268.45
h ☐ sharps

Hazardous Characteristics
a ☐ air reactive
w ☐ water reactive
c ☐ cyanide reactive
f ☐ sulfide reactive
e ☐ explosive
o ☐ oxidizing acid
p ☐ peroxide former
r ☐ radioactive or NRC regulated
s ☐ shock sensitive
t ☐ temp sensitive
m ☐ polymerization/monomer
n ☐ OSHA carcinogen
I ☐ infectious
h ☐ inhalation hazard Zone: NO

Odor
a none ☒
b mild ☐
c strong ☐
describe

Halogens
Br 0 % Bromine
Cl 0 % Chlorine
F 0 % Fluorine
I 0 % Iodine

Layers:	a ☐ **multilayered:**	b ☐ **bi-layered:**	c ☒ **single phase:**
	Top Layer	Second Layer	Bottom Layer
Viscosity by Layer:	☐ high (syrup) ☐ medium (oil) ☐ low (water) ☐ solid	☐ high (syrup) ☐ medium (oil) ☐ low (water) ☐ solid	☐ high (syrup) ☐ medium (oil) ☐ low (water) ☒ solid

Color

Used oil y(n) **HOC <1000 ppm** ☐ **or > 1000 ppm** ☐

WIP No.

7. **Chemical Composition** [**M** = Marine Pollutant, **S** - Severe Marine Pollutant, **O** = Ozone Depleting Substance, **U** = Underlying Hazardous Constituent, **B** = Benzene NESHAP, **T** = TRI Chemical, **C** = **OSHA** Carcinogen]

	Constituents	Range	Units		Constituents	Range	Units
	River sediment	100	%				

Total Composition Must Equal or Exceed 100%

Other:

8. Is the wastestream being imported into the USA? Yes ☐ No ☒
9. Does the wastestream contain PCBs regulated by 40CFR? Yes ☐ No ☒
   PCB concentration _____ppm
10. Is the wastestream subject to the Marine Pollutant Regulations? Yes ☐ No ☒
11. Is the wastestream subject to Benzene NESHAP? Yes ☐ No ☒
    If yes, is the wastestream subject to Notification and Control Requirements? Yes ☐ No ☒
    Benzene concentration _____ ppm
12. Is the wastestream subject to RCRA subpart CC controls? Yes ☐ No ☒
    Volatile organic concentration, if known _____ ppmw
    CC approved analytical method ☐ Generator Knowledge ☐
13. Is the wastestream from a CERCLA or state mandated cleanup? Yes ☐ No ☒

14. **Container Information** (*Identify UN container marking if known*)
    Packaging: **Bulk Solid** ☐ Type/Size: _____ **Bulk Liquid** ☐ Type/Size: _____ **Drum** ☒ Type/Size: DM/55 gallon
    Other
    Shipping Frequency: Units 2 Per Month ☐ Quarter ☐ Year ☐ One Time ☒ Other

15. **Additional Information**:

Site:
St. Louis River Reservoirs (SLR)
Duluth, MN 55802

Is analytical or an MSDS available that describes the waste? Yes ☒ No ☐ If yes, please attach.

GENERATOR CERTIFICATION

I hereby certify that all information submitted in this and all attached documents contains true and accurate descriptions of this waste. Any sample submitted is representative as defined in 40 CFR 261 - Appendix I or by using an equivalent method. All relevant information regarding known or suspected hazards in the possession of the generator has been disclosed. I authorize sampling of any waste shipment for purposes of recertification.

Heidi Bauman	218-302-6607	11/29/16
NAME (PRINT OR TYPE)	**PHONE**	**DATE**
Heidi Bauman	Project Manager	
SIGNATURE	**TITLE**	

FACILITY NOTIFICATION

If approved for management, Veolia ES has all the necessary permits and licenses for the waste that has been characterized and identified by this profile.

TSDF PROCESSING USE ONLY: **PPE REQUIRED** No _____ Yes _____ Describe _____

WIP No.

e-NON-HAZARDOUS WASTE MANIFEST

1 Generator ID Number	2 Page 1 of	3 Emergency Response Phone	4 Waste Tracking Number
MND982612368	1	ER: 800-451 8346	MW16132

GENERATOR

5 Generator's Name and Mailing Address
MPCA-Duluth
525 South Lake Ave, Suite 400
Duluth, MN 55802

Generator's Site Address (if different than mailing address)
SLR AOC
St Louis River Reservoirs
Duluth MN 55802

Generator's Phone 218 723 4837

6 Transporter 1 Company Name — U.S. EPA ID Number
BAY MERT LLC — MND982720547

7 Transporter 2 Company Name — U.S. EPA ID Number
Veolia ES Technical Solutions-MI — MIT080631369

8 Designated Facility Name and Site Address — U.S. EPA ID Number
Veolia ES Technical Solutions-WD
W124 N9452 Boundary Road
Menomonee Falls WI 53051
Facility's Phone 800-255-5096 — WID003967148

9 Waste Shipping Name and Description	10 Containers No	10 Containers Type	11 Total Quantity	12 Unit Wt./Vol.
1 Non DOT, Non PCBs Hazardous Waste,	01	DM	20	P
2 Non-DOT, Non RCRA Hazardous Waste	01	DF	20	P
3				
4				

13 Special Handling Instructions and Additional Information
1) 068821 - SLR Sediment 2.) 068821 - SLR Sediment
Job # 1160139 1
ER phone # is contracted by Bay Mert with 2K 'contract# 55671

14. GENERATOR'S/OFFEROR'S CERTIFICATION. I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labeled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Generator's/Offeror's Printed/Typed Name	Signature	Month	Day	Year
[illegible]	[illegible]	12	13	16

INTL

15 International Shipments ☐ Import to U.S. ☐ Export from U.S. Port of entry/exit
Transporter Signature (for exports only) Date leaving U.S.

TRANSPORTER

16 Transporter Acknowledgment of Receipt of Materials

Transporter 1 Printed/Typed Name	Signature	Month	Day	Year
Jim Leisz	[signature]	12	13	16
Transporter 2 Printed/Typed Name	Signature	Month	Day	Year

DESIGNATED FACILITY

17 Discrepancy
17a Discrepancy Indication Space ☐ Quantity ☐ Type ☐ Residue ☐ Partial Rejection ☐ Full Rejection
Manifest Reference Number
17b Alternate Facility (or Generator) U.S. EPA ID Number
Facility's Phone
17c Signature of Alternate Facility (or Generator) Month Day Year

18 Designated Facility Owner or Operator: Certification of receipt of materials covered by the manifest except as noted in Item 17a

Printed/Typed Name	Signature	Month	Day	Year
Robert L Kunn Jr	[signature]	12	28	16

169-BLS-C 6 10497 (Rev. 9/09) COPY

Appendix D

Laboratory Analytical Reports

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365196

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/18/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☒	☐	☐	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☐	☒	☐	

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☐	☒	

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☐	☒	☐	
b.	Are the lab recovery limits specified on the report?		☐	☐	☒	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☐	☐	☒	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☐	☒	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	

	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question			Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.		☒	☐	☐	
	i.	Have the required matrix spikes been prepared and reported?	☒	☐	☐	
	ii.	If no, is there and explanation in the report as to why?	☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?	☐	☐	☒	
	iv.	Are the lab limits specified on the report?	☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
	vi.	Are there compounds outside the lab limits?	☐	☒	☐	
		1. If yes, are the analytes above the lab limits?	☐	☐	☒	
		2. Below the lab limits?	☐	☐	☒	
		3. Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.		☐	☒	☐	RPDs discussed apply to MS/MSDs.
	i.	Is the RPD for the duplicate pair within the lab limits?	☒	☐	☐	
	ii.	If no, has the associated source sample been flagged?	☐	☐	☒	
c.	What is the impact of failed QC on this project?		☐	☐	☒	

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Samples BW16SR-005-0.0-0.15 and BW16SR-115-0.0-0.15 were collected as blind field duplicates. The mercury RPD (17.8%) was < the QC guideline of 50%. No data were qualified.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

October 18, 2016

Nancy McDonald
Bay West Inc
5 Empire Drive
Saint Paul, MN 55103

RE: Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Dear Nancy McDonald:
Enclosed are the analytical results for sample(s) received by the laboratory on October 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Lori Castille
lori.castille@pacelabs.com
Project Manager

Enclosures

cc: Paul Raymaker, Bay West
Jeff Smith, Pace Analytical Services, Inc

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

CERTIFICATIONS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
Alaska Certification UST-107
525 N 8th Street, Salina, KS 67401
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Virginia/VELAP Certification #: Pace
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

SAMPLE SUMMARY

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10365196001	**BW16SR-005-0.0-0.15**	Solid	09/30/16 11:20	10/06/16 20:25
10365196002	**BW16SR-115-0.0-0.15**	Solid	09/30/16 12:20	10/06/16 20:25

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SAMPLE ANALYTE COUNT

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Lab ID	Sample ID	Method	Analysts	Analytes Reported
10365196001	**BW16SR-005-0.0-0.15**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365196002	**BW16SR-115-0.0-0.15**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1

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PROJECT NARRATIVE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Method: **EPA 7471B**
Description: 7471B Mercury
Client: Bay West, Inc.
Date: October 18, 2016

General Information:
2 samples were analyzed for EPA 7471B. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

Hold Time:
The samples were analyzed within the method required hold times with any exceptions noted below.

Sample Preparation:
The samples were prepared in accordance with EPA 7471B with any exceptions noted below.

Initial Calibrations (including MS Tune as applicable):
All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:
All criteria were within method requirements with any exceptions noted below.

Method Blank:
All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

Laboratory Control Spike:
All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

Additional Comments:

This data package has been reviewed for quality and completeness and is approved for release.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Sample: BW16SR-005-0.0-0.15 **Lab ID: 10365196001** Collected: 09/30/16 11:20 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.11**	mg/kg	0.042	0.011	1	10/07/16 11:15	10/17/16 16:19	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**52.4**	%	0.10	0.10	1		10/10/16 15:29		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Sample: BW16SR-115-0.0-0.15 **Lab ID: 10365196002** Collected: 09/30/16 12:20 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.092**	mg/kg	0.037	0.0096	1	10/07/16 11:15	10/17/16 16:21	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**53.8**	%	0.10	0.10	1		10/10/16 15:29		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

QC Batch: 439774 Analysis Method: EPA 7471B
QC Batch Method: EPA 7471B Analysis Description: 7471B Mercury Solids
Associated Lab Samples: 10365196001, 10365196002

METHOD BLANK: 2390958 Matrix: Solid
Associated Lab Samples: 10365196001, 10365196002

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/kg	ND	0.017	0.0045	10/17/16 15:45	

LABORATORY CONTROL SAMPLE: 2390959

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/kg	.45	0.47	103	80-120	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2390960 2390961

Parameter	Units	10365188006 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/kg	0.12	.68	.68	0.77	0.84	95	106	75-125	9	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

QC Batch: 440231
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365196001, 10365196002

SAMPLE DUPLICATE: 2394092

Parameter	Units	10365310003 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	3.9	4.1	4	30	

SAMPLE DUPLICATE: 2394137

Parameter	Units	10365183004 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	26.9	29.4	9	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALIFIERS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365196

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10365196001	**BW16SR-005-0.0-0.15**	EPA 7471B	439774	EPA 7471B	440061
10365196002	**BW16SR-115-0.0-0.15**	EPA 7471B	439774	EPA 7471B	440061
10365196001	**BW16SR-005-0.0-0.15**	ASTM D2974	440231		
10365196002	**BW16SR-115-0.0-0.15**	ASTM D2974	440231		

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365196

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-3
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0.15	SO	G	3/12/15	1204													
1																			
2	BW16SR-005	BW16SR-005-0.0-0.15	SO	G	9/30/16	1120	3	3								1	1	1	a-1
3	BW16SR-005	BW16SR-115-0.0-0.15	SO	G	9/30/16	1220	2	2								1	1		a-2 Collect % moisture from 4 oz jar
4																			
5																			
6																			
7																			
8																			
9																			
10																			
11																			
12																			

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16		10/6/16	1444	Kristine Polson	10/6/16	1445	4.9	Y	N	Y
	Kristine Polson	10/6/16	1715		10/6/16	1715				
		10/6/16	2025	Client Pace	10-6-16	20:25	Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: CM DATE Signed (MM/DD/YY): 10/6/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: WO# : 10365196

10365196

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ SpeeDee ☐ Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☐ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.9, 0.6, 4.7 **Cooler Temp Corrected (°C):** 0.2, 0.8, 4.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** CAT 10.6.16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☑ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL	☑ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	Sample #
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☒ N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: **Date:** 10/7/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10367136

Work order number: 3000017136 Report date (mm/dd/yyyy): 11/04/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.		Is there a chain of custody (COC) with the report?	☒	☐	☐	COC includes samples for Scanlon Reservoir, Thomson and Boulder Lake. This data review checklist only applies to Scanlon Reservoir samples.
b.		Is there a sample condition form with the report?	☒	☐	☐	
c.		Were there samples requiring preservation?	☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.		Were samples received in the correct containers?	☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.		Were samples received with adequate holding time for sample prep for all requested analyses?	☒	☐	☐	
f.		Are there notes about sample condition or holding time issues on the COC? Explain impact.	☐	☒	☐	
g.		Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.	☐	☒	☐	

2. Calibration

Question			Yes	No	N/A	Comments
a.		Do the report narrative or data qualifiers indicate	☒	☐	☐	

	calibration problems for any analyses? If yes, explain the data impact.				

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	Low-level concentrations of Total TCDD, 1,2,3,4,6,7,8-HpCDD, Total HpCDD, and OCDD were detected in the method blank 52558.
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☒	☐	All sample results were > 10x the blank concentrations.

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☒	☐	☐	Dioxins/furans have internal standards instead of surrogates.
b.	Are the lab recovery limits specified on the report?		☒	☐	☐	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☒	☐	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	

	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	
	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question				Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.			☐	☒	☐	
	i.	Have the required matrix spikes been prepared and reported?		☐	☐	☒	
	ii.	If no, is there and explanation in the report as to why?		☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?		☐	☐	☒	
	iv.	Are the lab limits specified on the report?		☐	☐	☒	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?		☐	☐	☒	
	vi.	Are there compounds outside the lab limits?		☐	☐	☒	
		1.	If yes, are the analytes above the lab limits?	☐	☐	☒	
		2.	Below the lab limits?	☐	☐	☒	
		3.	Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.			☐	☐	☒	
	i.	Is the RPD for the duplicate pair within the lab limits?		☐	☐	☒	
	ii.	If no, has the associated source sample been flagged?		☐	☐	☒	
c.	What is the impact of failed QC on this project?			☐	☐	☒	

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Interfering substances impacted the determinations of PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where diphenyl ethers were present. All results flagged “I” or “P” were qualified “J” as estimated by the reviewer. The laboratory flagged concentrations > the calibration range “E”. These results were qualified “J” as estimated by the reviewer. Concentrations below the calibration range were flagged “J” as estimated by the laboratory.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

www.pacelabs.com

Report Prepared for:

Nancy McDonald
Bay West, Inc.
5 Empire Drive
Saint Paul MN 55103

REPORT OF LABORATORY ANALYSIS FOR PCDD/PCDF

Report Information:

Pace Project #: 10367136
Sample Receipt Date: 10/21/2016
Client Project #: J160139 SLR Sediment AOCs
Client Sub PO #: 108002
State Cert #: 027-053-137

Invoicing & Reporting Options:

The report provided has been invoiced as a Level 2 PCDD/PCDF Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

This report has been reviewed by:

November 29, 2016

Carolynne Trout, Project Manager
(612) 607-6351
(612) 607-6444 (fax)
Carolynne.Trout@pacelabs.com

Report Prepared Date:
November 29, 2016

Report of Laboratory Analysis

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

DISCUSSION

This report presents the results from the analyses performed on seven samples submitted by a representative of BayWest, Inc. The samples were analyzed for the presence or absence of polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs) using a modified version of USEPA Method 8290. The reporting limits were based on signal-to-noise measurements. Estimated Maximum Possible Concentration (EMPC) values were treated as positives in the toxic equivalence calculations. This report was revised to exclude results from a second analysis of sample BW16TR-008-0.0-0.15.

Second column confirmation analyses of 2,3,7,8-TCDF values obtained from the primary (DB5-MS) column are performed only when specifically requested for a project and only when the values are above the concentration of the lowest calibration standard. Typical resolution for this isomer using the DB5-MS column ranges from 25-30%.

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extracts ranged from 53-99%. All of the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. Also, since the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained.

Values were flagged "I" where incorrect isotope ratios were obtained and "P" where diphenylethers were present at the elution times of PCDFs. Concentrations below the calibration range were flagged "J" and should be regarded as estimates. Levels above the calibration range were flagged "E" and should be regarded as estimated concentrations.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to contain trace levels of selected congeners. These levels were below the calibration range of the method. The levels reported for the affected congeners in the field samples were higher than the corresponding blank levels by one or more orders of magnitude. These results indicate that the sample processing steps did not contribute significantly to the levels reported for the field samples.

A laboratory spike sample was also prepared with the sample batch using clean reference matrix that had been fortified with native standard materials. The results show that the spiked native compounds were recovered at 85-114%. These values were within the target range for this method. Matrix spikes were prepared using sample material from a separate project. Results are available upon request.

Pace Analytical Services, Inc.
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Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Montana	92
Alaska	MN00064	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN_00064_200
Arkansas	88-0680	New Jersey (NE	MN002
California	01155CA	New York (NEL	11647
Colorado	MN00064	North Carolina	27700
Connecticut	PH-0256	North Dakota	R-036
EPA Region 8	8TMS-Q	Ohio	4150
Florida (NELAP	E87605	Oklahoma	D9922
Georgia (DNR)	959	Oregon (ELAP)	MN200001-005
Guam	959	Oregon (OREL	MN300001-001
Hawaii	SLD	Pennsylvania	68-00563
Idaho	MN00064	Puerto Rico	MN00064
Illinois	200012	Saipan	MP0003
Indiana	C-MN-01	South Carolina	74003001
Indiana	C-MN-01	Tennessee	TN02818
Iowa	368	Texas	T104704192-08
Kansas	E-10167	Utah (NELAP)	MN00064
Kentucky	90062	Virginia	00251
Louisiana	03086	Washington	C755
Maine	2007029	West Virginia #	9952C
Maryland	322	West Virginia D	382
Michigan	9909	Wisconsin	999407970
Minnesota	027-053-137	Wyoming	8TMS-Q

REPORT OF LABORATORY ANALYSIS

Report No.....10367136

Appendix A

Sample Management

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Mailee Garton - Great Lake Environmental Center	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker - Bay West	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103	Nancy McDonald - Bay West	Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-ToxBio-02
Email To: mgarton@glec.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 231-941-2230	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking DW; Water W; Waste Water WW; Product P; Soil/Solid SO; Oil OL; Wipe WP; Air AR; Tissue TS; Other OT

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (7471B)	% Moisture	TOC (SW-846 9060A Quad Burn)	Comments
Ex	BW16MLW-005	BW14MLW-005-0-0.15	SO	G		1204														
1	BW16SR-004	BW16SR-004-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	001
2	BW16SR-016	BW16SR-016-0.15-0.60	SO	G	10/20/16	10:00	4									x	x	x	x	002
3	BW16TR-008	BW16TR-008-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	003
4	BW16TR-013	BW16TR-013-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	004
5	BW16TR-017	BW16TR-017-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	005
6	BW16TR-018	BW16TR-018-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	006
7	BW16BLR-001	BW16BLR-001-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	007
8																				
9																				
10																				
11																				
12																				

* 5 pm 10/20/16

Separate Cooler

Ref: pace.tox lab
Dep:

Date: 20Oct16
Wgt: 5.00 LBS

DV:

SHIPPING:	6.13
SPECIAL:	0.00
HANDLING:	0.00
0.00 TOTAL:	6.13

Svcs: PRIORITY OVERNIGHT
TRCK: 9802 5316 5172

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16	Mailee Garton/GLEC	10/20/16	14:00	PACE	10/21/16	9:45	0.7 0.6	Y	*	Y

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Mailee Garton

SIGNATURE of SAMPLER: Mailee Garton DATE Signed (MM/DD/YY): 10/20/16

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: F-MN-L-213-rev.17	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt **Client Name:** Bay West LLC **Project #:** WO#: 10367136

Courier: ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ SpeeDee ☐ Other:

Tracking Number: 9802 5318 5161 9802 5318 5172

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

Thermometer Used: ☒ 151401163 ☐ B88A912167504 ☐ 151401164 ☐ B88A0143310098 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.6, 0.0 **Cooler Temp Corrected (°C):** 0.8, 0.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☒ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** BC 10/21/16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL	☒ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☒ N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: Carolynne Trout **Date:** 10/24/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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Tel: 612-607-1700
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Reporting Flags

A = Reporting Limit based on signal to noise

B = Less than 10x higher than method blank level

C = Result obtained from confirmation analysis

D = Result obtained from analysis of diluted sample

E = Exceeds calibration range

I = Interference present

J = Estimated value

Nn = Value obtained from additional analysis

P = PCDE Interference

R = Recovery outside target range

S = Peak saturated

U = Analyte not detected

V = Result verified by confirmation analysis

X = %D Exceeds limits

Y = Calculated using average of daily RFs

* = See Discussion

Appendix B

Sample Analysis Summary

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-004-0.0-0.15		
Lab Sample ID	10367136001		
Filename	F161101B_11		
Injected By	SMT		
Total Amount Extracted	18.6 g	Matrix	Solid
% Moisture	58.7	Dilution	NA
Dry Weight Extracted	7.68 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 21:43

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	15.0	-----	0.29		2,3,7,8-TCDF-13C	2.00	80
Total TCDF	43.0	-----	0.29		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	80
2,3,7,8-TCDD	3.5	-----	0.21		2,3,4,7,8-PeCDF-13C	2.00	73
Total TCDD	22.0	-----	0.21		1,2,3,7,8-PeCDD-13C	2.00	79
					1,2,3,4,7,8-HxCDF-13C	2.00	93
1,2,3,7,8-PeCDF	-----	1.2	0.13	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	77
2,3,4,7,8-PeCDF	3.6	-----	0.21	J	2,3,4,6,7,8-HxCDF-13C	2.00	86
Total PeCDF	58.0	-----	0.17		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	80
1,2,3,7,8-PeCDD	4.2	-----	0.22	J	1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	51.0	-----	0.22		1,2,3,4,6,7,8-HpCDF-13C	2.00	60
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61
1,2,3,4,7,8-HxCDF	-----	15.0	4.70	P	1,2,3,4,6,7,8-HpCDD-13C	2.00	72
1,2,3,6,7,8-HxCDF	19.0	-----	0.82		OCDD-13C	4.00	67
2,3,4,6,7,8-HxCDF	7.9	-----	0.29				
1,2,3,7,8,9-HxCDF	3.8	-----	0.37	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	560.0	-----	1.60		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	7.4	-----	0.37		2,3,7,8-TCDD-37Cl4	0.20	87
1,2,3,6,7,8-HxCDD	55.0	-----	0.72				
1,2,3,7,8,9-HxCDD	16.0	-----	0.44				
Total HxCDD	350.0	-----	0.51				
1,2,3,4,6,7,8-HpCDF	870.0	-----	0.74		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	15.0	-----	0.84		Equivalence: 45 ng/Kg		
Total HpCDF	1900.0	-----	0.79		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	990.0	-----	2.40				
Total HpCDD	2000.0	-----	2.40				
OCDF	860.0	-----	0.56				
OCDD	11000.0	-----	0.39	E			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
P = PCDE Interference
E = Exceeds calibration range
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-016-0.15-0.60		
Lab Sample ID	10367136002		
Filename	F161101B_12		
Injected By	SMT		
Total Amount Extracted	17.5 g	Matrix	Solid
% Moisture	44.5	Dilution	NA
Dry Weight Extracted	9.71 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 22:31

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	12.0	-----	0.70		2,3,7,8-TCDF-13C	2.00	79
Total TCDF	68.0	-----	0.70		2,3,7,8-TCDD-13C	2.00	86
					1,2,3,7,8-PeCDF-13C	2.00	74
2,3,7,8-TCDD	6.1	-----	0.34		2,3,4,7,8-PeCDF-13C	2.00	63
Total TCDD	53.0	-----	0.34		1,2,3,7,8-PeCDD-13C	2.00	70
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	-----	7.2	0.24	P	1,2,3,6,7,8-HxCDF-13C	2.00	78
2,3,4,7,8-PeCDF	17.0	-----	0.40		2,3,4,6,7,8-HxCDF-13C	2.00	85
Total PeCDF	240.0	-----	0.32		1,2,3,7,8,9-HxCDF-13C	2.00	82
					1,2,3,4,7,8-HxCDD-13C	2.00	81
1,2,3,7,8-PeCDD	23.0	-----	0.13		1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	190.0	-----	0.13		1,2,3,4,6,7,8-HpCDF-13C	2.00	62
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	72.0	-----	0.58		1,2,3,4,6,7,8-HpCDD-13C	2.00	74
1,2,3,6,7,8-HxCDF	110.0	-----	0.80		OCDD-13C	4.00	61
2,3,4,6,7,8-HxCDF	19.0	-----	0.53				
1,2,3,7,8,9-HxCDF	11.0	-----	0.66		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	2500.0	-----	0.64		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	17.0	-----	0.82		2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	100.0	-----	0.84				
1,2,3,7,8,9-HxCDD	67.0	-----	0.71				
Total HxCDD	900.0	-----	0.79				
1,2,3,4,6,7,8-HpCDF	4300.0	-----	0.37	E	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	34.0	-----	2.90		Equivalence: 130 ng/Kg		
Total HpCDF	8300.0	-----	1.70	E	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	850.0	-----	1.40				
Total HpCDD	1700.0	-----	1.40				
OCDF	2000.0	-----	0.48				
OCDD	6700.0	-----	0.28				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
P = PCDE Interference
E = Exceeds calibration range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-008-0.0-0.15		
Lab Sample ID	10367136003		
Filename	F161101B_13		
Injected By	SMT		
Total Amount Extracted	18.2 g	Matrix	Solid
% Moisture	42.4	Dilution	NA
Dry Weight Extracted	10.5 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 23:19

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	0.74	-----	0.49	J	2,3,7,8-TCDF-13C	2.00	74
Total TCDF	2.50	-----	0.49		2,3,7,8-TCDD-13C	2.00	82
					1,2,3,7,8-PeCDF-13C	2.00	78
2,3,7,8-TCDD	ND	-----	0.54		2,3,4,7,8-PeCDF-13C	2.00	71
Total TCDD	2.20	-----	0.54		1,2,3,7,8-PeCDD-13C	2.00	74
					1,2,3,4,7,8-HxCDF-13C	2.00	84
1,2,3,7,8-PeCDF	ND	-----	0.44		1,2,3,6,7,8-HxCDF-13C	2.00	76
2,3,4,7,8-PeCDF	0.97	-----	0.35	J	2,3,4,6,7,8-HxCDF-13C	2.00	83
Total PeCDF	9.40	-----	0.40		1,2,3,7,8,9-HxCDF-13C	2.00	77
					1,2,3,4,7,8-HxCDD-13C	2.00	79
1,2,3,7,8-PeCDD	0.35	-----	0.31	J	1,2,3,6,7,8-HxCDD-13C	2.00	59
Total PeCDD	26.00	-----	0.31		1,2,3,4,6,7,8-HpCDF-13C	2.00	58
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	3.30	-----	0.51	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66
1,2,3,6,7,8-HxCDF	3.30	-----	0.26	J	OCDD-13C	4.00	55
2,3,4,6,7,8-HxCDF	2.20	-----	0.28	J			
1,2,3,7,8,9-HxCDF	-----	0.82	0.25	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	150.00	-----	0.32		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.50		2,3,7,8-TCDD-37Cl4	0.20	78
1,2,3,6,7,8-HxCDD	75.00	-----	0.60				
1,2,3,7,8,9-HxCDD	26.00	-----	0.37				
Total HxCDD	520.00	-----	0.49				
1,2,3,4,6,7,8-HpCDF	260.00	-----	0.33		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	2.00	-----	0.31	J	Equivalence: 16 ng/Kg		
Total HpCDF	470.00	-----	0.32		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	91.00	-----	0.39				
Total HpCDD	190.00	-----	0.39				
OCDF	87.00	-----	0.20				
OCDD	320.00	-----	0.21				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
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NC = Not Calculated

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I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-013-0.0-0.15		
Lab Sample ID	10367136004		
Filename	F161101B_14		
Injected By	SMT		
Total Amount Extracted	18.9 g	Matrix	Solid
% Moisture	53.5	Dilution	NA
Dry Weight Extracted	8.79 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 00:07

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	1.40	-----	0.42		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	5.60	-----	0.42		2,3,7,8-TCDD-13C	2.00	83
					1,2,3,7,8-PeCDF-13C	2.00	79
2,3,7,8-TCDD	ND	-----	0.31		2,3,4,7,8-PeCDF-13C	2.00	74
Total TCDD	6.40	-----	0.31		1,2,3,7,8-PeCDD-13C	2.00	74
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	0.78	-----	0.32	J	1,2,3,6,7,8-HxCDF-13C	2.00	79
2,3,4,7,8-PeCDF	1.20	-----	0.39	J	2,3,4,6,7,8-HxCDF-13C	2.00	85
Total PeCDF	16.00	-----	0.35		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	75
1,2,3,7,8-PeCDD	-----	0.80	0.53	IJ	1,2,3,6,7,8-HxCDD-13C	2.00	62
Total PeCDD	9.70	-----	0.53		1,2,3,4,6,7,8-HpCDF-13C	2.00	58
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	4.00	-----	0.98	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66
1,2,3,6,7,8-HxCDF	8.90	-----	0.36		OCDD-13C	4.00	57
2,3,4,6,7,8-HxCDF	2.80	-----	0.36	J			
1,2,3,7,8,9-HxCDF	-----	0.86	0.65	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	190.00	-----	0.59		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.73	-----	0.29	J	2,3,7,8-TCDD-37Cl4	0.20	77
1,2,3,6,7,8-HxCDD	6.10	-----	0.26				
1,2,3,7,8,9-HxCDD	2.30	-----	0.34	J			
Total HxCDD	55.00	-----	0.30				
1,2,3,4,6,7,8-HpCDF	320.00	-----	0.53		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	2.30	-----	0.50	J	Equivalence: 8.4 ng/Kg		
Total HpCDF	600.00	-----	0.51		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	85.00	-----	0.83				
Total HpCDD	190.00	-----	0.83				
OCDF	160.00	-----	0.19				
OCDD	1100.00	-----	0.28				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

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NC = Not Calculated

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I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-017-0.0-0.15		
Lab Sample ID	10367136005		
Filename	F161101B_15		
Injected By	SMT		
Total Amount Extracted	18.8 g	Matrix	Solid
% Moisture	58.9	Dilution	NA
Dry Weight Extracted	7.73 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 00:56

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	2.10	-----	0.30		2,3,7,8-TCDF-13C	2.00	79
Total TCDF	9.70	-----	0.30		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	85
2,3,7,8-TCDD	-----	0.35	0.20	IJ	2,3,4,7,8-PeCDF-13C	2.00	81
Total TCDD	5.10	-----	0.20		1,2,3,7,8-PeCDD-13C	2.00	83
					1,2,3,4,7,8-HxCDF-13C	2.00	89
1,2,3,7,8-PeCDF	0.57	-----	0.30	J	1,2,3,6,7,8-HxCDF-13C	2.00	85
2,3,4,7,8-PeCDF	0.84	-----	0.22	J	2,3,4,6,7,8-HxCDF-13C	2.00	91
Total PeCDF	14.00	-----	0.26		1,2,3,7,8,9-HxCDF-13C	2.00	85
					1,2,3,4,7,8-HxCDD-13C	2.00	81
1,2,3,7,8-PeCDD	0.65	-----	0.37	J	1,2,3,6,7,8-HxCDD-13C	2.00	65
Total PeCDD	12.00	-----	0.37		1,2,3,4,6,7,8-HpCDF-13C	2.00	60
					1,2,3,4,7,8,9-HpCDF-13C	2.00	62
1,2,3,4,7,8-HxCDF	2.80	-----	0.41	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	69
1,2,3,6,7,8-HxCDF	4.40	-----	0.35	J	OCDD-13C	4.00	59
2,3,4,6,7,8-HxCDF	1.80	-----	0.50	J			
1,2,3,7,8,9-HxCDF	0.88	-----	0.39	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	90.00	-----	0.41		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.67	-----	0.33	J	2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	5.20	-----	0.30	J			
1,2,3,7,8,9-HxCDD	2.30	-----	0.26	J			
Total HxCDD	47.00	-----	0.30				
1,2,3,4,6,7,8-HpCDF	140.00	-----	0.48		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	1.80	-----	0.33	J	Equivalence: 6.1 ng/Kg		
Total HpCDF	280.00	-----	0.40		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	95.00	-----	0.66				
Total HpCDD	220.00	-----	0.66				
OCDF	100.00	-----	0.50				
OCDD	1300.00	-----	0.30				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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EDL = Estimated Detection Limit

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NC = Not Calculated

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I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-018-0.0-0.15		
Lab Sample ID	10367136006		
Filename	F161101B_16		
Injected By	SMT		
Total Amount Extracted	18.6 g	Matrix	Solid
% Moisture	49.9	Dilution	NA
Dry Weight Extracted	9.32 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 01:44

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	1.20	-----	0.26		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	5.00	-----	0.26		2,3,7,8-TCDD-13C	2.00	83
					1,2,3,7,8-PeCDF-13C	2.00	78
2,3,7,8-TCDD	-----	0.30	0.27	IJ	2,3,4,7,8-PeCDF-13C	2.00	71
Total TCDD	5.60	-----	0.27		1,2,3,7,8-PeCDD-13C	2.00	76
					1,2,3,4,7,8-HxCDF-13C	2.00	85
1,2,3,7,8-PeCDF	0.49	-----	0.29	J	1,2,3,6,7,8-HxCDF-13C	2.00	74
2,3,4,7,8-PeCDF	0.91	-----	0.25	J	2,3,4,6,7,8-HxCDF-13C	2.00	83
Total PeCDF	12.00	-----	0.27		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	72
1,2,3,7,8-PeCDD	-----	0.62	0.26	IJ	1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	8.70	-----	0.26		1,2,3,4,6,7,8-HpCDF-13C	2.00	55
					1,2,3,4,7,8,9-HpCDF-13C	2.00	55
1,2,3,4,7,8-HxCDF	2.60	-----	0.42	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	64
1,2,3,6,7,8-HxCDF	5.60	-----	0.60		OCDD-13C	4.00	53
2,3,4,6,7,8-HxCDF	1.70	-----	0.50	J			
1,2,3,7,8,9-HxCDF	-----	0.62	0.35	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	140.00	-----	0.47		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.53	-----	0.26	J	2,3,7,8-TCDD-37Cl4	0.20	76
1,2,3,6,7,8-HxCDD	5.30	-----	0.27	J			
1,2,3,7,8,9-HxCDD	2.20	-----	0.30	J			
Total HxCDD	44.00	-----	0.28				
1,2,3,4,6,7,8-HpCDF	230.00	-----	0.32		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	1.60	-----	0.40	J	Equivalence: 6.5 ng/Kg		
Total HpCDF	440.00	-----	0.36		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	74.00	-----	0.40				
Total HpCDD	160.00	-----	0.40				
OCDF	130.00	-----	0.51				
OCDD	910.00	-----	0.38				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
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NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
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REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16BLR-001-0.0-0.15		
Lab Sample ID	10367136007		
Filename	F161101B_17		
Injected By	SMT		
Total Amount Extracted	21.4 g	Matrix	Solid
% Moisture	82.6	Dilution	NA
Dry Weight Extracted	3.72 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 02:32

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	1.70	-----	0.59	J	2,3,7,8-TCDF-13C	2.00	87
Total TCDF	14.00	-----	0.59		2,3,7,8-TCDD-13C	2.00	94
					1,2,3,7,8-PeCDF-13C	2.00	91
2,3,7,8-TCDD	ND	-----	0.47		2,3,4,7,8-PeCDF-13C	2.00	84
Total TCDD	0.82	-----	0.47	J	1,2,3,7,8-PeCDD-13C	2.00	89
					1,2,3,4,7,8-HxCDF-13C	2.00	95
1,2,3,7,8-PeCDF	0.75	-----	0.49	J	1,2,3,6,7,8-HxCDF-13C	2.00	93
2,3,4,7,8-PeCDF	-----	0.97	0.34	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	99
Total PeCDF	9.00	-----	0.41	J	1,2,3,7,8,9-HxCDF-13C	2.00	92
					1,2,3,4,7,8-HxCDD-13C	2.00	85
1,2,3,7,8-PeCDD	0.47	-----	0.43	J	1,2,3,6,7,8-HxCDD-13C	2.00	74
Total PeCDD	1.80	-----	0.43	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	65
					1,2,3,4,7,8,9-HpCDF-13C	2.00	68
1,2,3,4,7,8-HxCDF	-----	0.69	0.41	IJ	1,2,3,4,6,7,8-HpCDD-13C	2.00	75
1,2,3,6,7,8-HxCDF	0.83	-----	0.42	J	OCDD-13C	4.00	59
2,3,4,6,7,8-HxCDF	-----	0.68	0.41	IJ			
1,2,3,7,8,9-HxCDF	ND	-----	0.70		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	6.60	-----	0.48	J	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	-----	0.46	0.45	IJ	2,3,7,8-TCDD-37Cl4	0.20	87
1,2,3,6,7,8-HxCDD	-----	1.00	0.50	IJ			
1,2,3,7,8,9-HxCDD	1.10	-----	0.42	J			
Total HxCDD	12.00	-----	0.46	J			
1,2,3,4,6,7,8-HpCDF	3.50	-----	0.50	J	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.64		Equivalence: 1.6 ng/Kg		
Total HpCDF	5.50	-----	0.57	J	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	14.00	-----	0.37				
Total HpCDD	28.00	-----	0.37				
OCDF	5.40	-----	0.71	J			
OCDD	89.00	-----	0.74				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

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REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52558	Matrix	Solid
Filename	U161101B_15	Dilution	NA
Total Amount Extracted	20.4 g	Extracted	10/27/2016 16:25
ICAL ID	U161025	Analyzed	11/02/2016 01:42
CCal Filename(s)	U161101B_03 & U161101B_19	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.031		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	ND	-----	0.031		2,3,7,8-TCDD-13C	2.00	92
					1,2,3,7,8-PeCDF-13C	2.00	85
2,3,7,8-TCDD	ND	-----	0.033		2,3,4,7,8-PeCDF-13C	2.00	80
Total TCDD	0.042	-----	0.033	J	1,2,3,7,8-PeCDD-13C	2.00	99
					1,2,3,4,7,8-HxCDF-13C	2.00	76
1,2,3,7,8-PeCDF	ND	-----	0.039		1,2,3,6,7,8-HxCDF-13C	2.00	74
2,3,4,7,8-PeCDF	ND	-----	0.023		2,3,4,6,7,8-HxCDF-13C	2.00	78
Total PeCDF	ND	-----	0.031		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	84
1,2,3,7,8-PeCDD	ND	-----	0.029		1,2,3,6,7,8-HxCDD-13C	2.00	70
Total PeCDD	ND	-----	0.029		1,2,3,4,6,7,8-HpCDF-13C	2.00	75
					1,2,3,4,7,8,9-HpCDF-13C	2.00	79
1,2,3,4,7,8-HxCDF	ND	-----	0.027		1,2,3,4,6,7,8-HpCDD-13C	2.00	90
1,2,3,6,7,8-HxCDF	ND	-----	0.023		OCDD-13C	4.00	75
2,3,4,6,7,8-HxCDF	ND	-----	0.021				
1,2,3,7,8,9-HxCDF	ND	-----	0.026		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.024		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.036		2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	ND	-----	0.035				
1,2,3,7,8,9-HxCDD	ND	-----	0.037				
Total HxCDD	ND	-----	0.036				
1,2,3,4,6,7,8-HpCDF	ND	-----	0.036		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.038		Equivalence: 0.00051 ng/Kg		
Total HpCDF	ND	-----	0.037		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	-----	0.046	0.028	IJ			
Total HpCDD	0.076	-----	0.028	J			
OCDF	ND	-----	0.055				
OCDD	-----	0.170	0.061	IJ			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

Results reported on a total weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52559		
Filename	U161101B_18	Matrix	Solid
Total Amount Extracted	20.1 g	Dilution	NA
ICAL ID	U161025	Extracted	10/27/2016 16:25
CCal Filename(s)	U161101B_03 & U161101B_19	Analyzed	11/02/2016 04:01
Method Blank ID	BLANK-52558	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.19	96	2,3,7,8-TCDF-13C	2.0	67
Total TCDF				2,3,7,8-TCDD-13C	2.0	83
				1,2,3,7,8-PeCDF-13C	2.0	77
2,3,7,8-TCDD	0.20	0.17	85	2,3,4,7,8-PeCDF-13C	2.0	73
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	90
				1,2,3,4,7,8-HxCDF-13C	2.0	70
1,2,3,7,8-PeCDF	1.0	0.97	97	1,2,3,6,7,8-HxCDF-13C	2.0	67
2,3,4,7,8-PeCDF	1.0	1.0	104	2,3,4,6,7,8-HxCDF-13C	2.0	75
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	76
				1,2,3,4,7,8-HxCDD-13C	2.0	80
1,2,3,7,8-PeCDD	1.0	0.95	95	1,2,3,6,7,8-HxCDD-13C	2.0	63
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	75
				1,2,3,4,7,8,9-HpCDF-13C	2.0	81
1,2,3,4,7,8-HxCDF	1.0	1.1	107	1,2,3,4,6,7,8-HpCDD-13C	2.0	91
1,2,3,6,7,8-HxCDF	1.0	1.0	103	OCDD-13C	4.0	78
2,3,4,6,7,8-HxCDF	1.0	0.97	97			
1,2,3,7,8,9-HxCDF	1.0	1.0	101	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.1	109	2,3,7,8-TCDD-37Cl4	0.20	81
1,2,3,6,7,8-HxCDD	1.0	1.1	114			
1,2,3,7,8,9-HxCDD	1.0	1.1	112			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	107			
1,2,3,4,7,8,9-HpCDF	1.0	1.00	100			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	0.97	97			
Total HpCDD						
OCDF	2.0	1.9	95			
OCDD	2.0	2.1	106			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range
Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365183

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/24/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☐	☒	☐	

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☐	☒	

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☐	☒	☐	
b.	Are the lab recovery limits specified on the report?		☐	☐	☒	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☐	☐	☒	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☐	☒	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	

	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question			Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.		☐	☒	☐	
	i.	Have the required matrix spikes been prepared and reported?	☐	☐	☒	
	ii.	If no, is there and explanation in the report as to why?	☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?	☐	☒	☐	Batch MS/MSDs were performed on a TOC sample from SDG 10364112.
	iv.	Are the lab limits specified on the report?	☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
	vi.	Are there compounds outside the lab limits?	☐	☒	☐	
		1. If yes, are the analytes above the lab limits?	☐	☐	☒	
		2. Below the lab limits?	☐	☐	☒	
		3. Is the source sample also flagged for compounds outside lab limits?	☐	☒	☐	The source sample is not included in this SDG.
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.		☐	☒	☐	RPDs discussed apply to MS/MSDs.
	i.	Is the RPD for the duplicate pair within the lab limits?	☒	☐	☐	
	ii.	If no, has the associated source sample been flagged?	☐	☒	☐	The source sample was not included in this SDG.
c.	What is the impact of failed QC on this project?		☒	☐	☐	No qualifiers were applied based on batch QC.

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) No blind field duplicates were collected with the TOC samples in this SDG.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

October 24, 2016

Nancy McDonald
Bay West Inc
5 Empire Drive
Saint Paul, MN 55103

RE: Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Dear Nancy McDonald:
Enclosed are the analytical results for sample(s) received by the laboratory on October 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Lori Castille
lori.castille@pacelabs.com
Project Manager

Enclosures

cc: Paul Raymaker, Bay West
Jeff Smith, Pace Analytical Services, Inc

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
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CERTIFICATIONS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
525 N 8th Street, Salina, KS 67401
Alaska Certification UST-107
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Virginia/VELAP Certification #: Pace
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792
Alaska Certification UST-107
Alaska Certification UST-107
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445
North Dakota Certification: # R-203
Wisconsin DNR Certification # : 998027470
WA Department of Ecology Lab ID# C1007
Nevada DNR #MN010842015-1
Oklahoma Department of Environmental Quality

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

SAMPLE SUMMARY

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10365183001	**BW16SR-006-0.27-0.52**	Solid	09/23/16 09:37	10/06/16 20:25
10365183002	**BW16SR-007-0.06-0.31**	Solid	09/23/16 10:14	10/06/16 20:25
10365183003	**BW16SR-008-0.23-0.48**	Solid	09/23/16 10:33	10/06/16 20:25
10365183004	**BW16SR-009-0.17-0.42**	Solid	09/23/16 10:38	10/06/16 20:25
10365183005	**BW16SR-010-0.0-0.24**	Solid	09/23/16 10:50	10/06/16 20:25
10365183006	**BW16SR-011-0.16-0.41**	Solid	09/23/16 11:02	10/06/16 20:25
10365183007	**BW16SR-012-0.0-0.25**	Solid	09/23/16 11:20	10/06/16 20:25
10365183008	**BW16SR-013-0.11-0.36**	Solid	09/23/16 11:33	10/06/16 20:25
10365183009	**BW16SR-002-0.0-0.15**	Solid	09/30/16 11:12	10/06/16 20:25
10365183010	**BW16SR-005-0.0-0.15**	Solid	09/30/16 11:20	10/06/16 20:25

Pace Analytical Services, LLC
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SAMPLE ANALYTE COUNT

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10365183001	**BW16SR-006-0.27-0.52**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183002	**BW16SR-007-0.06-0.31**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183003	**BW16SR-008-0.23-0.48**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183004	**BW16SR-009-0.17-0.42**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183005	**BW16SR-010-0.0-0.24**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183006	**BW16SR-011-0.16-0.41**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183007	**BW16SR-012-0.0-0.25**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183008	**BW16SR-013-0.11-0.36**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183009	**BW16SR-002-0.0-0.15**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V
10365183010	**BW16SR-005-0.0-0.15**	ASTM D2974	JDL	1	PASI-M
		EPA 9060A	KRV	5	PASI-V

Pace Analytical Services, LLC
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PROJECT NARRATIVE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Method: **EPA 9060A**
Description: Total Organic Carbon Quad
Client: Bay West, Inc.
Date: October 24, 2016

General Information:
10 samples were analyzed for EPA 9060A. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

Hold Time:
The samples were analyzed within the method required hold times with any exceptions noted below.

Method Blank:
All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

Laboratory Control Spike:
All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

Additional Comments:

This data package has been reviewed for quality and completeness and is approved for release.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-006-0.27-0.52 **Lab ID: 10365183001** Collected: 09/23/16 09:37 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**24.3**	%	0.10	0.10	1		10/10/16 14:16		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**7340**	mg/kg	4930	789	1		10/15/16 21:05	7440-44-0	
Total Organic Carbon	**7330**	mg/kg	1600	256	1		10/15/16 21:13	7440-44-0	
Total Organic Carbon	**8060**	mg/kg	1710	274	1		10/15/16 21:20	7440-44-0	
Total Organic Carbon	**5640**	mg/kg	1720	274	1		10/15/16 21:27	7440-44-0	
Mean Total Organic Carbon	**7090**	mg/kg	2490	398	1		10/15/16 21:27	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-007-0.06-0.31 **Lab ID: 10365183002** Collected: 09/23/16 10:14 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**37.0**	%	0.10	0.10	1		10/10/16 14:17		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**28200**	mg/kg	1430	229	1		10/15/16 21:35	7440-44-0	
Total Organic Carbon	**41300**	mg/kg	1800	289	1		10/15/16 21:42	7440-44-0	
Total Organic Carbon	**27400**	mg/kg	1720	275	1		10/15/16 21:49	7440-44-0	
Total Organic Carbon	**36500**	mg/kg	1560	250	1		10/15/16 21:56	7440-44-0	
Mean Total Organic Carbon	**33300**	mg/kg	1630	261	1		10/15/16 21:56	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-008-0.23-0.48 **Lab ID: 10365183003** Collected: 09/23/16 10:33 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**38.2**	%	0.10	0.10	1		10/10/16 14:17		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**48200**	mg/kg	1670	268	1		10/15/16 22:04	7440-44-0	
Total Organic Carbon	**52000**	mg/kg	2040	326	1		10/15/16 22:11	7440-44-0	
Total Organic Carbon	**46300**	mg/kg	1860	297	1		10/15/16 22:18	7440-44-0	
Total Organic Carbon	**46600**	mg/kg	1720	275	1		10/15/16 22:26	7440-44-0	
Mean Total Organic Carbon	**48300**	mg/kg	1820	291	1		10/15/16 22:26	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-009-0.17-0.42 **Lab ID: 10365183004** Collected: 09/23/16 10:38 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**26.9**	%	0.10	0.10	1		10/10/16 15:27		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**21200**	mg/kg	2440	390	1		10/15/16 22:33	7440-44-0	
Total Organic Carbon	**22300**	mg/kg	2290	367	1		10/15/16 22:40	7440-44-0	
Total Organic Carbon	**20600**	mg/kg	2500	400	1		10/15/16 22:48	7440-44-0	
Total Organic Carbon	**16600**	mg/kg	2490	398	1		10/15/16 22:55	7440-44-0	
Mean Total Organic Carbon	**20200**	mg/kg	2430	389	1		10/15/16 22:55	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-010-0.0-0.24 **Lab ID: 10365183005** Collected: 09/23/16 10:50 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**47.8**	%	0.10	0.10	1		10/10/16 15:28		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**55600**	mg/kg	2880	460	1		10/15/16 23:02	7440-44-0	
Total Organic Carbon	**60900**	mg/kg	2610	417	1		10/15/16 23:10	7440-44-0	
Total Organic Carbon	**36000**	mg/kg	2690	430	1		10/15/16 23:17	7440-44-0	
Total Organic Carbon	**54200**	mg/kg	2830	453	1		10/15/16 23:24	7440-44-0	
Mean Total Organic Carbon	**51700**	mg/kg	2750	440	1		10/15/16 23:24	7440-44-0	

Pace Analytical Services, LLC
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Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-011-0.16-0.41 **Lab ID: 10365183006** Collected: 09/23/16 11:02 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**38.1**	%	0.10	0.10	1		10/10/16 15:28		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**29700**	mg/kg	2320	371	1		10/15/16 23:31	7440-44-0	
Total Organic Carbon	**35700**	mg/kg	2480	396	1		10/15/16 23:39	7440-44-0	
Total Organic Carbon	**27700**	mg/kg	2510	402	1		10/15/16 23:46	7440-44-0	
Total Organic Carbon	**30600**	mg/kg	2780	444	1		10/15/16 23:53	7440-44-0	
Mean Total Organic Carbon	**30900**	mg/kg	2520	403	1		10/15/16 23:53	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-012-0.0-0.25 **Lab ID: 10365183007** Collected: 09/23/16 11:20 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**57.1**	%	0.10	0.10	1		10/10/16 15:28		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**60300**	mg/kg	1970	315	1		10/18/16 16:40	7440-44-0	
Total Organic Carbon	**60000**	mg/kg	3080	492	1		10/18/16 16:49	7440-44-0	
Total Organic Carbon	**61600**	mg/kg	3340	535	1		10/18/16 16:56	7440-44-0	
Total Organic Carbon	**58700**	mg/kg	3230	516	1		10/18/16 17:06	7440-44-0	
Mean Total Organic Carbon	**60100**	mg/kg	2900	464	1		10/18/16 17:06	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-013-0.11-0.36 **Lab ID: 10365183008** Collected: 09/23/16 11:33 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**33.0**	%	0.10	0.10	1		10/10/16 15:28		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**18500**	mg/kg	3330	533	1		10/18/16 17:28	7440-44-0	
Total Organic Carbon	**20500**	mg/kg	2430	388	1		10/18/16 17:35	7440-44-0	
Total Organic Carbon	**18100**	mg/kg	2350	377	1		10/18/16 17:43	7440-44-0	
Total Organic Carbon	**15600**	mg/kg	2340	375	1		10/18/16 17:50	7440-44-0	
Mean Total Organic Carbon	**18200**	mg/kg	2610	418	1		10/18/16 17:50	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-002-0.0-0.15 **Lab ID: 10365183009** Collected: 09/30/16 11:12 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**54.3**	%	0.10	0.10	1		10/10/16 15:29		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**40600**	mg/kg	2600	417	1		10/18/16 17:57	7440-44-0	
Total Organic Carbon	**54900**	mg/kg	2540	406	1		10/18/16 18:05	7440-44-0	
Total Organic Carbon	**42400**	mg/kg	2670	427	1		10/18/16 18:12	7440-44-0	
Total Organic Carbon	**50400**	mg/kg	2610	417	1		10/18/16 18:19	7440-44-0	
Mean Total Organic Carbon	**47100**	mg/kg	2600	417	1		10/18/16 18:19	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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Minneapolis, MN 55414
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Sample: BW16SR-005-0.0-0.15 **Lab ID: 10365183010** Collected: 09/30/16 11:20 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**52.9**	%	0.10	0.10	1		10/17/16 15:39		
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**34500**	mg/kg	2960	473	1		10/18/16 18:27	7440-44-0	
Total Organic Carbon	**38300**	mg/kg	2970	475	1		10/18/16 18:35	7440-44-0	
Total Organic Carbon	**30300**	mg/kg	2830	453	1		10/18/16 18:42	7440-44-0	
Total Organic Carbon	**32800**	mg/kg	2940	471	1		10/18/16 18:49	7440-44-0	
Mean Total Organic Carbon	**33900**	mg/kg	2920	468	1		10/18/16 18:49	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

QC Batch: 440203 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365183001, 10365183002, 10365183003

SAMPLE DUPLICATE: 2393916

Parameter	Units	10365183003 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	38.2	37.7	1	30	

SAMPLE DUPLICATE: 2394002

Parameter	Units	10364900001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	17.0	15.9	7	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

QC Batch: 440231 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365183004, 10365183005, 10365183006, 10365183007, 10365183008, 10365183009

SAMPLE DUPLICATE: 2394092

Parameter	Units	10365310003 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	3.9	4.1	4	30	

SAMPLE DUPLICATE: 2394137

Parameter	Units	10365183004 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	26.9	29.4	9	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

QC Batch: 441541
QC Batch Method: ASTM D2974
Analysis Method: ASTM D2974
Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365183010

SAMPLE DUPLICATE: 2403248

Parameter	Units	10365048013 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	26.6	25.8	3	30	

SAMPLE DUPLICATE: 2403249

Parameter	Units	10365188006 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	37.1	35.8	4	30	

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REPORT OF LABORATORY ANALYSIS

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(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

QC Batch: 97333 Analysis Method: EPA 9060A
QC Batch Method: EPA 9060A Analysis Description: 9060 TOC Average
Associated Lab Samples: 10365183001, 10365183002, 10365183003, 10365183004, 10365183005, 10365183006

METHOD BLANK: 384973 Matrix: Solid
Associated Lab Samples: 10365183001, 10365183002, 10365183003, 10365183004, 10365183005, 10365183006

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mean Total Organic Carbon	mg/kg	ND	302	48.3	10/14/16 13:37	

LABORATORY CONTROL SAMPLE: 384974

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mean Total Organic Carbon	mg/kg	5820	4230	73	49-151	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 384975 384976

Parameter	Units	40138572045 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mean Total Organic Carbon	mg/kg	19200	16900	16900	36100	37600	100	109	70-130	4	25	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

QC Batch: 97524 Analysis Method: EPA 9060A
QC Batch Method: EPA 9060A Analysis Description: 9060 TOC Average
Associated Lab Samples: 10365183007, 10365183008, 10365183009, 10365183010

METHOD BLANK: 385805 Matrix: Solid
Associated Lab Samples: 10365183007, 10365183008, 10365183009, 10365183010

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mean Total Organic Carbon	mg/kg	ND	300	48.0	10/18/16 07:29	

LABORATORY CONTROL SAMPLE: 385806

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mean Total Organic Carbon	mg/kg	5820	4730	81	49-151	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 385809 385810

Parameter	Units	10364112010 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mean Total Organic Carbon	mg/kg	5100	10100	10100	15000	15100	98	99	70-130	1	25	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALIFIERS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-M	Pace Analytical Services - Minneapolis
PASI-V	Pace Analytical Services - Virginia

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365183

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10365183001	**BW16SR-006-0.27-0.52**	ASTM D2974	440203		
10365183002	**BW16SR-007-0.06-0.31**	ASTM D2974	440203		
10365183003	**BW16SR-008-0.23-0.48**	ASTM D2974	440203		
10365183004	**BW16SR-009-0.17-0.42**	ASTM D2974	440231		
10365183005	**BW16SR-010-0.0-0.24**	ASTM D2974	440231		
10365183006	**BW16SR-011-0.16-0.41**	ASTM D2974	440231		
10365183007	**BW16SR-012-0.0-0.25**	ASTM D2974	440231		
10365183008	**BW16SR-013-0.11-0.36**	ASTM D2974	440231		
10365183009	**BW16SR-002-0.0-0.15**	ASTM D2974	440231		
10365183010	**BW16SR-005-0.0-0.15**	ASTM D2974	441541		
10365183001	**BW16SR-006-0.27-0.52**	EPA 9060A	97333		
10365183001	**BW16SR-006-0.27-0.52**	EPA 9060A	97395		
10365183002	**BW16SR-007-0.06-0.31**	EPA 9060A	97333		
10365183002	**BW16SR-007-0.06-0.31**	EPA 9060A	97395		
10365183003	**BW16SR-008-0.23-0.48**	EPA 9060A	97333		
10365183003	**BW16SR-008-0.23-0.48**	EPA 9060A	97395		
10365183004	**BW16SR-009-0.17-0.42**	EPA 9060A	97333		
10365183004	**BW16SR-009-0.17-0.42**	EPA 9060A	97395		
10365183005	**BW16SR-010-0.0-0.24**	EPA 9060A	97333		
10365183005	**BW16SR-010-0.0-0.24**	EPA 9060A	97395		
10365183006	**BW16SR-011-0.16-0.41**	EPA 9060A	97333		
10365183006	**BW16SR-011-0.16-0.41**	EPA 9060A	97395		
10365183007	**BW16SR-012-0.0-0.25**	EPA 9060A	97524		
10365183007	**BW16SR-012-0.0-0.25**	EPA 9060A	97558		
10365183008	**BW16SR-013-0.11-0.36**	EPA 9060A	97524		
10365183008	**BW16SR-013-0.11-0.36**	EPA 9060A	97558		
10365183009	**BW16SR-002-0.0-0.15**	EPA 9060A	97524		
10365183009	**BW16SR-002-0.0-0.15**	EPA 9060A	97558		
10365183010	**BW16SR-005-0.0-0.15**	EPA 9060A	97524		
10365183010	**BW16SR-005-0.0-0.15**	EPA 9060A	97558		

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

105365183

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-2
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: TOC (SW-846 9080A Quad Bum)	Grain Size (ASTM D422 w/ hydrometer)	Comments
Ex.	*BW15MLW-005*	*BW14MLW-005-0-0.15*	SO	G	*3/12/15*	*1204*												
1	BW16SR-006	BW16SR-006-0.27-0.52	SO	G	9/23/16	0937	2	2								1	1	w1
2	BW16SR-007	BW16SR-007-0.06-0.31	SO	G	9/23/16	1014	2	2								1	1	w2
3	BW16SR-008	BW16SR-008-0.23-0.48	SO	G	9/23/16	1033	2	2								1	1	w3
4	BW16SR-009	BW16SR-009-0.17-0.42	SO	G	9/23/16	1038	2	2								1	1	w4
5	BW16SR-010	BW16SR-010-0.0-0.24	SO	G	9/23/16	1050	2	2								1	1	w5
6	BW16SR-011	BW16SR-011-0.16-0.41	SO	G	9/23/16	1102	2	2								1	1	w6
7	BW16SR-012	BW16SR-012-0.0-0.25	SO	G	9/23/16	1120	2	2								1	1	w7
8	BW16SR-013	BW16SR-013-0.11-0.36	SO	G	9/23/16	1133	2	2								1	1	w8
9	BW16SR-002	BW16SR-002-0.0-0.15	SO	G	9/30/16	1112	2	2								1	1	004
10																		010
11																		
12																		

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME
		10/6/16	1444	Kristena Polson	10/6/16	1445
Reference Subcontractor Goods and/or Services Purchase Order Form signed by Bay West on 9/19/16	Kristena Polson	10/6/16	1715		10/6/16	1715
		10/6/16	2025		10-6-16	20:25

SAMPLE CONDITIONS			
Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
0.2 / 4.9	Y	N	Y
0.6			

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: CJM DATE Signed (MM/DD/YY): 10/6/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: **WO# : 10365183**

10365183

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ SpeeDee ☐ Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☐ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.0, 0.6, 4.7 **Cooler Temp Corrected (°C):** 0.2, 0.8, 4.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☒ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** CLW 10.6.16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☑ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	☒ Yes	☒ No	☐ N/A	12. Rec'd extra sample not on C.O.C
-Includes Date/Time/ID/Analysis Matrix: SL CLW 10.6.16				BW16SR-005-0.0-0.15' (9.30.16 11:20)
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	Sample #
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☒ N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: **Date:** 10/7/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Intra-Regional Chain of Custody

SCANNED

Pace Analytical®
www.pacelabs.com

Workorder: 10365183 **Workorder Name:** J160139 SLR Sediment AOCs **Owner Received Date:** 10/6/2016 **Due Date:** 10/20/2016

Received at:	Send To Lab:
Pace Analytical Minnesota 1700 Elm Street Suite 200 Minneapolis, MN 55414 Phone (612)607-1700	Pace Analytical Billings MT 150 N Ninth Street Billings, MT 59101 Phone (406)254-7226

Report To:
Oyeyemi Odujole

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: Other	Requested Analysis: ASTM D422	LAB USE ONLY
1	BW16SR-006-0.27-0.52	PS	9/23/2016 09:37	10365183001	Solid	1	X	
2	BW16SR-007-0.06-0.31	PS	9/23/2016 10:14	10365183002	Solid	1	X	
3	BW16SR-008-0.23-0.48	PS	9/23/2016 10:33	10365183003	Solid	1	X	
4	BW16SR-009-0.17-0.42	PS	9/23/2016 10:38	10365183004	Solid	1	X	
5	BW16SR-010-0.0-0.24	PS	9/23/2016 10:50	10365183005	Solid	1	X	
6	BW16SR-011-0.16-0.41	PS	9/23/2016 11:02	10365183006	Solid	1	X	
7	BW16SR-012-0.0-0.25	PS	9/23/2016 11:20	10365183007	Solid	1	X	
8	BW16SR-013-0.11-0.36	PS	9/23/2016 11:33	10365183008	Solid	1	X	
9	BW16SR-002-0.0-0.15	PS	9/30/2016 11:12	10365183009	Solid	1	X	
10	BW16SR-005-0.0-0.15	PS	9/30/2016 11:20	10365183010	Solid	1	X	

Transfers	Released By	Date/Time	Received By	Date/Time
1	Pace	10/7/16 1513		
2	Fed Ex		M Walters - Pace	10/8/16 0950
3				
4				

Comments: **Admin work**

Cooler Temperature on Receipt 2.1 °C | **Custody Seal** (Y) or N | **Received on Ice** (Y) or N | **Samples Intact** (Y) or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.*
This chain of custody is considered complete as is since this information is available in the owner laboratory.

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 04Aug2016 Page 1 of 1
	Document No.: F-MT-C-184-Rev.10	Issuing Authority: Pace Montana Quality Office

Sample Condition Upon Receipt

Client Name: Pace MN

Project #: 10365183

Courier: ☑ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other:

Tracking Number: 6751 5826 4964, 4995 (2)

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No Seals Intact? ☑ Yes ☐ No Optional: Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other: Temp Blank? ☑ Yes ☐ No

Thermometer Used: ☒ 160285052 ☐ 140279186 Type of Ice: ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun ☐ NA

Cooler Temp Read: 2.4, 2.2 Date and Initials of Person Examining Contents: MW 10/18/16

Cooler Temp Corrected: 2.1, 1.7 Biological Tissue Frozen? ☐ Yes ☑ No

Temp should be above freezing to 6°C

				Comments:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☐ Yes	☒ No	☐ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☐ Yes	☒ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container.
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: Soil	☒ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☑ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☑ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water)	☐ Yes	☒ No		Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased): NA				

CLIENT NOTIFICATION/RESOLUTION Field Data Required? ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: Lori Peterson Date: 10/14/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

Chain of Custody

WO#: 1276613

PM: CLJ **Due Date: 10/20/16**

CLIENT: PACE MPLS

Analytical® www.pacelabs.com

Workorder: 10365183 **Workorder Name: J160139 SLR Sediment AOCs** **Owner Received Date: 10/6/2016** **Results Requested By: 10/20/2016**

Report To	Subcontract To
Oyeyemi Odujole Pace Analytical Minnesota 1700 Elm Street Suite 200 Minneapolis, MN 55414 Phone (612)607-1700	Pace Analytical Virginia MN 315 Chestnut Street Virginia, MN 55792 Phone (218)742-1042

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: Unpreserved	Requested Analysis: TOC	LAB USE ONLY
1	BW16SR-006-0.27-0.52	PS	9/23/2016 09:37	10365183001	Solid	1	X	
2	BW16SR-007-0.06-0.31	PS	9/23/2016 10:14	10365183002	Solid	1	X	
3	BW16SR-008-0.23-0.48	PS	9/23/2016 10:33	10365183003	Solid	1	X	
4	BW16SR-009-0.17-0.42	PS	9/23/2016 10:38	10365183004	Solid	1	X	
5	BW16SR-010-0.0-0.24	PS	9/23/2016 10:50	10365183005	Solid	1	X	
6	BW16SR-011-0.16-0.41	PS	9/23/2016 11:02	10365183006	Solid	1	X	
7	BW16SR-012-0.0-0.25	PS	9/23/2016 11:20	10365183007	Solid	1	X	
8	BW16SR-013-0.11-0.36	PS	9/23/2016 11:33	10365183008	Solid	1	X	
9	BW16SR-002-0.0-0.15	PS	9/30/2016 11:12	10365183009	Solid	1	X	
10	BW16SR-005-0.0-0.15	PS	9/30/2016 11:20	10365183010	Solid	1	X	

Transfers	Released By	Date/Time	Received By	Date/Time
1	Pace	10/7/16 1518		10/7/16 1435
2		10/7/16 2330		10-8-16 11:00
3				

Comments: **Admin work**

Cooler Temperature on Receipt 3.4 °C | **Custody Seal** Y or N | **Received on Ice** (Y) or N | **Samples Intact** (Y) or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.*

This chain of custody is considered complete as is since this information is available in the owner laboratory.

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Pace MIL

Project #:

WO# : 1276613

1276613

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No Seals Intact? ☑ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☑ Other: Haz Pac

Temp Blank? ☑ Yes ☐ No

Thermometer Used: ☑ 140792808 Type of Ice: ☑ Wet ☐ Blue ☐ None ☑ Samples on ice, cooling process has begun

Cooler Temp Read °C: 9.1 Cooler Temp Corrected °C: 3.4

Temp should be above freezing to 6°C Correction Factor: +0.3

Biological Tissue Frozen? ☐ Yes ☐ No ☑ NA

Date and Initials of Person Examining Contents: JPC 10/7/16

Comments: LM 10-8-16

Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☐ Yes	☑ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	☑ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	☐ No	☑ N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐ Yes	☐ No	☑ N/A	13.
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

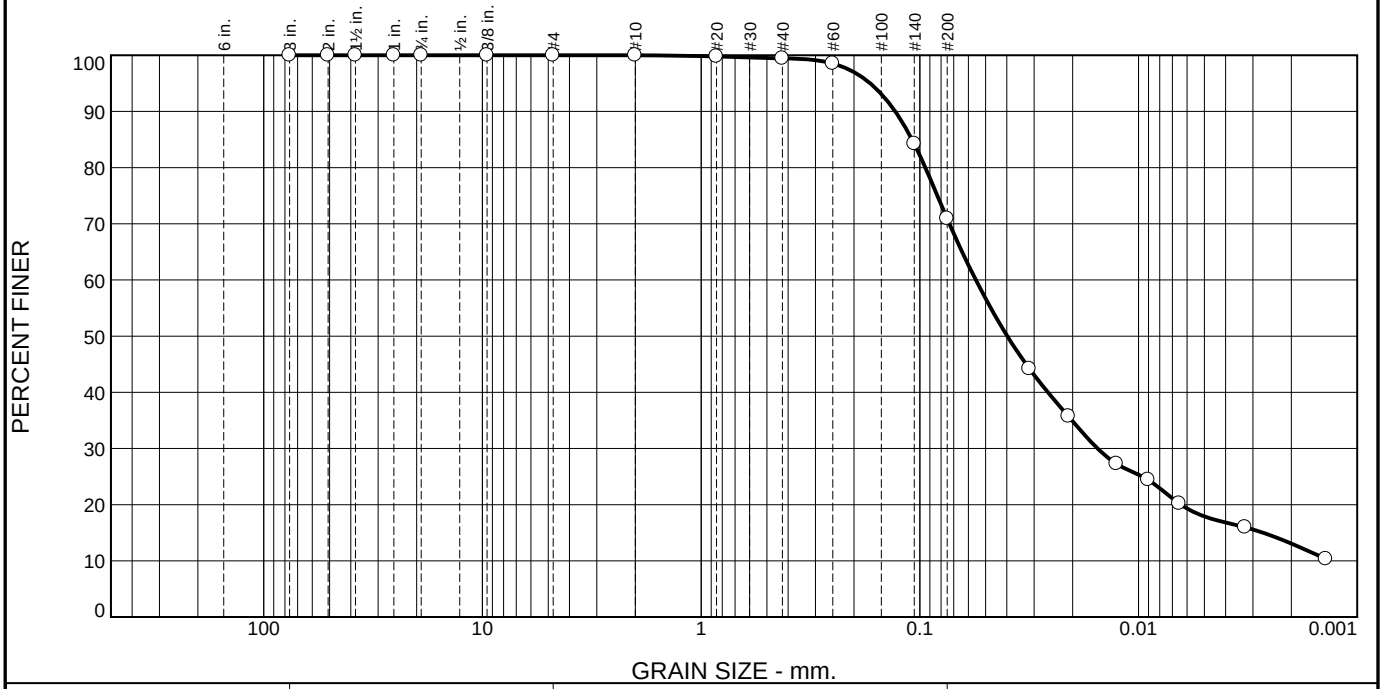
FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: Carrie Jern Date: 10/10/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	28	53	18

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	100		
#40	99		
#60	99		
#140	84		
#200	71		
0.0315 mm.	44		
0.0209 mm.	36		
0.0126 mm.	27		
0.0090 mm.	24		
0.0065 mm.	20		
0.0033 mm.	16		
0.0014 mm.	10		

* (no specification provided)

Material Description

silt with sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.1295 D_{85}= 0.1085 D_{60}= 0.0555
D_{50}= 0.0398 D_{30}= 0.0154 D_{15}= 0.0027
D_{10}= C_u= C_c=

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-006-0.27-0.52
Sample Number: 10365183-1

Date Sampled: 9/23/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-006-0.27-0.52
Sample Number: 10365183-1
Material Description: silt with sand
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
841.94	562.21	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
70.92	0.00	#20	0.15	0.00	100
		#40	0.24	0.00	99
		#60	0.66	0.00	99
		#140	10.14	0.00	84
		#200	9.43	0.00	71

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =70.92
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	39.0	31.3	0.0142	39.0	9.9	0.0315	44.2
5.00	17.0	33.0	25.3	0.0142	33.0	10.9	0.0209	35.7
15.00	17.0	27.0	19.3	0.0142	27.0	11.9	0.0126	27.3
30.00	17.0	25.0	17.3	0.0142	25.0	12.2	0.0090	24.4
60.00	17.0	22.0	14.3	0.0142	22.0	12.7	0.0065	20.2
250.00	17.0	19.0	11.3	0.0142	19.0	13.2	0.0033	16.0
1440.00	17.0	15.0	7.3	0.0142	15.0	13.8	0.0014	10.3

Pace Analytical Services, Inc.

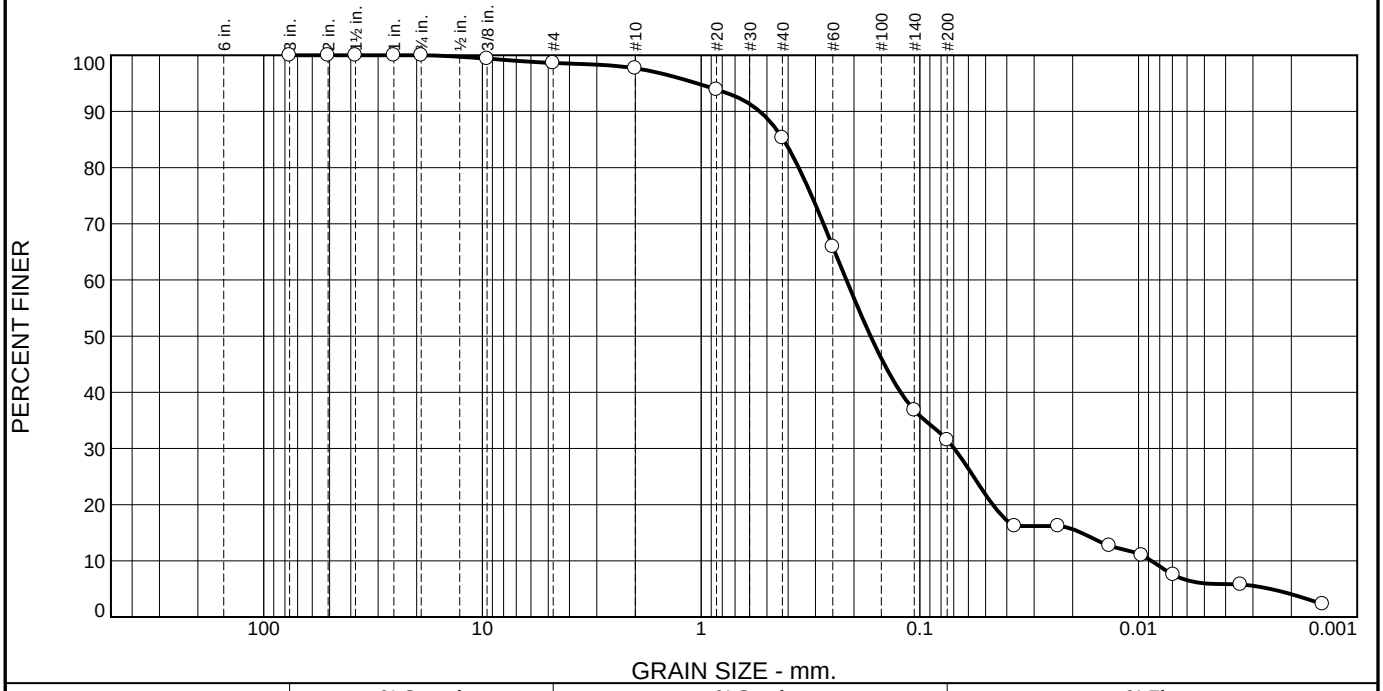
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	28	29	53	18	71

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
		0.0027	0.0064	0.0154	0.0260	0.0398	0.0555	0.0943	0.1085	0.1295	0.1685

Fineness Modulus
0.08

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	1	1	13	53	26	6

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	99		
#4	99		
#10	98		
#20	94		
#40	85		
#60	66		
#140	37		
#200	32		
0.0368 mm.	16		
0.0233 mm.	16		
0.0136 mm.	13		
0.0097 mm.	11		
0.0069 mm.	7.5		
0.0034 mm.	5.8		
0.0014 mm.	2.3		

* (no specification provided)

Material Description

silty sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= SM **AASHTO (M 145)=** A-2-4(0)

Coefficients

D_{90}= 0.5392 D_{85}= 0.4207 D_{60}= 0.2167
D_{50}= 0.1679 D_{30}= 0.0696 D_{15}= 0.0183
D_{10}= 0.0087 C_u= 24.83 C_c= 2.56

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-007-0.06-0.31 **Date Sampled:** 9/23/16
Sample Number: 10365183-2

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-007-0.06-0.31
Sample Number: 10365183-2
Material Description: silty sand
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: SM **AASHTO Classification:** A-2-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
773.17	583.52	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	1.15	0.00	99
		#4	1.45	0.00	99
		#10	1.81	0.00	98
56.28	0.00	#20	2.18	0.00	94
		#40	4.97	0.00	85
		#60	11.15	0.00	66
		#140	16.77	0.00	37
		#200	3.05	0.00	32

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 98
Weight of hydrometer sample =56.28
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	17.0	9.3	0.0142	17.0	13.5	0.0368	16.2
5.00	17.0	17.0	9.3	0.0142	17.0	13.5	0.0233	16.2
15.00	17.0	15.0	7.3	0.0142	15.0	13.8	0.0136	12.7
30.00	17.0	14.0	6.3	0.0142	14.0	14.0	0.0097	11.0
60.00	17.0	12.0	4.3	0.0142	12.0	14.3	0.0069	7.5
250.00	17.0	11.0	3.3	0.0142	11.0	14.5	0.0034	5.8
1440.00	17.0	9.0	1.3	0.0142	9.0	14.8	0.0014	2.3

Pace Analytical Services, Inc.

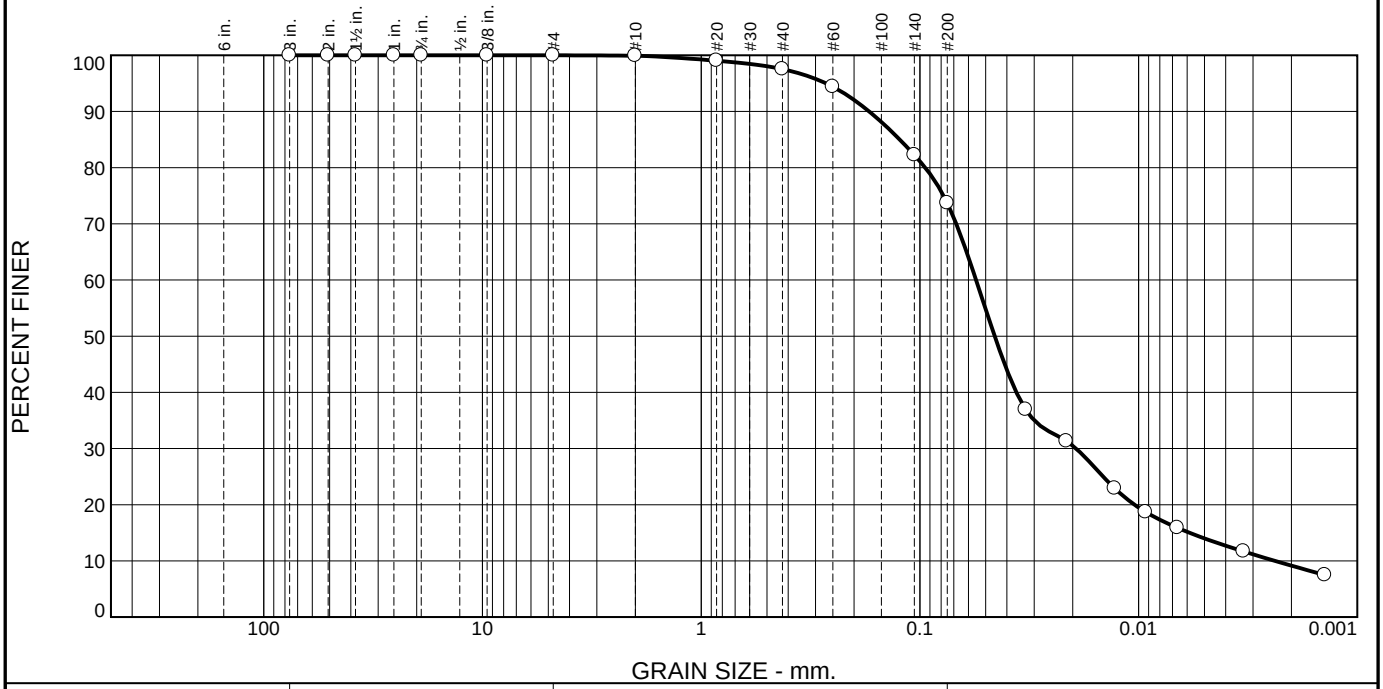
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	1	1	1	13	53	67	26	6	32

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0025	0.0087	0.0183	0.0464	0.0696	0.1224	0.1679	0.2167	0.3578	0.4207	0.5392	1.0448

Fineness Modulus	C_u	C_c
0.98	24.83	2.56

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	2	24	60	14

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	98		
#60	94		
#140	82		
#200	74		
0.0328 mm.	37		
0.0214 mm.	31		
0.0129 mm.	23		
0.0093 mm.	19		
0.0066 mm.	16		
0.0033 mm.	12		
0.0014 mm.	7.5		

* (no specification provided)

Material Description

silt with sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=** NP

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.1706 D_{85}= 0.1237 D_{60}= 0.0553
D_{50}= 0.0455 D_{30}= 0.0193 D_{15}= 0.0058
D_{10}= 0.0024 C_u= 23.27 C_c= 2.84

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-008-0.23-0.48 **Date Sampled:** 9/23/16
Sample Number: 10365183-3

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/21/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-008-0.23-0.48
Sample Number: 10365183-3
Material Description: silt with sand
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV **PI:** NP
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
933.64	566.93	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.01	0.00	100
		#10	0.25	0.00	100
71.23	0.00	#20	0.64	0.00	99
		#40	1.07	0.00	98
		#60	2.24	0.00	94
		#140	8.66	0.00	82
		#200	6.07	0.00	74

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =71.23
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	34.0	26.3	0.0142	34.0	10.7	0.0328	36.9
5.00	17.0	30.0	22.3	0.0142	30.0	11.4	0.0214	31.3
15.00	17.0	24.0	16.3	0.0142	24.0	12.4	0.0129	22.9
30.00	17.0	21.0	13.3	0.0142	21.0	12.9	0.0093	18.7
60.00	17.0	19.0	11.3	0.0142	19.0	13.2	0.0066	15.9
250.00	17.0	16.0	8.3	0.0142	16.0	13.7	0.0033	11.7
1440.00	17.0	13.0	5.3	0.0142	13.0	14.2	0.0014	7.5

Pace Analytical Services, Inc.

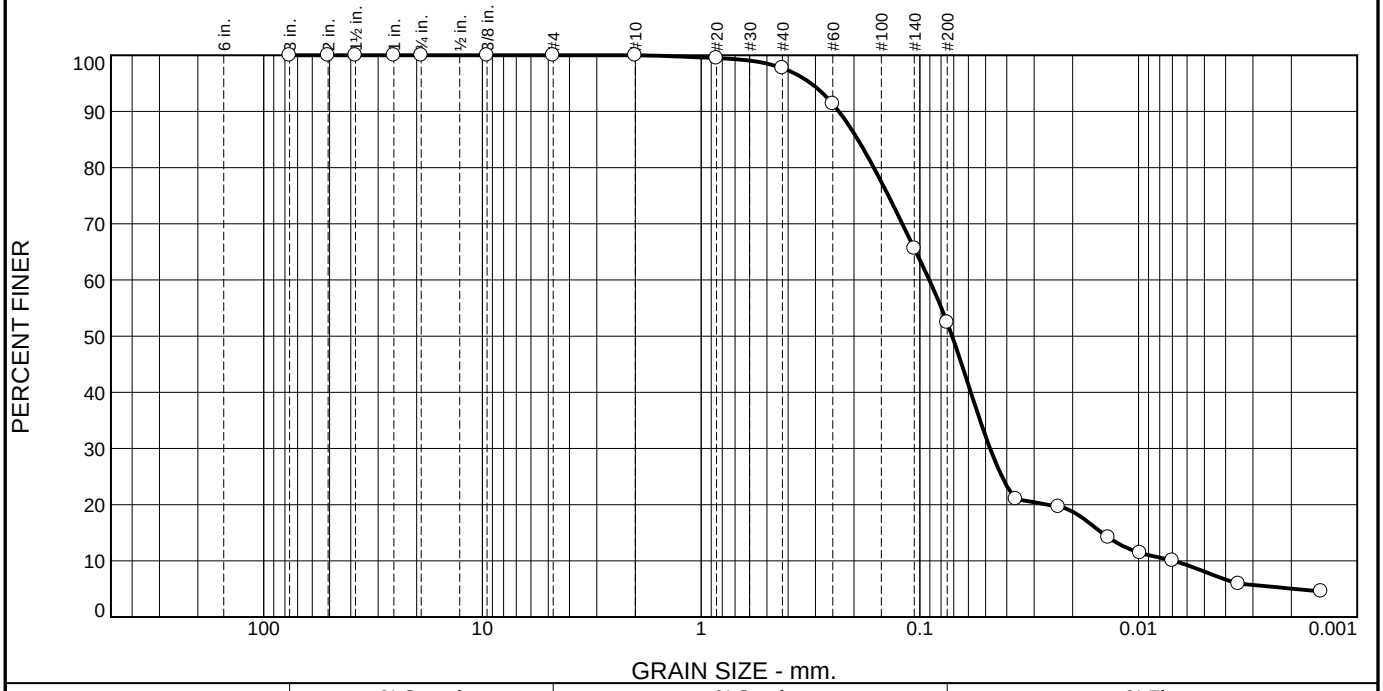
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	2	24	26	60	14	74

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0024	0.0058	0.0104	0.0193	0.0362	0.0455	0.0553	0.0945	0.1237	0.1706	0.2688

Fineness Modulus	C_u	C_c
0.18	23.27	2.84

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	2	46	44	8

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	98		
#60	91		
#140	66		
#200	52		
0.0365 mm.	21		
0.0232 mm.	20		
0.0137 mm.	14		
0.0098 mm.	11		
0.0070 mm.	10		
0.0035 mm.	5.9		
0.0015 mm.	4.6		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.2344 D_{85}= 0.1921 D_{60}= 0.0905
D_{50}= 0.0712 D_{30}= 0.0477 D_{15}= 0.0147
D_{10}= 0.0069 C_u= 13.07 C_c= 3.64

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-009-0.17-0.42
Sample Number: 10365183-4

Date Sampled: 9/23/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-009-0.17-0.42
Sample Number: 10365183-4
Material Description: sandy silt
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1016.05	601.39	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
74.53	0.00	#20	0.38	0.00	99
		#40	1.33	0.00	98
		#60	4.74	0.00	91
		#140	19.18	0.00	66
		#200	9.82	0.00	52

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =74.53
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.56
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	23.0	15.3	0.0146	23.0	12.5	0.0365	21.0
5.00	17.0	22.0	14.3	0.0146	22.0	12.7	0.0232	19.6
15.00	17.0	18.0	10.3	0.0146	18.0	13.3	0.0137	14.2
30.00	17.0	16.0	8.3	0.0146	16.0	13.7	0.0098	11.4
60.00	17.0	15.0	7.3	0.0146	15.0	13.8	0.0070	10.1
250.00	17.0	12.0	4.3	0.0146	12.0	14.3	0.0035	5.9
1440.00	17.0	11.0	3.3	0.0146	11.0	14.5	0.0015	4.6

Pace Analytical Services, Inc.

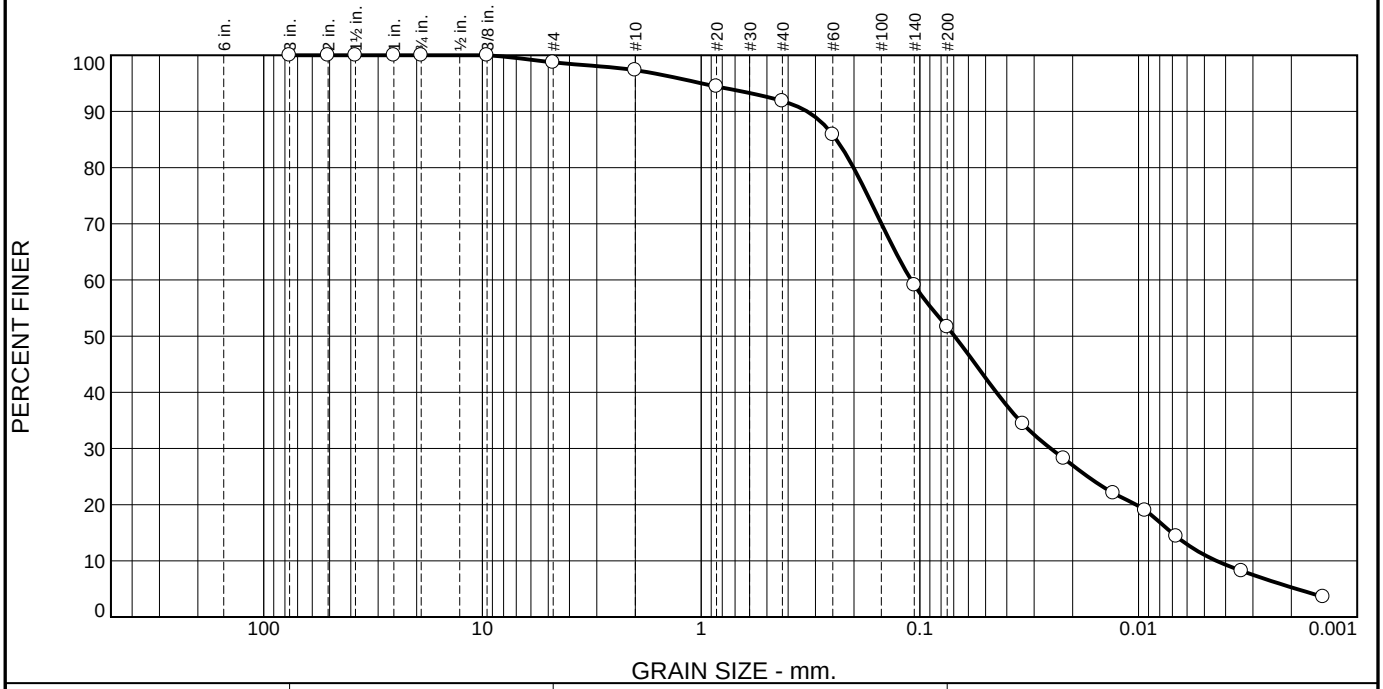
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	2	46	48	44	8	52

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0019	0.0069	0.0147	0.0261	0.0477	0.0584	0.0712	0.0905	0.1624	0.1921	0.2344	0.3144

Fineness Modulus	C_u	C_c
0.29	13.07	3.64

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	1	2	5	40	41	11

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	99		
#10	97		
#20	94		
#40	92		
#60	86		
#140	59		
#200	52		
0.0338 mm.	34		
0.0220 mm.	28		
0.0130 mm.	22		
0.0093 mm.	19		
0.0067 mm.	14		
0.0034 mm.	8.2		
0.0014 mm.	3.6		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.3275 D_{85}= 0.2408 D_{60}= 0.1096
D_{50}= 0.0694 D_{30}= 0.0252 D_{15}= 0.0070
D_{10}= 0.0044 C_u= 24.92 C_c= 1.32

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-010-0.0-0.24
Sample Number: 10365183-5

Date Sampled: 9/23/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-010-0.0-0.24
Sample Number: 10365183-5
Material Description: sandy silt
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
739.69	613.43	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	1.60	0.00	99
		#10	1.76	0.00	97
63.21	0.00	#20	1.88	0.00	94
		#40	1.69	0.00	92
		#60	3.89	0.00	86
		#140	17.37	0.00	59
		#200	4.84	0.00	52

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 97
Weight of hydrometer sample =63.21
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	30.0	22.3	0.0142	30.0	11.4	0.0338	34.4
5.00	17.0	26.0	18.3	0.0142	26.0	12.0	0.0220	28.2
15.00	17.0	22.0	14.3	0.0142	22.0	12.7	0.0130	22.1
30.00	17.0	20.0	12.3	0.0142	20.0	13.0	0.0093	19.0
60.00	17.0	17.0	9.3	0.0142	17.0	13.5	0.0067	14.4
250.00	17.0	13.0	5.3	0.0142	13.0	14.2	0.0034	8.2
1440.00	17.0	10.0	2.3	0.0142	10.0	14.7	0.0014	3.6

Pace Analytical Services, Inc.

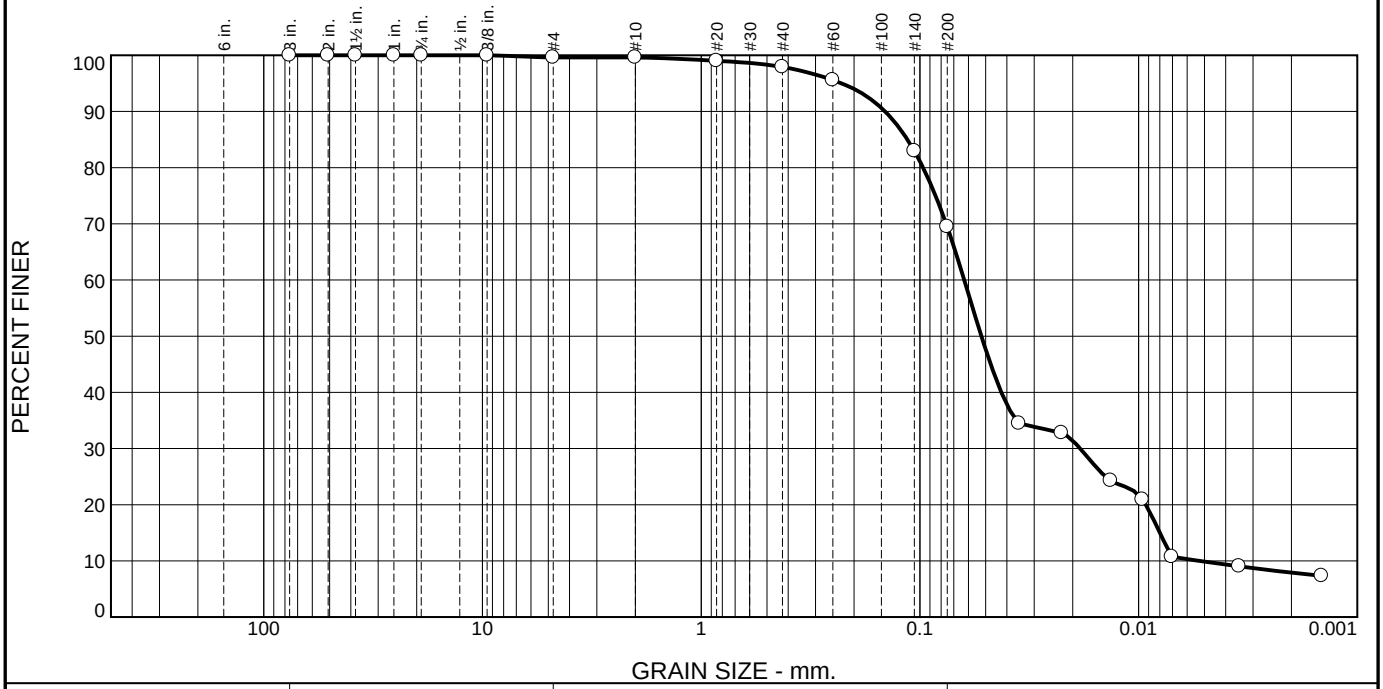
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	1	1	2	5	40	47	41	11	52

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0019	0.0044	0.0070	0.0103	0.0252	0.0447	0.0694	0.1096	0.2012	0.2408	0.3275	0.9943

Fineness Modulus	C_u	C_c
0.56	24.92	1.32

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	2	29	59	10

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	98		
#60	96		
#140	83		
#200	69		
0.0353 mm.	34		
0.0225 mm.	33		
0.0134 mm.	24		
0.0096 mm.	21		
0.0070 mm.	11		
0.0035 mm.	9.0		
0.0015 mm.	7.3		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.1442 D_{85}= 0.1141 D_{60}= 0.0627
D_{50}= 0.0523 D_{30}= 0.0185 D_{15}= 0.0080
D_{10}= 0.0053 C_u= 11.93 C_c= 1.04

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-011-0.16-0.41 **Date Sampled:** 9/23/16
Sample Number: 10365183-6

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-011-0.16-0.41
Sample Number: 10365183-6
Material Description: sandy silt
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
752.89	606.87	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.57	0.00	100
60.03	0.00	#10	0.00	0.00	100
		#20	0.36	0.00	99
		#40	0.66	0.00	98
		#60	1.41	0.00	96
		#140	7.61	0.00	83
		#200	8.12	0.00	69

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =60.03
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.56
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	28.0	20.3	0.0146	28.0	11.7	0.0353	34.5
5.00	17.0	27.0	19.3	0.0146	27.0	11.9	0.0225	32.8
15.00	17.0	22.0	14.3	0.0146	22.0	12.7	0.0134	24.3
30.00	17.0	20.0	12.3	0.0146	20.0	13.0	0.0096	20.9
60.00	17.0	14.0	6.3	0.0146	14.0	14.0	0.0070	10.7
250.00	17.0	13.0	5.3	0.0146	13.0	14.2	0.0035	9.0
1440.00	17.0	12.0	4.3	0.0146	12.0	14.3	0.0015	7.3

Pace Analytical Services, Inc.

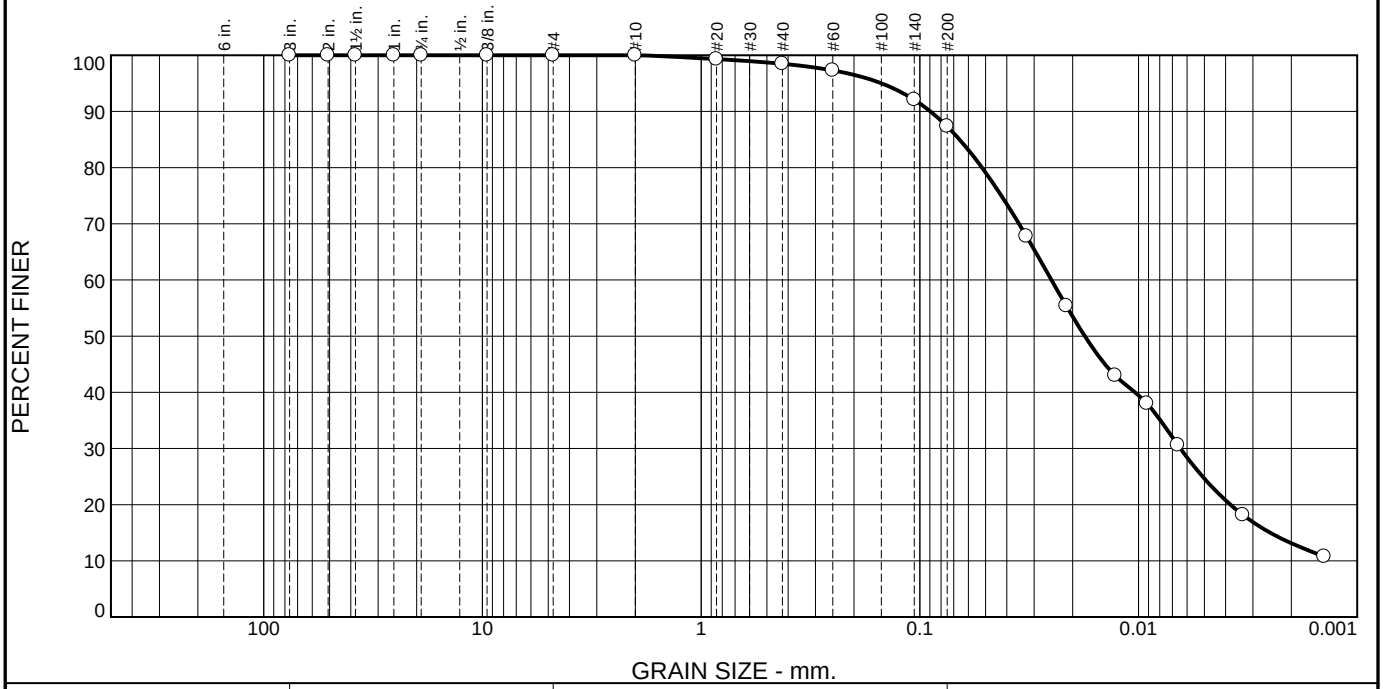
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	2	29	31	59	10	69

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0053	0.0080	0.0093	0.0185	0.0424	0.0523	0.0627	0.0967	0.1141	0.1442	0.2281

Fineness Modulus	C_u	C_c
0.16	11.93	1.04

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	2	11	62	25

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	98		
#60	97		
#140	92		
#200	87		
0.0326 mm.	68		
0.0214 mm.	55		
0.0128 mm.	43		
0.0092 mm.	38		
0.0066 mm.	31		
0.0033 mm.	18		
0.0014 mm.	11		

* (no specification provided)

Material Description

silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0894 D_{85}= 0.0658 D_{60}= 0.0250
D_{50}= 0.0176 D_{30}= 0.0064 D_{15}= 0.0025
D_{10}= C_u= C_c=

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-012-0.0-0.25
Sample Number: 10365183-7

Date Sampled: 9/23/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-012-0.0-0.25
Sample Number: 10365183-7
Material Description: silt
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
635.80	592.43	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
40.32	0.00	#20	0.28	0.00	99
		#40	0.33	0.00	98
		#60	0.48	0.00	97
		#140	2.11	0.00	92
		#200	1.90	0.00	87

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =40.32
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	35.0	27.3	0.0142	35.0	10.6	0.0326	67.8
5.00	17.0	30.0	22.3	0.0142	30.0	11.4	0.0214	55.4
15.00	17.0	25.0	17.3	0.0142	25.0	12.2	0.0128	43.0
30.00	17.0	23.0	15.3	0.0142	23.0	12.5	0.0092	38.0
60.00	17.0	20.0	12.3	0.0142	20.0	13.0	0.0066	30.6
250.00	17.0	15.0	7.3	0.0142	15.0	13.8	0.0033	18.2
1440.00	17.0	12.0	4.3	0.0142	12.0	14.3	0.0014	10.7

Pace Analytical Services, Inc.

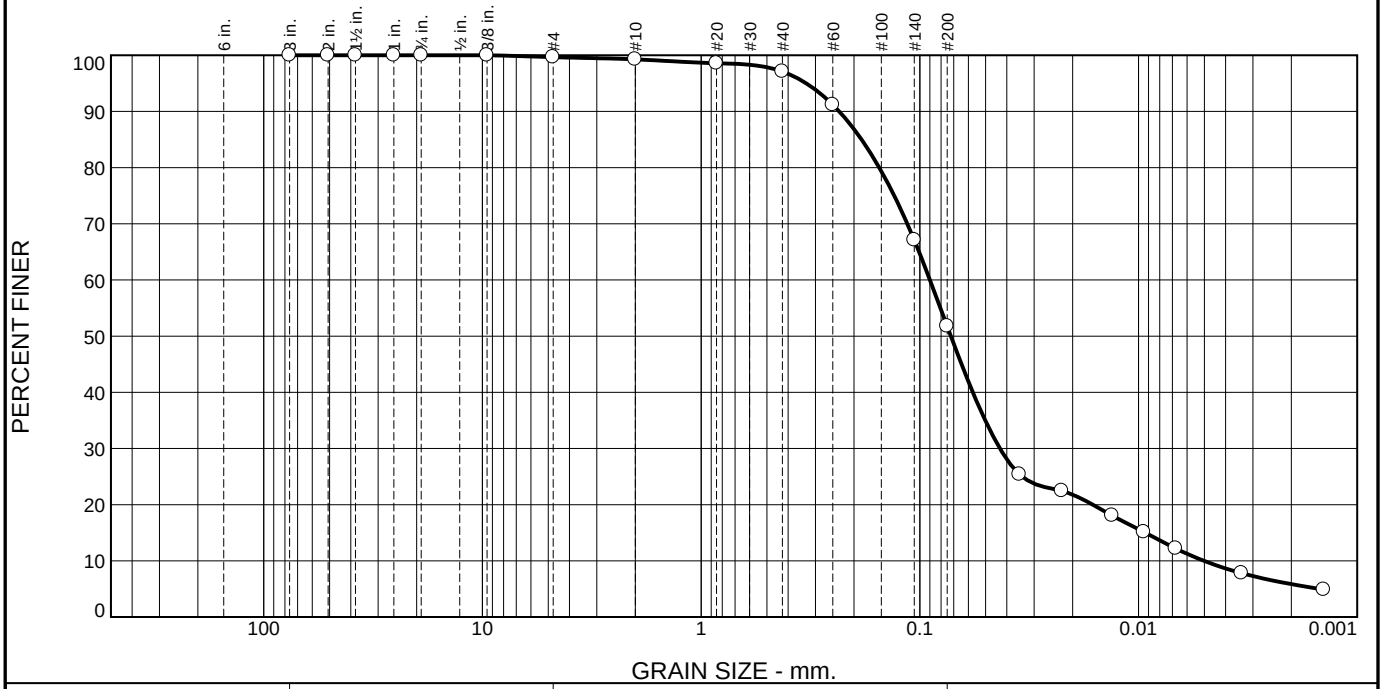
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	2	11	13	62	25	87

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
		0.0025	0.0038	0.0064	0.0104	0.0176	0.0250	0.0520	0.0658	0.0894	0.1496

Fineness Modulus
0.09

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	1	2	45	42	10

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	99		
#20	99		
#40	97		
#60	91		
#140	67		
#200	52		
0.0350 mm.	25		
0.0224 mm.	22		
0.0132 mm.	18		
0.0095 mm.	15		
0.0068 mm.	12		
0.0034 mm.	7.8		
0.0014 mm.	4.9		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.2338 D_{85}= 0.1847 D_{60}= 0.0899
D_{50}= 0.0721 D_{30}= 0.0430 D_{15}= 0.0093
D_{10}= 0.0050 C_u= 17.93 C_c= 4.11

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-013-0.11-0.36 **Date Sampled:** 9/23/16
Sample Number: 10365183-8

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-013-0.11-0.36
Sample Number: 10365183-8
Material Description: sandy silt
Sample Date: 9/23/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
810.34	641.57	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.57	0.00	100
		#10	0.67	0.00	99
67.76	0.00	#20	0.47	0.00	99
		#40	1.01	0.00	97
		#60	4.04	0.00	91
		#140	16.44	0.00	67
		#200	10.46	0.00	52

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 99
Weight of hydrometer sample =67.76
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	25.0	17.3	0.0142	25.0	12.2	0.0350	25.4
5.00	17.0	23.0	15.3	0.0142	23.0	12.5	0.0224	22.5
15.00	17.0	20.0	12.3	0.0142	20.0	13.0	0.0132	18.1
30.00	17.0	18.0	10.3	0.0142	18.0	13.3	0.0095	15.1
60.00	17.0	16.0	8.3	0.0142	16.0	13.7	0.0068	12.2
250.00	17.0	13.0	5.3	0.0142	13.0	14.2	0.0034	7.8
1440.00	17.0	11.0	3.3	0.0142	11.0	14.5	0.0014	4.9

Pace Analytical Services, Inc.

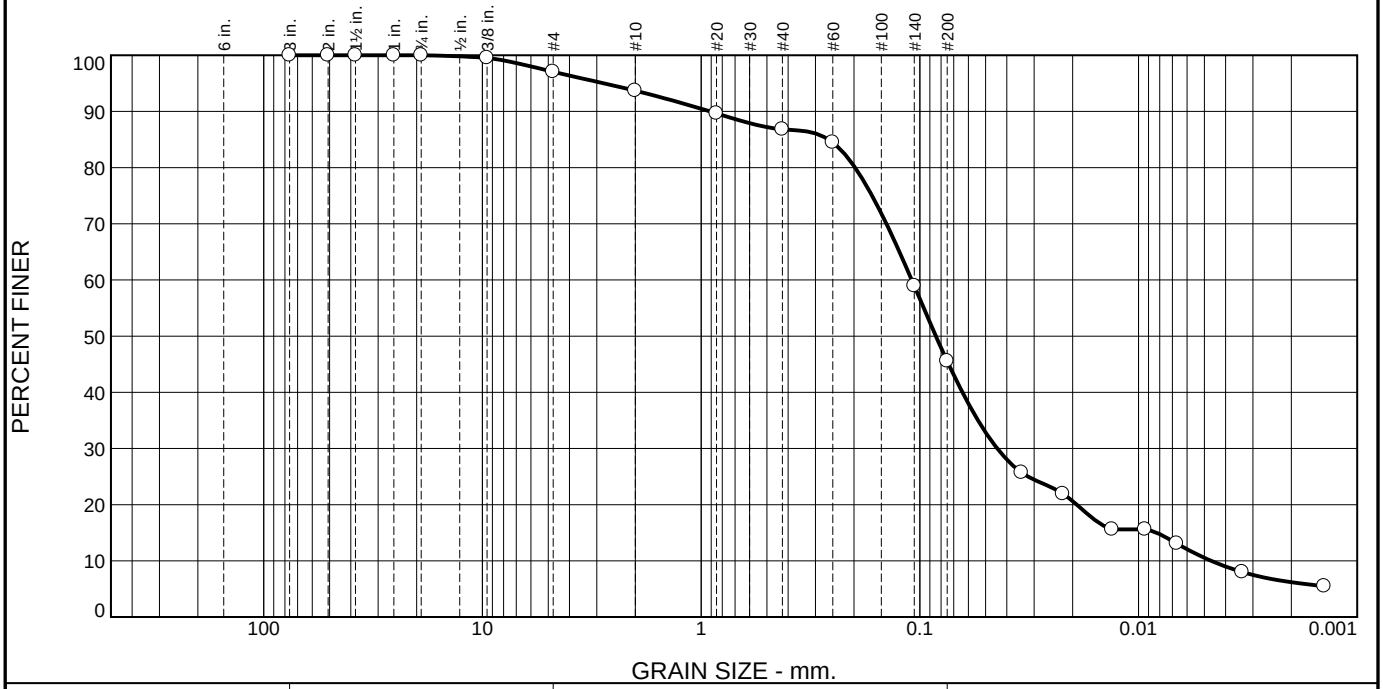
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	1	2	45	48	42	10	52

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0015	0.0050	0.0093	0.0162	0.0430	0.0572	0.0721	0.0899	0.1533	0.1847	0.2338	0.3304

Fineness Modulus	C_u	C_c
0.31	17.93	4.11

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	3	3	7	41	35	11

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	97		
#10	94		
#20	90		
#40	87		
#60	84		
#140	59		
#200	46		
0.0343 mm.	26		
0.0221 mm.	22		
0.0132 mm.	16		
0.0093 mm.	16		
0.0067 mm.	13		
0.0034 mm.	8.0		
0.0014 mm.	5.5		

* (no specification provided)

Material Description

silty sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= SM **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.9096 D_{85}= 0.2612 D_{60}= 0.1089
D_{50}= 0.0844 D_{30}= 0.0443 D_{15}= 0.0083
D_{10}= 0.0046 C_u= 23.42 C_c= 3.87

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-002-0.0-0.15
Sample Number: 10365183-9

Date Sampled: 9/30/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-002-0.0-0.15
Sample Number: 10365183-9
Material Description: silty sand
Sample Date: 9/30/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: SM **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1015.81	624.40	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	1.79	0.00	100
		#4	9.70	0.00	97
		#10	13.22	0.00	94
74.03	0.00	#20	3.19	0.00	90
		#40	2.24	0.00	87
		#60	1.85	0.00	84
		#140	20.17	0.00	59
		#200	10.59	0.00	46

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 94
Weight of hydrometer sample =74.03
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	28.0	20.3	0.0142	28.0	11.7	0.0343	25.7
5.00	17.0	25.0	17.3	0.0142	25.0	12.2	0.0221	21.9
15.00	17.0	20.0	12.3	0.0142	20.0	13.0	0.0132	15.6
30.00	17.0	20.0	12.3	0.0142	20.0	13.0	0.0093	15.6
60.00	17.0	18.0	10.3	0.0142	18.0	13.3	0.0067	13.1
250.00	17.0	14.0	6.3	0.0142	14.0	14.0	0.0034	8.0
1440.00	17.0	12.0	4.3	0.0142	12.0	14.3	0.0014	5.5

Pace Analytical Services, Inc.

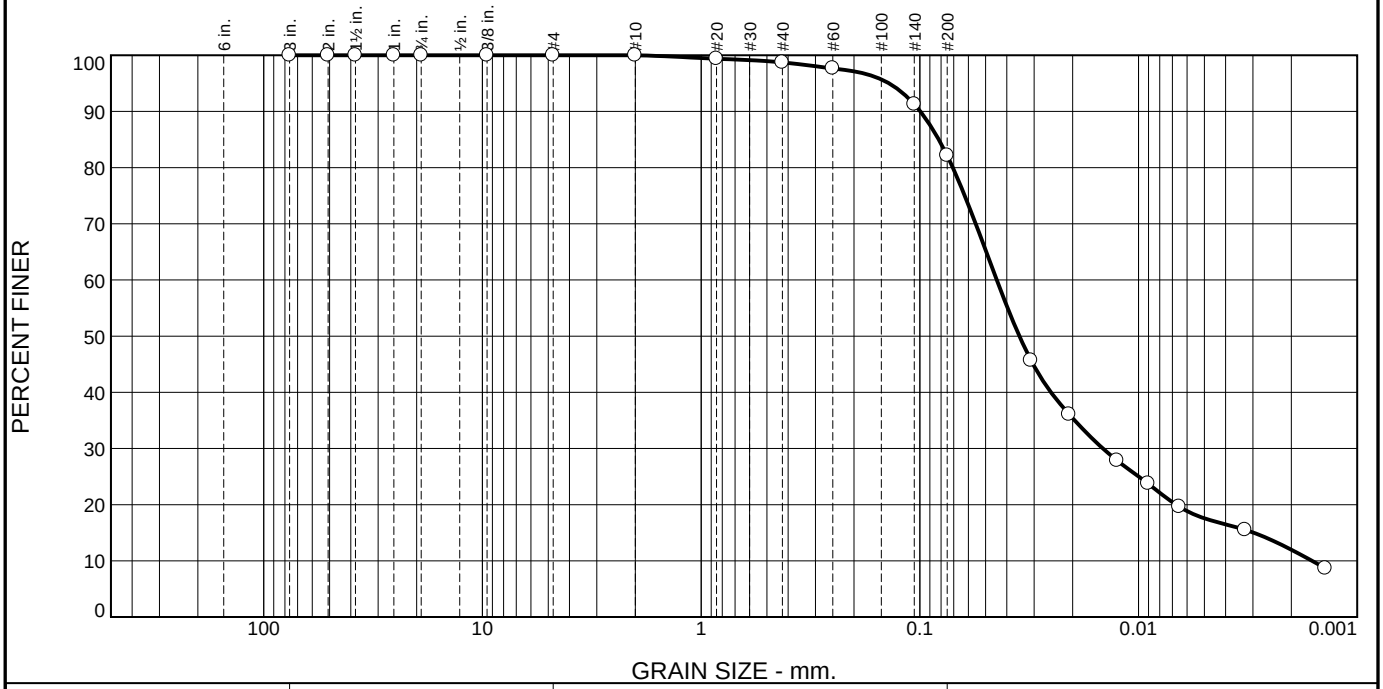
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	3	3	3	7	41	51	35	11	46

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0046	0.0083	0.0191	0.0443	0.0639	0.0844	0.1089	0.1977	0.2612	0.9096	2.7845

Fineness Modulus	C_u	C_c
0.72	23.42	3.87

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	17	64	18

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	99		
#60	98		
#140	91		
#200	82		
0.0310 mm.	46		
0.0208 mm.	36		
0.0125 mm.	28		
0.0090 mm.	24		
0.0065 mm.	20		
0.0033 mm.	16		
0.0014 mm.	8.7		

* (no specification provided)

Material Description

silt with sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0994 D_{85}= 0.0819 D_{60}= 0.0445
D_{50}= 0.0351 D_{30}= 0.0145 D_{15}= 0.0030
D_{10}= 0.0016 C_u= 27.66 C_c= 2.95

Remarks

Date Received: 10/6/16 **Date Tested:** 10/19/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-005-0.0-0.15
Sample Number: 10365183-10

Date Sampled: 9/30/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/20/2016

Client: Bay West, Inc
Project: J 160139 SLR Sediment AOCs
Location: BW16SR-005-0.0-0.15
Sample Number: 10365183-10
Material Description: silt with sand
Sample Date: 9/30/16
Date Received: 10/6/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/19/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1041.46	608.33	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
72.98	0.00	#20	0.45	0.00	99
		#40	0.48	0.00	99
		#60	0.78	0.00	98
		#140	4.65	0.00	91
		#200	6.66	0.00	82

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =72.98
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -7
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	17.0	41.0	33.3	0.0142	41.0	9.6	0.0310	45.7
5.00	17.0	34.0	26.3	0.0142	34.0	10.7	0.0208	36.1
15.00	17.0	28.0	20.3	0.0142	28.0	11.7	0.0125	27.9
30.00	17.0	25.0	17.3	0.0142	25.0	12.2	0.0090	23.7
60.00	17.0	22.0	14.3	0.0142	22.0	12.7	0.0065	19.6
250.00	17.0	19.0	11.3	0.0142	19.0	13.2	0.0033	15.5
1440.00	17.0	14.0	6.3	0.0142	14.0	14.0	0.0014	8.7

Pace Analytical Services, Inc.

Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	17	18	64	18	82

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0016	0.0030	0.0067	0.0145	0.0251	0.0351	0.0445	0.0706	0.0819	0.0994	0.1382

Fineness Modulus	C_u	C_c
0.07	27.66	2.95

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365188

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/19/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☐	☒	☐	

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☐	☒	

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☐	☒	☐	
b.	Are the lab recovery limits specified on the report?		☐	☐	☒	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☐	☐	☒	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☐	☒	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	

	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question			Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.		☒	☐	☐	
	i.	Have the required matrix spikes been prepared and reported?	☒	☐	☐	
	ii.	If no, is there and explanation in the report as to why?	☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?	☐	☐	☒	
	iv.	Are the lab limits specified on the report?	☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
	vi.	Are there compounds outside the lab limits?	☐	☒	☐	
		1. If yes, are the analytes above the lab limits?	☐	☐	☒	
		2. Below the lab limits?	☐	☐	☒	
		3. Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.		☐	☒	☐	RPDs discussed apply to MS/MSDs.
	i.	Is the RPD for the duplicate pair within the lab limits?	☒	☐	☐	
	ii.	If no, has the associated source sample been flagged?	☐	☐	☒	
c.	What is the impact of failed QC on this project?		☐	☐	☒	

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Samples BW16SR-012-0.0-0.25 and BW16SR-112-0.0-0.25 were collected as blind field duplicates. The mercury RPD (64.1%) was > the QC guideline of 50%. Mercury results in samples BW16SR-012-0.0-0.25 and BW16SR-112-0.0-0.25.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
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(612)607-1700

October 19, 2016

Nancy McDonald
Bay West Inc
5 Empire Drive
Saint Paul, MN 55103

RE: Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Dear Nancy McDonald:
Enclosed are the analytical results for sample(s) received by the laboratory on October 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Lori Castille
lori.castille@pacelabs.com
Project Manager

Enclosures

cc: Paul Raymaker, Bay West
Jeff Smith, Pace Analytical Services, Inc

Pace Analytical Services, LLC
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CERTIFICATIONS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
Alaska Certification UST-107
525 N 8th Street, Salina, KS 67401
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Virginia/VELAP Certification #: Pace
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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SAMPLE SUMMARY

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10365188001	**BW16SR-006-0.27-0.52**	Solid	09/23/16 09:37	10/06/16 20:25
10365188002	**BW16SR-007-0.06-0.31**	Solid	09/23/16 10:14	10/06/16 20:25
10365188003	**BW16SR-008-0.23-0.48**	Solid	09/23/16 10:33	10/06/16 20:25
10365188004	**BW16SR-009-0.17-0.42**	Solid	09/23/16 10:38	10/06/16 20:25
10365188005	**BW16SR-010-0.0-0.24**	Solid	09/23/16 10:50	10/06/16 20:25
10365188006	**BW16SR-011-0.16-0.41**	Solid	09/23/16 11:02	10/06/16 20:25
10365188007	**BW16SR-012-0.0-0.25**	Solid	09/23/16 11:20	10/06/16 20:25
10365188008	**BW16SR-112-0.0-0.25**	Solid	09/23/16 11:25	10/06/16 20:25
10365188009	**BW16SR-013-0.11-0.36**	Solid	09/23/16 11:33	10/06/16 20:25
10365188010	**BW16SR-002-0.0-0.15**	Solid	09/30/16 11:12	10/06/16 20:25

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SAMPLE ANALYTE COUNT

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Lab ID	Sample ID	Method	Analysts	Analytes Reported
10365188001	**BW16SR-006-0.27-0.52**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188002	**BW16SR-007-0.06-0.31**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188003	**BW16SR-008-0.23-0.48**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188004	**BW16SR-009-0.17-0.42**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188005	**BW16SR-010-0.0-0.24**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188006	**BW16SR-011-0.16-0.41**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188007	**BW16SR-012-0.0-0.25**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188008	**BW16SR-112-0.0-0.25**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188009	**BW16SR-013-0.11-0.36**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1
10365188010	**BW16SR-002-0.0-0.15**	EPA 7471B	LMW	1
		ASTM D2974	JDL	1

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PROJECT NARRATIVE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Method: **EPA 7471B**
Description: 7471B Mercury
Client: Bay West, Inc.
Date: October 19, 2016

General Information:
10 samples were analyzed for EPA 7471B. All samples were received in acceptable condition with any exceptions noted below or on the chain-of custody and/or the sample condition upon receipt form (SCUR) attached at the end of this report.

Hold Time:
The samples were analyzed within the method required hold times with any exceptions noted below.

Sample Preparation:
The samples were prepared in accordance with EPA 7471B with any exceptions noted below.

Initial Calibrations (including MS Tune as applicable):
All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:
All criteria were within method requirements with any exceptions noted below.

Method Blank:
All analytes were below the report limit in the method blank, where applicable, with any exceptions noted below.

Laboratory Control Spike:
All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:
All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

Additional Comments:

This data package has been reviewed for quality and completeness and is approved for release.

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-006-0.27-0.52 **Lab ID: 10365188001** Collected: 09/23/16 09:37 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.019J**	mg/kg	0.022	0.0057	1	10/07/16 11:15	10/17/16 15:49	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**23.4**	%	0.10	0.10	1		10/17/16 15:39		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-007-0.06-0.31 **Lab ID: 10365188002** Collected: 09/23/16 10:14 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.11**	mg/kg	0.028	0.0072	1	10/07/16 11:15	10/17/16 15:51	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**38.0**	%	0.10	0.10	1		10/17/16 15:40		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-008-0.23-0.48 **Lab ID: 10365188003** Collected: 09/23/16 10:33 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.54**	mg/kg	0.028	0.0073	1	10/07/16 11:15	10/17/16 15:53	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**37.2**	%	0.10	0.10	1		10/17/16 15:40		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-009-0.17-0.42 **Lab ID: 10365188004** Collected: 09/23/16 10:38 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.072**	mg/kg	0.026	0.0067	1	10/07/16 11:15	10/17/16 15:55	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**27.2**	%	0.10	0.10	1		10/17/16 15:40		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-010-0.0-0.24 **Lab ID: 10365188005** Collected: 09/23/16 10:50 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.099**	mg/kg	0.031	0.0081	1	10/07/16 11:15	10/17/16 15:57	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**45.2**	%	0.10	0.10	1		10/17/16 15:41		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-011-0.16-0.41 **Lab ID: 10365188006** Collected: 09/23/16 11:02 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.12**	mg/kg	0.027	0.0069	1	10/07/16 11:15	10/17/16 16:00	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**37.1**	%	0.10	0.10	1		10/17/16 15:41		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-012-0.0-0.25 **Lab ID: 10365188007** Collected: 09/23/16 11:20 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.35**	mg/kg	0.042	0.011	1	10/07/16 11:15	10/17/16 16:10	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**58.2**	%	0.10	0.10	1		10/18/16 10:58		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-112-0.0-0.25 **Lab ID: 10365188008** Collected: 09/23/16 11:25 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.68**	mg/kg	0.026	0.0066	1	10/07/16 11:15	10/17/16 16:12	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**24.1**	%	0.10	0.10	1		10/18/16 10:59		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-013-0.11-0.36 **Lab ID: 10365188009** Collected: 09/23/16 11:33 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.034**	mg/kg	0.028	0.0072	1	10/07/16 11:15	10/17/16 16:14	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**27.3**	%	0.10	0.10	1		10/18/16 10:59		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Sample: BW16SR-002-0.0-0.15 **Lab ID: 10365188010** Collected: 09/30/16 11:12 Received: 10/06/16 20:25 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.042**	mg/kg	0.040	0.010	1	10/07/16 11:15	10/17/16 16:17	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**57.0**	%	0.10	0.10	1		10/18/16 10:59		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

QC Batch: 439774 Analysis Method: EPA 7471B
QC Batch Method: EPA 7471B Analysis Description: 7471B Mercury Solids
Associated Lab Samples: 10365188001, 10365188002, 10365188003, 10365188004, 10365188005, 10365188006, 10365188007, 10365188008, 10365188009, 10365188010

METHOD BLANK: 2390958 Matrix: Solid
Associated Lab Samples: 10365188001, 10365188002, 10365188003, 10365188004, 10365188005, 10365188006, 10365188007, 10365188008, 10365188009, 10365188010

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/kg	ND	0.017	0.0045	10/17/16 15:45	

LABORATORY CONTROL SAMPLE: 2390959

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/kg	.45	0.47	103	80-120	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2390960 2390961

Parameter	Units	10365188006 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/kg	0.12	.68	.68	0.77	0.84	95	106	75-125	9	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

QC Batch: 441541 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365188001, 10365188002, 10365188003, 10365188004, 10365188005, 10365188006

SAMPLE DUPLICATE: 2403248

Parameter	Units	10365048013 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	26.6	25.8	3	30	

SAMPLE DUPLICATE: 2403249

Parameter	Units	10365188006 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	37.1	35.8	4	30	

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REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

QC Batch: 441644 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10365188007, 10365188008, 10365188009, 10365188010

SAMPLE DUPLICATE: 2404092

Parameter	Units	10365195006 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	88.3	88.5	0	30	

SAMPLE DUPLICATE: 2404495

Parameter	Units	10366384001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	17.9	16.5	8	30	

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REPORT OF LABORATORY ANALYSIS

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QUALIFIERS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365188

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10365188001	**BW16SR-006-0.27-0.52**	EPA 7471B	439774	EPA 7471B	440061
10365188002	**BW16SR-007-0.06-0.31**	EPA 7471B	439774	EPA 7471B	440061
10365188003	**BW16SR-008-0.23-0.48**	EPA 7471B	439774	EPA 7471B	440061
10365188004	**BW16SR-009-0.17-0.42**	EPA 7471B	439774	EPA 7471B	440061
10365188005	**BW16SR-010-0.0-0.24**	EPA 7471B	439774	EPA 7471B	440061
10365188006	**BW16SR-011-0.16-0.41**	EPA 7471B	439774	EPA 7471B	440061
10365188007	**BW16SR-012-0.0-0.25**	EPA 7471B	439774	EPA 7471B	440061
10365188008	**BW16SR-112-0.0-0.25**	EPA 7471B	439774	EPA 7471B	440061
10365188009	**BW16SR-013-0.11-0.36**	EPA 7471B	439774	EPA 7471B	440061
10365188010	**BW16SR-002-0.0-0.15**	EPA 7471B	439774	EPA 7471B	440061
10365188001	**BW16SR-006-0.27-0.52**	ASTM D2974	441541		
10365188002	**BW16SR-007-0.06-0.31**	ASTM D2974	441541		
10365188003	**BW16SR-008-0.23-0.48**	ASTM D2974	441541		
10365188004	**BW16SR-009-0.17-0.42**	ASTM D2974	441541		
10365188005	**BW16SR-010-0.0-0.24**	ASTM D2974	441541		
10365188006	**BW16SR-011-0.16-0.41**	ASTM D2974	441541		
10365188007	**BW16SR-012-0.0-0.25**	ASTM D2974	441644		
10365188008	**BW16SR-112-0.0-0.25**	ASTM D2974	441644		
10365188009	**BW16SR-013-0.11-0.36**	ASTM D2974	441644		
10365188010	**BW16SR-002-0.0-0.15**	ASTM D2974	441644		

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365188

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-1
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0.15	SO	G	3/12/15	1204													~~001~~
1	BW16SR-006	BW16SR-006-0.27-0.52	SO	G	9/23/16	0937	3	3								1	1	1	002
2	BW16SR-007	BW16SR-007-0.06-0.31	SO	G	9/23/16	1014	3	3								1	1	1	003
3	BW16SR-008	BW16SR-008-0.23-0.48	SO	G	9/23/16	1033	3	3								1	1	1	004
4	BW16SR-009	BW16SR-009-0.17-0.42	SO	G	9/23/16	1038	3	3								1	1	1	005
5	BW16SR-010	BW16SR-010-0.0-0.24	SO	G	9/23/16	1050	3	3								1	1	1	006
6	BW16SR-011	BW16SR-011-0.16-0.41	SO	G	9/23/16	1102	5	5								2	2	1	MS/MSD 007
7	BW16SR-012	BW16SR-012-0.0-0.25	SO	G	9/23/16	1120	3	3								1	1	1	008
8	BW16SR-012	BW16SR-112-0.0-0.25	SO	G	9/23/16	1125	3	3								1	1	1	009
9	BW16SR-013	BW16SR-013-0.11-0.36	SO	G	9/23/16	1133	3	3								1	1	1	010
10	BW16SR-002	BW16SR-002-0.0-0.15	SO	G	9/30/16	1112	3	3								1	1	1	
11																			
12																			

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16		10/6/16	1444	Kristina Polson	10/6/16	1445	Temp (°C) 4.9	Received on Ice (Y/N) Y	Custody Sealed Cooler (Y/N) N	Samples Intact (Y/N) Y
	Kristina Polson	10/6/16	1715		10/6/16	1715				
		10/6/16	2025							

SAMPLER NAME AND SIGNATURE		
PRINT Name of SAMPLER:	Chris Musson	
SIGNATURE of SAMPLER:		DATE Signed (MM/DD/YY): 10/6/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: WO# : 10365188

10365188

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ SpeeDee ☐ Other:_____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☐ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.9, 0.6, 4.7 **Cooler Temp Corrected (°C):** 0.2, 0.8, 4.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** CW 10.6.16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☑ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	~~☒ Yes~~	☒ No	☐ N/A	12. Did Recd a Dry Wt container for Samplation?
-Includes Date/Time/ID/Analysis Matrix: SL	CW 10.6.16			
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☒ N/A	Initial when completed: _____ Lot # of added preservative: _____
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased): _____				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: _____ **Date:** 10/7/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365190

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/21/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☒	☐	☐	The response obtained for the native OCDF in the calibration standard analyses U161012A_17 was outside the target range. As specified in the Pace procedures, the

					average of the daily response factors for this compound was used in the calculations for the samples from this analytical run. The affected values were flagged "Y" on the results tables. No data were qualified.

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	Low-level concentrations of Total TCDF, Total HxCDD, 1,2,3,4,6,7,8-HpCDD, and Total HpCDD were detected in the method blank.
	i.	If yes, are the same compounds present in the samples?	☒	☐	☐	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☒	☐	All sample results were > 10x the blank concentration.

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☒	☐	☐	Dioxins/furans have internal standards instead of surrogates.
b.	Are the lab recovery limits specified on the report?		☒	☐	☐	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☒	☐	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	

	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	
	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question				Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.			☐	☒	☐	
	i.	Have the required matrix spikes been prepared and reported?		☒	☐	☐	
	ii.	If no, is there and explanation in the report as to why?		☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?		☐	☐	☒	
	iv.	Are the lab limits specified on the report?		☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?		☒	☐	☐	
	vi.	Are there compounds outside the lab limits?		☒	☐	☐	
		1.	If yes, are the analytes above the lab limits?	☒	☐	☐	Background-subtracted recoveries for 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,6,7,8-HpCDD, OCDF, and OCDD were biased high and outside QC limits in the MS/MSD performed on sample BW16SR-011-0.16-0.41.
		2.	Below the lab limits?	☐	☒	☐	
		3.	Is the source sample also flagged for compounds outside lab limits?	☐	☒	☐	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.			☐	☐	☒	RPDs apply to MS/MSD sample BW16SR-011-0.16-0.41.
	i.	Is the RPD for the duplicate pair within the lab limits?		☐	☒	☐	RPDs for 1,2,3,4,6,7,8-HpCDF, OCDF, and OCDD were above the QC criterion in sample BW16SR-011-0.16-0.41.
	ii.	If no, has the associated source sample been flagged?		☐	☒	☐	
c.	What is the impact of failed QC on this project?			☐	☐	☒	Results for 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,6,7,8-HpCDD, OCDF, and OCDD were qualified "J" as estimated in parent sample BW16SR-011-0.16-0.41.

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report: **X**

(1) Samples BW16SR-012-0.0-0.25 and Samples BW16SR-112-0.0-0.25 field duplicates were included in this SDG All RPDs were within the QC guideline of ≤ 50% except for the following. RPDs for 2,3,7,8-TCDF (82.0%), Total TCDF (82.0%), 2,3,7,8-TCDD (82.0%), 2,3,7,8-PeCDF (153%), Total PeCDF (181%), 1,2,3,7,8-PeCDD (66.7%), Total PeCDD (64.8%), 1,2,3,4,7,8-HxCDF (55.6%), 2,3,4,6,7,8-HxCDF (62.5%), 1,2,3,4,7,8-HxCDD (88.7%), 1,2,3,6,7,8-HxCDD (66.7%), Total HxCDD (75.0%), 1,2,3,4,7,8,9-HpCDF (76.9%), Total HpCDF (59.6%), 1,2,3,4,6,7,8-HpCDD (89.7%), Total HpCDD (93.7%), and OCDD (143%) exceeded the QC guideline. Results for 2,3,7,8-TCDF, Total TCDF, 2,3,7,8-TCDD, 2,3,7,8-PeCDF, Total PeCDF, 1,2,3,7,8-PeCDD, Total PeCDD, 1,2,3,4,7,8-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,4,7,8-HxCDD, 1,2,3,6,7,8-HxCDD, Total HxCDD, 1,2,3,4,7,8,9-HpCDF, Total HpCDF, 1,2,3,4,6,7,8-HpCDD, Total HpCDD, and OCDD were qualified “J” as estimated in samples BW16SR-012-0.0-0.25 and Samples BW16SR-112-0.0-0.25.

(2) In some cases, interfering substances impacted the determinations of PCDD or PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where polychlorinated diphenyl ethers were present. Concentrations below the calibration range were flagged "J" and should be regarded as estimates. Concentrations above the calibration range were flagged "E" and should also be regarded as estimates. The OCDD result in one sample was obtained from the analysis of a diluted extract.

(3) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

www.pacelabs.com

Report Prepared for:

Nancy McDonald
Bay West, Inc.
5 Empire Drive
Saint Paul MN 55103

REPORT OF LABORATORY ANALYSIS FOR PCDD/PCDF

Report Prepared Date:
October 21, 2016

Report Information:

Pace Project #: 10365190
Sample Receipt Date: 10/06/2016
Client Project #: J160139 SLR Sediment AOCs
Client Sub PO #: 108002
State Cert #: 027-053-137

Invoicing & Reporting Options:

The report provided has been invoiced as a Level 2 PCDD/PCDF Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

This report has been reviewed by:

October 21, 2016

Carolynne Trout, Project Manager
(612) 607-6351
(612) 607-6444 (fax)
Carolynne.Trout@pacelabs.com

Report of Laboratory Analysis

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

DISCUSSION

This report presents the results from the analyses performed on ten samples submitted by a representative of BayWest, Inc. The samples were analyzed for the presence or absence of polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs) using a modified version of USEPA Method 8290. The reporting limits were based on signal-to-noise measurements. Estimated Maximum Possible Concentration (EMPC) values were treated as positives in the toxic equivalence calculations.

Second column confirmation analyses of 2,3,7,8-TCDF values obtained from the primary (DB5-MS) column are performed only when specifically requested for a project and only when the values are above the concentration of the lowest calibration standard. Typical resolution for this isomer using the DB5-MS column ranges from 25-30%.

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extracts ranged from 40-94%. All the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. Also, since the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained.

In some cases, interfering substances impacted the determinations of PCDD or PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where polychlorinated diphenyl ethers were present. Concentrations below the calibration range were flagged "J" and should be regarded as estimates. Concentrations above the calibration range were flagged "E" and should also be regarded as estimates. One OCDD value was obtained from the analysis of a diluted extract and was flagged "D" and "N2".

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to contain trace levels of selected congeners. These levels were below the calibration range of the method. The levels reported for the affected congeners in the field samples were higher than the corresponding blank levels by one or more orders of magnitude. These results indicate that the sample processing steps did not contribute significantly to the levels reported for the field samples.

Laboratory and matrix spike samples were also prepared with the sample batch using clean sand or sample matrix that had been fortified with native standard materials. The results show that the spiked native compounds were generally recovered at 81-124% with relative percent differences (RPDs) generally from 0.5-10.8%. The background-subtracted recovery values obtained for 1,2,3,4,6,7,8-HxCDF, HpCDD, OCDF, and OCDD in the matrix spike and/or matrix spike duplicate were above the 70-130% target range. Also, the RPD values obtained for HpCDD, OCDF, and OCDD were above the 20% target upper limit. These deviations may be due to the levels of the affected congeners in the sample material and/or sample inhomogeneity.

The response obtained for the native OCDF in calibration standard analysis U161012A_17 was outside the target range. As specified in the method, the average of the daily response factors for this compound was used in the calculations for the samples from this runshift. The affected values were flagged "Y" on the results tables. It should be noted that the accuracy of the native congener determinations for submitted field samples was not impacted by this deviation.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Montana	92
Alaska	MN00064	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN_00064_200
Arkansas	88-0680	New Jersey (NE	MN002
California	01155CA	New York (NEL	11647
Colorado	MN00064	North Carolina	27700
Connecticut	PH-0256	North Dakota	R-036
EPA Region 8	8TMS-Q	Ohio	4150
Florida (NELAP	E87605	Oklahoma	D9922
Georgia (DNR)	959	Oregon (ELAP)	MN200001-005
Guam	959	Oregon (OREL	MN300001-001
Hawaii	SLD	Pennsylvania	68-00563
Idaho	MN00064	Puerto Rico	MN00064
Illinois	200012	Saipan	MP0003
Indiana	C-MN-01	South Carolina	74003001
Indiana	C-MN-01	Tennessee	TN02818
Iowa	368	Texas	T104704192-08
Kansas	E-10167	Utah (NELAP)	MN00064
Kentucky	90062	Virginia	00251
Louisiana	03086	Washington	C755
Maine	2007029	West Virginia #	9952C
Maryland	322	West Virginia D	382
Michigan	9909	Wisconsin	999407970
Minnesota	027-053-137	Wyoming	8TMS-Q

REPORT OF LABORATORY ANALYSIS

Appendix A

Sample Management

Report No....10365190_8290

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365190

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# : SLR-SR-1
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	*BW15MLW-005*	*BW14MLW-005-0-0.15*	SO	G	*3/12/15*	*1204*													
1	BW16SR-006	BW16SR-006-0.27-0.52	SO	G	9/23/16	0937	3	3								1	1	1	cw1
2	BW16SR-007	BW16SR-007-0.06-0.31	SO	G	9/23/16	1014	3	3								1	1	1	cw2
3	BW16SR-008	BW16SR-008-0.23-0.48	SO	G	9/23/16	1033	3	3								1	1	1	cw3
4	BW16SR-009	BW16SR-009-0.17-0.42	SO	G	9/23/16	1038	3	3								1	1	1	cw4
5	BW16SR-010	BW16SR-010-0.0-0.24	SO	G	9/23/16	1050	3	3								1	1	1	cw5
6	BW16SR-011	BW16SR-011-0.16-0.41	SO	G	9/23/16	1102	5	5								2	2	1	MS/MSD cw6
7	BW16SR-012	BW16SR-012-0.0-0.25	SO	G	9/23/16	1120	3	3								1	1	1	cw7
8	BW16SR-012	BW16SR-112-0.0-0.25	SO	G	9/23/16	1125	3	3								1	1	1	cw8
9	BW16SR-013	BW16SR-013-0.11-0.36	SO	G	9/23/16	1133	3	3								1	1	1	cw9
10	BW16SR-002	BW16SR-002-0.0-0.15	SO	G	9/30/16	1112	3	3								1	1	1	cw10
11																			
12																			

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16		10/6/16	1444	Kristina Polson	10/6/16	1445	4.9	Y	N	Y
	Kristina Polson	10/6/16	1715		10/6/16	1715				
		10/6/16	2025							

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: CJM DATE Signed (MM/DD/YY): 10/6/16

Pace Analytical®	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: WO# : 10365190

10365190

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☒ Pace ☐ SpeeDee ☐ Other:_____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☐ Yes ☐ No

Thermometer Used: ☒ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 09, 0.6, 4.7 **Cooler Temp Corrected (°C):** 0.2, 0.8, 4.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☒ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** CLT 10.6.16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	☒ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☒ N/A	Initial when completed:_____ Lot # of added preservative:_____
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased):_____				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: Carolynne Trout **Date:** 10/10/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Reporting Flags

A = Reporting Limit based on signal to noise

B = Less than 10x higher than method blank level

C = Result obtained from confirmation analysis

D = Result obtained from analysis of diluted sample

E = Exceeds calibration range

I = Interference present

J = Estimated value

Nn = Value obtained from additional analysis

P = PCDE Interference

R = Recovery outside target range

S = Peak saturated

U = Analyte not detected

V = Result verified by confirmation analysis

X = %D Exceeds limits

Y = Calculated using average of daily RFs

* = See Discussion

Appendix B

Sample Analysis Summary

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-006-0.27-0.52		
Lab Sample ID	10365190001		
Filename	Y161012B_06		
Injected By	SMT		
Total Amount Extracted	17.4 g	Matrix	Solid
% Moisture	24.9	Dilution	NA
Dry Weight Extracted	13.1 g	Collected	09/23/2016 09:37
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 16:25

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.34	-----	0.18	J	2,3,7,8-TCDF-13C	2.00	84
Total TCDF	3.10	-----	0.18		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	76
2,3,7,8-TCDD	ND	-----	0.23		2,3,4,7,8-PeCDF-13C	2.00	72
Total TCDD	1.40	-----	0.23		1,2,3,7,8-PeCDD-13C	2.00	73
					1,2,3,4,7,8-HxCDF-13C	2.00	86
1,2,3,7,8-PeCDF	ND	-----	0.16		1,2,3,6,7,8-HxCDF-13C	2.00	80
2,3,4,7,8-PeCDF	ND	-----	0.12		2,3,4,6,7,8-HxCDF-13C	2.00	87
Total PeCDF	ND	-----	0.14		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	80
1,2,3,7,8-PeCDD	ND	-----	0.22		1,2,3,6,7,8-HxCDD-13C	2.00	70
Total PeCDD	0.38	-----	0.22	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	61
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	ND	-----	0.15		1,2,3,4,6,7,8-HpCDD-13C	2.00	61
1,2,3,6,7,8-HxCDF	ND	-----	0.28		OCDD-13C	4.00	58
2,3,4,6,7,8-HxCDF	ND	-----	0.35				
1,2,3,7,8,9-HxCDF	ND	-----	0.29		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.27		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.42		2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	ND	-----	0.41				
1,2,3,7,8,9-HxCDD	ND	-----	0.40				
Total HxCDD	3.70	-----	0.41	J			
1,2,3,4,6,7,8-HpCDF	-----	0.56	0.46	IJ	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	0.87	0.65	IJ	Equivalence: 0.060 ng/Kg		
Total HpCDF	ND	-----	0.55		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	1.00	-----	0.51	J			
Total HpCDD	2.10	-----	0.51	J			
OCDF	ND	-----	0.90				
OCDD	5.80	-----	1.50	J			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-007-0.06-0.31		
Lab Sample ID	10365190002		
Filename	Y161012B_07		
Injected By	SMT		
Total Amount Extracted	12.6 g	Matrix	Solid
% Moisture	37.4	Dilution	NA
Dry Weight Extracted	7.89 g	Collected	09/23/2016 10:14
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 17:08

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	8.4	-----	0.32		2,3,7,8-TCDF-13C	2.00	81
Total TCDF	55.0	-----	0.32		2,3,7,8-TCDD-13C	2.00	85
					1,2,3,7,8-PeCDF-13C	2.00	59
2,3,7,8-TCDD	2.3	-----	0.39		2,3,4,7,8-PeCDF-13C	2.00	50
Total TCDD	38.0	-----	0.39		1,2,3,7,8-PeCDD-13C	2.00	56
					1,2,3,4,7,8-HxCDF-13C	2.00	84
1,2,3,7,8-PeCDF	-----	2.4	0.65	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	72
2,3,4,7,8-PeCDF	22.0	-----	0.70		2,3,4,6,7,8-HxCDF-13C	2.00	74
Total PeCDF	160.0	-----	0.67		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	70
1,2,3,7,8-PeCDD	5.4	-----	0.54	J	1,2,3,6,7,8-HxCDD-13C	2.00	68
Total PeCDD	97.0	-----	0.54		1,2,3,4,6,7,8-HpCDF-13C	2.00	55
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61
1,2,3,4,7,8-HxCDF	150.0	-----	1.50		1,2,3,4,6,7,8-HpCDD-13C	2.00	69
1,2,3,6,7,8-HxCDF	40.0	-----	1.10		OCDD-13C	4.00	76
2,3,4,6,7,8-HxCDF	94.0	-----	1.40				
1,2,3,7,8,9-HxCDF	71.0	-----	1.70		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	18000.0	-----	1.40	E	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	3.20		2,3,7,8-TCDD-37Cl4	0.20	80
1,2,3,6,7,8-HxCDD	1800.0	-----	3.60				
1,2,3,7,8,9-HxCDD	69.0	-----	3.20				
Total HxCDD	4600.0	-----	3.40				
1,2,3,4,6,7,8-HpCDF	12000.0	-----	0.69	E	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	630.0	-----	0.88		Equivalence: 800 ng/Kg		
Total HpCDF	13000.0	-----	0.79	E	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	37000.0	-----	0.48	E			
Total HpCDD	58000.0	-----	0.48	E			
OCDF	63000.0	-----	1.30	E			
OCDD	160000.0	-----	2.40	EDN2			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
E = Exceeds calibration range
I = Interference present
D = Result obtained from analysis of diluted sample
Nn = Value obtained from additional analysis

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-008-0.23-0.48		
Lab Sample ID	10365190003		
Filename	Y161012B_08		
Injected By	SMT		
Total Amount Extracted	14.0 g	Matrix	Solid
% Moisture	39.1	Dilution	NA
Dry Weight Extracted	8.53 g	Collected	09/23/2016 10:33
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 17:51

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	6.1	-----	0.72		2,3,7,8-TCDF-13C	2.00	80
Total TCDF	28.0	-----	0.72		2,3,7,8-TCDD-13C	2.00	87
					1,2,3,7,8-PeCDF-13C	2.00	69
2,3,7,8-TCDD	4.8	-----	0.65		2,3,4,7,8-PeCDF-13C	2.00	60
Total TCDD	45.0	-----	0.65		1,2,3,7,8-PeCDD-13C	2.00	69
					1,2,3,4,7,8-HxCDF-13C	2.00	81
1,2,3,7,8-PeCDF	4.6	-----	0.65	J	1,2,3,6,7,8-HxCDF-13C	2.00	71
2,3,4,7,8-PeCDF	9.4	-----	0.39		2,3,4,6,7,8-HxCDF-13C	2.00	70
Total PeCDF	90.0	-----	0.52		1,2,3,7,8,9-HxCDF-13C	2.00	76
					1,2,3,4,7,8-HxCDD-13C	2.00	70
1,2,3,7,8-PeCDD	15.0	-----	0.61		1,2,3,6,7,8-HxCDD-13C	2.00	67
Total PeCDD	130.0	-----	0.61		1,2,3,4,6,7,8-HpCDF-13C	2.00	53
					1,2,3,4,7,8,9-HpCDF-13C	2.00	55
1,2,3,4,7,8-HxCDF	39.0	-----	0.62		1,2,3,4,6,7,8-HpCDD-13C	2.00	64
1,2,3,6,7,8-HxCDF	37.0	-----	0.60		OCDD-13C	4.00	56
2,3,4,6,7,8-HxCDF	17.0	-----	0.42				
1,2,3,7,8,9-HxCDF	14.0	-----	0.84		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	1700.0	-----	0.62		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	12.0	-----	0.62		2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	63.0	-----	0.86				
1,2,3,7,8,9-HxCDD	40.0	-----	0.22				
Total HxCDD	610.0	-----	0.57				
1,2,3,4,6,7,8-HpCDF	3700.0	-----	2.30		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	33.0	-----	1.30		Equivalence: 96 ng/Kg		
Total HpCDF	7000.0	-----	1.80		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	930.0	-----	0.83				
Total HpCDD	2000.0	-----	0.83				
OCDF	1900.0	-----	1.90				
OCDD	11000.0	-----	2.10	E			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
E = Exceeds calibration range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-009-0.17-0.42		
Lab Sample ID	10365190004		
Filename	Y161012B_09		
Injected By	SMT		
Total Amount Extracted	14.0 g	Matrix	Solid
% Moisture	30.0	Dilution	NA
Dry Weight Extracted	9.80 g	Collected	09/23/2016 10:38
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 18:34

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	2.10	-----	0.29		2,3,7,8-TCDF-13C	2.00	79
Total TCDF	9.20	-----	0.29		2,3,7,8-TCDD-13C	2.00	84
					1,2,3,7,8-PeCDF-13C	2.00	73
2,3,7,8-TCDD	0.58	-----	0.38	J	2,3,4,7,8-PeCDF-13C	2.00	68
Total TCDD	5.70	-----	0.38		1,2,3,7,8-PeCDD-13C	2.00	71
					1,2,3,4,7,8-HxCDF-13C	2.00	92
1,2,3,7,8-PeCDF	0.59	-----	0.34	J	1,2,3,6,7,8-HxCDF-13C	2.00	77
2,3,4,7,8-PeCDF	-----	0.80	0.22	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	84
Total PeCDF	8.70	-----	0.28		1,2,3,7,8,9-HxCDF-13C	2.00	84
					1,2,3,4,7,8-HxCDD-13C	2.00	77
1,2,3,7,8-PeCDD	-----	0.81	0.44	IJ	1,2,3,6,7,8-HxCDD-13C	2.00	71
Total PeCDD	13.00	-----	0.44		1,2,3,4,6,7,8-HpCDF-13C	2.00	60
					1,2,3,4,7,8,9-HpCDF-13C	2.00	62
1,2,3,4,7,8-HxCDF	1.60	-----	0.40	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66
1,2,3,6,7,8-HxCDF	3.40	-----	0.28	J	OCDD-13C	4.00	62
2,3,4,6,7,8-HxCDF	1.70	-----	0.33	J			
1,2,3,7,8,9-HxCDF	1.20	-----	0.60	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	78.00	-----	0.40		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	1.30	-----	0.28	J	2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	7.10	-----	0.26				
1,2,3,7,8,9-HxCDD	3.10	-----	0.24	J			
Total HxCDD	68.00	-----	0.26				
1,2,3,4,6,7,8-HpCDF	85.00	-----	0.69		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	2.00	0.60	IJ	Equivalence: 6.6 ng/Kg		
Total HpCDF	210.00	-----	0.64		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	130.00	-----	1.00				
Total HpCDD	300.00	-----	1.00				
OCDF	76.00	-----	1.30				
OCDD	1900.00	-----	0.93				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
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ND = Not Detected
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Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
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REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-010-0.0-0.24		
Lab Sample ID	10365190005		
Filename	Y161012B_10		
Injected By	SMT		
Total Amount Extracted	14.8 g	Matrix	Solid
% Moisture	44.5	Dilution	NA
Dry Weight Extracted	8.21 g	Collected	09/23/2016 10:50
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 19:16

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	3.2	-----	0.29		2,3,7,8-TCDF-13C	2.00	87
Total TCDF	21.0	-----	0.29		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	73
2,3,7,8-TCDD	2.2	-----	0.41		2,3,4,7,8-PeCDF-13C	2.00	59
Total TCDD	24.0	-----	0.41		1,2,3,7,8-PeCDD-13C	2.00	65
					1,2,3,4,7,8-HxCDF-13C	2.00	94
1,2,3,7,8-PeCDF	1.9	-----	0.44	J	1,2,3,6,7,8-HxCDF-13C	2.00	81
2,3,4,7,8-PeCDF	4.8	-----	0.34	J	2,3,4,6,7,8-HxCDF-13C	2.00	86
Total PeCDF	60.0	-----	0.39		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	82
1,2,3,7,8-PeCDD	9.8	-----	0.66		1,2,3,6,7,8-HxCDD-13C	2.00	69
Total PeCDD	95.0	-----	0.66		1,2,3,4,6,7,8-HpCDF-13C	2.00	61
					1,2,3,4,7,8,9-HpCDF-13C	2.00	68
1,2,3,4,7,8-HxCDF	14.0	-----	0.55		1,2,3,4,6,7,8-HpCDD-13C	2.00	73
1,2,3,6,7,8-HxCDF	18.0	-----	0.49		OCDD-13C	4.00	68
2,3,4,6,7,8-HxCDF	8.4	-----	0.50				
1,2,3,7,8,9-HxCDF	5.4	-----	0.64	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	760.0	-----	0.55		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	9.5	-----	0.56		2,3,7,8-TCDD-37Cl4	0.20	86
1,2,3,6,7,8-HxCDD	44.0	-----	0.48				
1,2,3,7,8,9-HxCDD	29.0	-----	0.48				
Total HxCDD	410.0	-----	0.51				
1,2,3,4,6,7,8-HpCDF	1700.0	-----	1.10		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	12.0	-----	1.30		Equivalence: 48 ng/Kg		
Total HpCDF	3100.0	-----	1.20		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	310.0	-----	1.10				
Total HpCDD	610.0	-----	1.10				
OCDF	690.0	-----	1.40				
OCDD	2300.0	-----	0.95				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-011-0.16-0.41		
Lab Sample ID	10365190006		
Filename	Y161012B_11		
Injected By	SMT		
Total Amount Extracted	14.0 g	Matrix	Solid
% Moisture	39.0	Dilution	NA
Dry Weight Extracted	8.54 g	Collected	09/23/2016 11:02
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 19:59

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	7.6	-----	0.49		2,3,7,8-TCDF-13C	2.00	86
Total TCDF	34.0	-----	0.49		2,3,7,8-TCDD-13C	2.00	90
					1,2,3,7,8-PeCDF-13C	2.00	74
2,3,7,8-TCDD	2.5	-----	0.55		2,3,4,7,8-PeCDF-13C	2.00	68
Total TCDD	24.0	-----	0.55		1,2,3,7,8-PeCDD-13C	2.00	74
					1,2,3,4,7,8-HxCDF-13C	2.00	87
1,2,3,7,8-PeCDF	1.6	-----	0.68	J	1,2,3,6,7,8-HxCDF-13C	2.00	73
2,3,4,7,8-PeCDF	3.3	-----	0.54	J	2,3,4,6,7,8-HxCDF-13C	2.00	78
Total PeCDF	52.0	-----	0.61		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	78
1,2,3,7,8-PeCDD	5.3	-----	0.73	J	1,2,3,6,7,8-HxCDD-13C	2.00	70
Total PeCDD	46.0	-----	0.73		1,2,3,4,6,7,8-HpCDF-13C	2.00	59
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61
1,2,3,4,7,8-HxCDF	6.5	-----	0.91		1,2,3,4,6,7,8-HpCDD-13C	2.00	63
1,2,3,6,7,8-HxCDF	14.0	-----	0.62		OCDD-13C	4.00	65
2,3,4,6,7,8-HxCDF	6.1	-----	0.55				
1,2,3,7,8,9-HxCDF	2.5	-----	1.00	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	350.0	-----	0.78		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	4.1	-----	0.91	J	2,3,7,8-TCDD-37Cl4	0.20	85
1,2,3,6,7,8-HxCDD	31.0	-----	0.78				
1,2,3,7,8,9-HxCDD	14.0	-----	0.93				
Total HxCDD	300.0	-----	0.87				
1,2,3,4,6,7,8-HpCDF	390.0	-----	1.00		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	8.7	0.72	I	Equivalence: 32 ng/Kg		
Total HpCDF	990.0	-----	0.86		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	680.0	-----	1.30				
Total HpCDD	1600.0	-----	1.30				
OCDF	420.0	-----	1.70				
OCDD	11000.0	-----	1.80	E			

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E = Exceeds calibration range
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-012-0.0-0.25		
Lab Sample ID	10365190007		
Filename	Y161012B_12		
Injected By	SMT		
Total Amount Extracted	16.3 g	Matrix	Solid
% Moisture	56.4	Dilution	NA
Dry Weight Extracted	7.11 g	Collected	09/23/2016 11:20
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 20:42

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	23.0	-----	0.55		2,3,7,8-TCDF-13C	2.00	81
Total TCDF	86.0	-----	0.55		2,3,7,8-TCDD-13C	2.00	82
					1,2,3,7,8-PeCDF-13C	2.00	56
2,3,7,8-TCDD	6.5	-----	0.63		2,3,4,7,8-PeCDF-13C	2.00	46
Total TCDD	48.0	-----	0.63		1,2,3,7,8-PeCDD-13C	2.00	52
					1,2,3,4,7,8-HxCDF-13C	2.00	86
1,2,3,7,8-PeCDF	-----	3.0	0.83	PJ	1,2,3,6,7,8-HxCDF-13C	2.00	73
2,3,4,7,8-PeCDF	5.6	-----	0.68	J	2,3,4,6,7,8-HxCDF-13C	2.00	73
Total PeCDF	89.0	-----	0.76		1,2,3,7,8,9-HxCDF-13C	2.00	74
					1,2,3,4,7,8-HxCDD-13C	2.00	80
1,2,3,7,8-PeCDD	9.5	-----	0.37		1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	97.0	-----	0.37		1,2,3,4,6,7,8-HpCDF-13C	2.00	46
					1,2,3,4,7,8,9-HpCDF-13C	2.00	46
1,2,3,4,7,8-HxCDF	13.0	-----	0.55		1,2,3,4,6,7,8-HpCDD-13C	2.00	52
1,2,3,6,7,8-HxCDF	20.0	-----	0.43		OCDD-13C	4.00	40
2,3,4,6,7,8-HxCDF	11.0	-----	0.50				
1,2,3,7,8,9-HxCDF	4.7	-----	1.10	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	570.0	-----	0.66		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	8.1	-----	0.99		2,3,7,8-TCDD-37Cl4	0.20	77
1,2,3,6,7,8-HxCDD	60.0	-----	0.73				
1,2,3,7,8,9-HxCDD	27.0	-----	0.78				
Total HxCDD	500.0	-----	0.83				
1,2,3,4,6,7,8-HpCDF	790.0	-----	1.10		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	16.0	1.40	I	Equivalence: 57 ng/Kg		
Total HpCDF	2000.0	-----	1.30		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	990.0	-----	2.20				
Total HpCDD	2100.0	-----	2.20				
OCDF	1100.0	-----	1.90				
OCDD	12000.0	-----	1.90				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

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NC = Not Calculated

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J = Estimated value
P = PCDE Interference
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-112-0.0-0.25		
Lab Sample ID	10365190008		
Filename	Y161012B_13		
Injected By	SMT		
Total Amount Extracted	17.1 g	Matrix	Solid
% Moisture	68.3	Dilution	NA
Dry Weight Extracted	5.42 g	Collected	09/23/2016 11:25
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 21:24

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	55.0	-----	0.96		2,3,7,8-TCDF-13C	2.00	83
Total TCDF	160.0	-----	0.96		2,3,7,8-TCDD-13C	2.00	84
					1,2,3,7,8-PeCDF-13C	2.00	66
2,3,7,8-TCDD	17.0	-----	1.30		2,3,4,7,8-PeCDF-13C	2.00	58
Total TCDD	98.0	-----	1.30		1,2,3,7,8-PeCDD-13C	2.00	64
					1,2,3,4,7,8-HxCDF-13C	2.00	74
1,2,3,7,8-PeCDF	-----	4.0	1.20	PJ	1,2,3,6,7,8-HxCDF-13C	2.00	68
2,3,4,7,8-PeCDF	9.6	-----	1.10		2,3,4,6,7,8-HxCDF-13C	2.00	70
Total PeCDF	190.0	-----	1.10		1,2,3,7,8,9-HxCDF-13C	2.00	73
					1,2,3,4,7,8-HxCDD-13C	2.00	68
1,2,3,7,8-PeCDD	19.0	-----	1.10		1,2,3,6,7,8-HxCDD-13C	2.00	62
Total PeCDD	190.0	-----	1.10		1,2,3,4,6,7,8-HpCDF-13C	2.00	46
					1,2,3,4,7,8,9-HpCDF-13C	2.00	48
1,2,3,4,7,8-HxCDF	23.0	-----	1.40		1,2,3,4,6,7,8-HpCDD-13C	2.00	56
1,2,3,6,7,8-HxCDF	28.0	-----	0.82		OCDD-13C	4.00	50
2,3,4,6,7,8-HxCDF	21.0	-----	1.10				
1,2,3,7,8,9-HxCDF	-----	5.8	2.30	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	410.0	-----	1.40		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	21.0	-----	1.20		2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	120.0	-----	1.20				
1,2,3,7,8,9-HxCDD	56.0	-----	0.72				
Total HxCDD	1100.0	-----	1.10				
1,2,3,4,6,7,8-HpCDF	1100.0	-----	3.10		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	36.0	-----	3.50		Equivalence: 120 ng/Kg		
Total HpCDF	3700.0	-----	3.30		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	2600.0	-----	2.00				
Total HpCDD	5800.0	-----	2.00				
OCDF	2000.0	-----	2.50				
OCDD	40000.0	-----	2.60	E			

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EMPC = Estimated Maximum Possible Concentration
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J = Estimated value
P = PCDE Interference
E = Exceeds calibration range
I = Interference present

REPORT OF LABORATORY ANALYSIS

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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-013-0.11-0.36		
Lab Sample ID	10365190009		
Filename	Y161012B_14		
Injected By	SMT		
Total Amount Extracted	14.4 g	Matrix	Solid
% Moisture	33.6	Dilution	NA
Dry Weight Extracted	9.56 g	Collected	09/23/2016 11:33
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 22:07

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	1.3	-----	0.48		2,3,7,8-TCDF-13C	2.00	86
Total TCDF	23.0	-----	0.48		2,3,7,8-TCDD-13C	2.00	87
					1,2,3,7,8-PeCDF-13C	2.00	74
2,3,7,8-TCDD	1.4	-----	0.58		2,3,4,7,8-PeCDF-13C	2.00	69
Total TCDD	11.0	-----	0.58		1,2,3,7,8-PeCDD-13C	2.00	69
					1,2,3,4,7,8-HxCDF-13C	2.00	93
1,2,3,7,8-PeCDF	1.4	-----	0.37	J	1,2,3,6,7,8-HxCDF-13C	2.00	80
2,3,4,7,8-PeCDF	5.9	-----	0.13		2,3,4,6,7,8-HxCDF-13C	2.00	85
Total PeCDF	83.0	-----	0.25		1,2,3,7,8,9-HxCDF-13C	2.00	86
					1,2,3,4,7,8-HxCDD-13C	2.00	89
1,2,3,7,8-PeCDD	6.6	-----	0.55		1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	58.0	-----	0.55		1,2,3,4,6,7,8-HpCDF-13C	2.00	61
					1,2,3,4,7,8,9-HpCDF-13C	2.00	70
1,2,3,4,7,8-HxCDF	15.0	-----	0.50		1,2,3,4,6,7,8-HpCDD-13C	2.00	71
1,2,3,6,7,8-HxCDF	17.0	-----	0.36		OCDD-13C	4.00	66
2,3,4,6,7,8-HxCDF	9.5	-----	0.36				
1,2,3,7,8,9-HxCDF	4.0	-----	0.42	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	700.0	-----	0.41		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	5.0	-----	0.58	J	2,3,7,8-TCDD-37Cl4	0.20	87
1,2,3,6,7,8-HxCDD	35.0	-----	0.53				
1,2,3,7,8,9-HxCDD	20.0	-----	0.65				
Total HxCDD	280.0	-----	0.59				
1,2,3,4,6,7,8-HpCDF	1300.0	-----	0.76		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	8.9	-----	1.30		Equivalence: 36 ng/Kg		
Total HpCDF	2400.0	-----	1.00		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	190.0	-----	0.62				
Total HpCDD	390.0	-----	0.62				
OCDF	510.0	-----	1.40				
OCDD	1100.0	-----	0.85				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
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ND = Not Detected
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J = Estimated value

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-002-0.0-0.15		
Lab Sample ID	10365190010		
Filename	Y161012B_15		
Injected By	SMT		
Total Amount Extracted	13.8 g	Matrix	Solid
% Moisture	51.1	Dilution	NA
Dry Weight Extracted	6.75 g	Collected	09/23/2016 11:12
ICAL ID	Y160816A	Received	10/06/2016 20:25
CCal Filename(s)	Y161012B_01 & Y161012B_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 22:50

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.81	-----	0.52	J	2,3,7,8-TCDF-13C	2.00	85
Total TCDF	4.10	-----	0.52		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	80
2,3,7,8-TCDD	ND	-----	0.71		2,3,4,7,8-PeCDF-13C	2.00	72
Total TCDD	14.00	-----	0.71		1,2,3,7,8-PeCDD-13C	2.00	76
					1,2,3,4,7,8-HxCDF-13C	2.00	88
1,2,3,7,8-PeCDF	0.89	-----	0.67	J	1,2,3,6,7,8-HxCDF-13C	2.00	79
2,3,4,7,8-PeCDF	2.10	-----	0.47	J	2,3,4,6,7,8-HxCDF-13C	2.00	82
Total PeCDF	19.00	-----	0.57		1,2,3,7,8,9-HxCDF-13C	2.00	82
					1,2,3,4,7,8-HxCDD-13C	2.00	78
1,2,3,7,8-PeCDD	6.50	-----	0.75	J	1,2,3,6,7,8-HxCDD-13C	2.00	68
Total PeCDD	110.00	-----	0.75		1,2,3,4,6,7,8-HpCDF-13C	2.00	55
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61
1,2,3,4,7,8-HxCDF	5.10	-----	1.70	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	62
1,2,3,6,7,8-HxCDF	7.40	-----	0.60		OCDD-13C	4.00	58
2,3,4,6,7,8-HxCDF	3.80	-----	0.87	J			
1,2,3,7,8,9-HxCDF	2.20	-----	0.80	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	290.00	-----	1.00		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	22.00	-----	1.60		2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	86.00	-----	0.77				
1,2,3,7,8,9-HxCDD	59.00	-----	0.64				
Total HxCDD	860.00	-----	1.00				
1,2,3,4,6,7,8-HpCDF	230.00	-----	1.70		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	2.9	1.40	IJ	Equivalence: 44 ng/Kg		
Total HpCDF	600.00	-----	1.60		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	1500.00	-----	2.20				
Total HpCDD	2700.00	-----	2.20				
OCDF	87.00	-----	2.00				
OCDD	3600.00	-----	2.80				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52316	Matrix	Solid
Filename	U161012A_06	Dilution	NA
Total Amount Extracted	10.2 g	Extracted	10/10/2016 16:10
ICAL ID	U161011	Analyzed	10/12/2016 13:53
CCal Filename(s)	U161012A_01 & U161012A_17	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.046		2,3,7,8-TCDF-13C	2.00	64
Total TCDF	0.054	-----	0.046	J	2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	73
2,3,7,8-TCDD	ND	-----	0.064		2,3,4,7,8-PeCDF-13C	2.00	69
Total TCDD	ND	-----	0.064		1,2,3,7,8-PeCDD-13C	2.00	92
					1,2,3,4,7,8-HxCDF-13C	2.00	69
1,2,3,7,8-PeCDF	ND	-----	0.059		1,2,3,6,7,8-HxCDF-13C	2.00	66
2,3,4,7,8-PeCDF	ND	-----	0.040		2,3,4,6,7,8-HxCDF-13C	2.00	70
Total PeCDF	ND	-----	0.049		1,2,3,7,8,9-HxCDF-13C	2.00	64
					1,2,3,4,7,8-HxCDD-13C	2.00	77
1,2,3,7,8-PeCDD	ND	-----	0.044		1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	ND	-----	0.044		1,2,3,4,6,7,8-HpCDF-13C	2.00	68
					1,2,3,4,7,8,9-HpCDF-13C	2.00	69
1,2,3,4,7,8-HxCDF	ND	-----	0.063		1,2,3,4,6,7,8-HpCDD-13C	2.00	84
1,2,3,6,7,8-HxCDF	ND	-----	0.068		OCDD-13C	4.00	58
2,3,4,6,7,8-HxCDF	ND	-----	0.060				
1,2,3,7,8,9-HxCDF	ND	-----	0.063		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.063		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.074		2,3,7,8-TCDD-37Cl4	0.20	77
1,2,3,6,7,8-HxCDD	ND	-----	0.076				
1,2,3,7,8,9-HxCDD	ND	-----	0.079				
Total HxCDD	0.270	-----	0.076	J			
1,2,3,4,6,7,8-HpCDF	ND	-----	0.140		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.180		Equivalence: 0.00087 ng/Kg		
Total HpCDF	ND	-----	0.160		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	0.087	-----	0.086	J			
Total HpCDD	0.087	-----	0.086	J			
OCDF	ND	-----	0.170				
OCDD	ND	-----	0.290				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

Results reported on a total weight basis and are valid to no more than 2 significant figures.
J = Estimated value

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52317		
Filename	U161012A_04	Matrix	Solid
Total Amount Extracted	10.4 g	Dilution	NA
ICAL ID	U161011	Extracted	10/10/2016 16:10
CCal Filename(s)	U161012A_01 & U161012A_17	Analyzed	10/12/2016 12:20
Method Blank ID	BLANK-52316	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.21	103	2,3,7,8-TCDF-13C	2.0	67
Total TCDF				2,3,7,8-TCDD-13C	2.0	95
				1,2,3,7,8-PeCDF-13C	2.0	79
2,3,7,8-TCDD	0.20	0.16	82	2,3,4,7,8-PeCDF-13C	2.0	73
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	94
				1,2,3,4,7,8-HxCDF-13C	2.0	73
1,2,3,7,8-PeCDF	1.0	1.00	100	1,2,3,6,7,8-HxCDF-13C	2.0	69
2,3,4,7,8-PeCDF	1.0	1.1	108	2,3,4,6,7,8-HxCDF-13C	2.0	71
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	64
				1,2,3,4,7,8-HxCDD-13C	2.0	83
1,2,3,7,8-PeCDD	1.0	0.93	93	1,2,3,6,7,8-HxCDD-13C	2.0	69
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	70
				1,2,3,4,7,8,9-HpCDF-13C	2.0	72
1,2,3,4,7,8-HxCDF	1.0	1.1	112	1,2,3,4,6,7,8-HpCDD-13C	2.0	87
1,2,3,6,7,8-HxCDF	1.0	1.0	102	OCDD-13C	4.0	63
2,3,4,6,7,8-HxCDF	1.0	1.0	102			
1,2,3,7,8,9-HxCDF	1.0	1.0	100	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.0	101	2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	1.0	1.1	112			
1,2,3,7,8,9-HxCDD	1.0	1.1	108			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	106			
1,2,3,4,7,8,9-HpCDF	1.0	0.98	98			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	1.0	101			
Total HpCDD						
OCDF	2.0	2.3	117 Y			
OCDD	2.0	2.2	108			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range

Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
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Method 8290 Spiked Sample Report

Client - Bay West, Inc.

Client's Sample ID	BW16SR-011-0.16-0.41-MS		
Lab Sample ID	10365190006-MS		
Filename	Y161012B_02	Matrix	Solid
Total Amount Extracted	14.1 g	Dilution	NA
ICAL ID	Y160816A	Extracted	10/10/2016 16:10
CCal Filename(s)	Y161012B_01 & Y161012B_17	Analyzed	10/12/2016 13:35
Method Blank ID	BLANK-52316	Injected By	SMT

Native Isomers	Qs (ng)	Qm (ng)	% Rec.	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	0.20	0.31	157	2,3,7,8-TCDF-13C	2.00	76
				2,3,7,8-TCDD-13C	2.00	81
				1,2,3,7,8-PeCDF-13C	2.00	53
2,3,7,8-TCDD	0.20	0.22	108	2,3,4,7,8-PeCDF-13C	2.00	45
				1,2,3,7,8-PeCDD-13C	2.00	51
				1,2,3,4,7,8-HxCDF-13C	2.00	81
1,2,3,7,8-PeCDF	1.00	1.16	116	1,2,3,6,7,8-HxCDF-13C	2.00	72
2,3,4,7,8-PeCDF	1.00	1.22	122	2,3,4,6,7,8-HxCDF-13C	2.00	76
				1,2,3,7,8,9-HxCDF-13C	2.00	74
				1,2,3,4,7,8-HxCDD-13C	2.00	77
1,2,3,7,8-PeCDD	1.00	1.09	109	1,2,3,6,7,8-HxCDD-13C	2.00	68
				1,2,3,4,6,7,8-HpCDF-13C	2.00	50
				1,2,3,4,7,8,9-HpCDF-13C	2.00	52
1,2,3,4,7,8-HxCDF	1.00	1.23	123	1,2,3,4,6,7,8-HpCDD-13C	2.00	58
1,2,3,6,7,8-HxCDF	1.00	1.27	127	OCDD-13C	4.00	47
2,3,4,6,7,8-HxCDF	1.00	1.13	113			
1,2,3,7,8,9-HxCDF	1.00	1.08	108	1,2,3,4-TCDD-13C	2.00	NA
				1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	1.00	1.28	128	2,3,7,8-TCDD-37Cl4	0.20	78
1,2,3,6,7,8-HxCDD	1.00	1.41	141			
1,2,3,7,8,9-HxCDD	1.00	1.31	131			
1,2,3,4,6,7,8-HpCDF	1.00	5.67	567			
1,2,3,4,7,8,9-HpCDF	1.00	1.18	118			
1,2,3,4,6,7,8-HpCDD	1.00	7.54	754			
OCDF	2.00	7.27	363			
OCDD	2.00	96.09	4804 E			

Qs = Quantity Spiked Qm = Quantity Measured Rec. = Recovery (Expressed as Percent)

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
E = Exceeds calibration range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Spiked Sample Report

Client - Bay West, Inc.

Client's Sample ID	BW16SR-011-0.16-0.41-MSD		
Lab Sample ID	10365190006-MSD		
Filename	Y161012B_03	Matrix	Solid
Total Amount Extracted	14.0 g	Dilution	NA
ICAL ID	Y160816A	Extracted	10/10/2016 16:10
CCal Filename(s)	Y161012B_01 & Y161012B_17	Analyzed	10/12/2016 14:17
Method Blank ID	BLANK-52316	Injected By	SMT

Native Isomers	Qs (ng)	Qm (ng)	% Rec.	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	0.20	0.30	150	2,3,7,8-TCDF-13C	2.00	81
				2,3,7,8-TCDD-13C	2.00	89
				1,2,3,7,8-PeCDF-13C	2.00	73
2,3,7,8-TCDD	0.20	0.22	109	2,3,4,7,8-PeCDF-13C	2.00	65
				1,2,3,7,8-PeCDD-13C	2.00	73
				1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	1.00	1.13	113	1,2,3,6,7,8-HxCDF-13C	2.00	75
2,3,4,7,8-PeCDF	1.00	1.21	121	2,3,4,6,7,8-HxCDF-13C	2.00	75
				1,2,3,7,8,9-HxCDF-13C	2.00	77
				1,2,3,4,7,8-HxCDD-13C	2.00	74
1,2,3,7,8-PeCDD	1.00	1.04	104	1,2,3,6,7,8-HxCDD-13C	2.00	68
				1,2,3,4,6,7,8-HpCDF-13C	2.00	59
				1,2,3,4,7,8,9-HpCDF-13C	2.00	64
1,2,3,4,7,8-HxCDF	1.00	1.18	118	1,2,3,4,6,7,8-HpCDD-13C	2.00	73
1,2,3,6,7,8-HxCDF	1.00	1.19	119	OCDD-13C	4.00	67
2,3,4,6,7,8-HxCDF	1.00	1.11	111			
1,2,3,7,8,9-HxCDF	1.00	1.08	108	1,2,3,4-TCDD-13C	2.00	NA
				1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	1.00	1.23	123	2,3,7,8-TCDD-37Cl4	0.20	83
1,2,3,6,7,8-HxCDD	1.00	1.50	150			
1,2,3,7,8,9-HxCDD	1.00	1.32	132			
1,2,3,4,6,7,8-HpCDF	1.00	5.09	509			
1,2,3,4,7,8,9-HpCDF	1.00	1.12	112			
1,2,3,4,6,7,8-HpCDD	1.00	11.21	1121			
OCDF	2.00	10.87	544			
OCDD	2.00	148.85	7443 E			

Qs = Quantity Spiked Qm = Quantity Measured Rec. = Recovery (Expressed as Percent)

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
E = Exceeds calibration range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Spike Sample Results

Client - Bay West, Inc.

Client Sample ID	BW16SR-011-0.16-0.41			Dry Weights	
Lab Sample ID	10365190006	Sample Filename	Y161012B_11	Sample Amount	8.54 g
MS ID	10365190006-MS	MS Filename	Y161012B_02	MS Amount	8.6 g
MSD ID	10365190006-MSD	MSD Filename	Y161012B_03	MSD Amount	8.5 g

Analyte	Sample Conc. ng/Kg	MS/MSD Qs (ng)	MS Qm (ng)	MSD Qm (ng)	RPD	Background Subtracted MS % Rec.	MSD % Rec.	RPD
2,3,7,8-TCDF	7.617	0.20	0.31	0.30	4.5	124	118	5.5
2,3,7,8-TCDD	2.523	0.20	0.22	0.22	0.5	97	98	0.7
1,2,3,7,8-PeCDF	1.560	1.00	1.16	1.13	2.4	115	112	2.4
2,3,4,7,8-PeCDF	3.339	1.00	1.22	1.21	1.0	120	118	1.0
1,2,3,7,8-PeCDD	5.348	1.00	1.09	1.04	4.6	104	99	4.8
1,2,3,4,7,8-HxCDF	6.526	1.00	1.23	1.18	3.9	117	113	4.1
1,2,3,6,7,8-HxCDF	14.048	1.00	1.27	1.19	6.5	114	107	7.1
2,3,4,6,7,8-HxCDF	6.148	1.00	1.13	1.11	1.8	108	106	1.9
1,2,3,7,8,9-HxCDF	2.483	1.00	1.08	1.08	0.5	106	106	0.5
1,2,3,4,7,8-HxCDD	4.149	1.00	1.28	1.23	3.7	124	120	3.8
1,2,3,6,7,8-HxCDD	30.738	1.00	1.41	1.50	5.7	115	124	7.2
1,2,3,7,8,9-HxCDD	13.833	1.00	1.31	1.32	1.0	119	120	1.2
1,2,3,4,6,7,8-HpCDF	386.320	1.00	5.67	5.09	10.8	235	179	26.9
1,2,3,4,7,8,9-HpCDF	0.000	1.00	1.18	1.12	5.0	111	105	5.3
1,2,3,4,6,7,8-HpCDD	684.485	1.00	7.54	11.21	39.2	165	536	105.8
OCDF	415.964	2.00	7.27	10.87	39.7	185	366	65.9
OCDD	10982.005	2.00	96.09	148.85	43.1	81	2753	188.5

Definitions

MS = Matrix Spike
MSD = Matrix Spike Duplicate
Qm = Quantity Measured
Qs = Quantity Spiked
% Rec. = Percent Recovery
RPD = Relative Percent Difference
NA = Not Applicable
NC = Not Calculated

CDD = Chlorinated dibenzo-p-dioxin
CDF = Chlorinated dibenzo-p-furan
T = Tetra
Pe = Penta
Hx = Hexa
Hp = Hepta
O = Octa

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Report Prepared for:

Nancy McDonald
Bay West, Inc.
5 Empire Drive
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REPORT OF LABORATORY ANALYSIS FOR PCDD/PCDF

Report Prepared Date:
October 18, 2016

Report Information:

Pace Project #: 10365198
Sample Receipt Date: 10/06/2016
Client Project #: J160139 SLR Sediment AOCs
Client Sub PO #: 108002
State Cert #: 027-053-137

Invoicing & Reporting Options:

The report provided has been invoiced as a Level 2 PCDD/PCDF Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

This report has been reviewed by:

October 18, 2016

Carolynne Trout, Project Manager
(612) 607-6351
(612) 607-6444 (fax)
Carolynne.Trout@pacelabs.com

Report of Laboratory Analysis

Pace Analytical Services, Inc.
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DISCUSSION

This report presents the results from the analyses performed on two samples submitted by a representative of Bay West LLC. The samples were analyzed for the presence or absence of polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs) using a modified version of USEPA Method 8290. The reporting limits were based on signal-to-noise measurements. Estimated Maximum Possible Concentration (EMPC) values were treated as positives in the toxic equivalence calculations.

Second column confirmation analyses of 2,3,7,8-TCDF values obtained from the primary (DB5-MS) column are performed only when specifically requested for a project and only when the values are above the concentration of the lowest calibration standard. Typical resolution for this isomer using the DB5-MS column ranges from 25-30%.

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extracts ranged from 46-100%. All of the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. Also, since the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained. Concentrations below the calibration range were flagged "J" and should be regarded as estimates.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to contain trace levels of selected congeners. These levels were below the calibration range of the method. The levels reported for the affected congeners in the field samples were higher than the corresponding blank levels by one or more orders of magnitude. These results indicate that the sample processing steps did not contribute significantly to the levels reported for the field samples.

A laboratory spike sample was also prepared with the sample batch using clean reference matrix that had been fortified with native standard materials. The results show that the spiked native compounds were recovered at 82-117%. These results were within the target range for the method. Matrix spikes were prepared with the sample batch using sample material from a separate project; results from these analyses will be provided upon request.

The response obtained for the native OCDF in calibration standard analyses U161012A_17 was outside the target range. As specified in our procedures, the average of the daily response factors for this compound was used in the calculations for the samples from this runshift. The affected values were flagged "Y" on the results tables.

REPORT OF LABORATORY ANALYSIS

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Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Montana	92
Alaska	MN00064	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN_00064_200
Arkansas	88-0680	New Jersey (NE	MN002
California	01155CA	New York (NEL	11647
Colorado	MN00064	North Carolina	27700
Connecticut	PH-0256	North Dakota	R-036
EPA Region 8	8TMS-Q	Ohio	4150
Florida (NELAP	E87605	Oklahoma	D9922
Georgia (DNR)	959	Oregon (ELAP)	MN200001-005
Guam	959	Oregon (OREL	MN300001-001
Hawaii	SLD	Pennsylvania	68-00563
Idaho	MN00064	Puerto Rico	MN00064
Illinois	200012	Saipan	MP0003
Indiana	C-MN-01	South Carolina	74003001
Indiana	C-MN-01	Tennessee	TN02818
Iowa	368	Texas	T104704192-08
Kansas	E-10167	Utah (NELAP)	MN00064
Kentucky	90062	Virginia	00251
Louisiana	03086	Washington	C755
Maine	2007029	West Virginia #	9952C
Maryland	322	West Virginia D	382
Michigan	9909	Wisconsin	999407970
Minnesota	027-053-137	Wyoming	8TMS-Q

REPORT OF LABORATORY ANALYSIS

Appendix A

Sample Management

Report No....10365198_8290

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365198

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-3
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0.15	SO	G	3/12/15	1204													
1																			
2	BW16SR-005	BW16SR-005-0.0-0.15	SO	G	9/30/16	1120	3	3								1	1	1	a1
3	BW16SR-005	BW16SR-115-0.0-0.15	SO	G	9/30/16	1220	2	2								1	1		a2 Collect % moisture from 4 oz jar
4																			
5																			
6																			
7																			
8																			
9																			
10																			
11																			
12																			

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16	[signature]	10/6/16	1444	Kristina Polson	10/6/16	1445	4.9	Y	N	Y
	Kristina Polson	10/6/16	1715	[signature]	10/6/16	1715				
	[signature]	10/6/16	2025	Client Pace	10.6.16	20:25				

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: CJM DATE Signed (MM/DD/YY): 10/6/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: WO# : 10365198

10365198

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ SpeeDee ☐ Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☐ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ 888A0143310098 **Type of Ice:** ☐ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.9, 0.6, 4.7 **Cooler Temp Corrected (°C):** 0.2, 0.8, 4.0 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** CAT 10.6.16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☒ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☒ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☑ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☑ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	☑ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☑ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☑ N/A	Sample #
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☑ N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: Carolyne Trout **Date:** 10/10/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Reporting Flags

A = Reporting Limit based on signal to noise

B = Less than 10x higher than method blank level

C = Result obtained from confirmation analysis

D = Result obtained from analysis of diluted sample

E = Exceeds calibration range

I = Interference present

J = Estimated value

Nn = Value obtained from additional analysis

P = PCDE Interference

R = Recovery outside target range

S = Peak saturated

U = Analyte not detected

V = Result verified by confirmation analysis

X = %D Exceeds limits

Y = Calculated using average of daily RFs

* = See Discussion

REPORT OF LABORATORY ANALYSIS

Appendix B

Sample Analysis Summary

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-005-0.0-0.15		
Lab Sample ID	10365198001		
Filename	U161012A_14		
Injected By	SMT		
Total Amount Extracted	15.6 g	Matrix	Solid
% Moisture	52.5	Dilution	NA
Dry Weight Extracted	7.41 g	Collected	09/30/2016 11:20
ICAL ID	U161011	Received	10/06/2016 20:25
CCal Filename(s)	U161012A_01 & U161012A_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 20:07

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	4.1	-----	0.046		2,3,7,8-TCDF-13C	2.00	70
Total TCDF	23.0	-----	0.046		2,3,7,8-TCDD-13C	2.00	95
					1,2,3,7,8-PeCDF-13C	2.00	84
2,3,7,8-TCDD	2.0	-----	0.077		2,3,4,7,8-PeCDF-13C	2.00	75
Total TCDD	19.0	-----	0.077		1,2,3,7,8-PeCDD-13C	2.00	100
					1,2,3,4,7,8-HxCDF-13C	2.00	58
1,2,3,7,8-PeCDF	1.7	-----	0.130	J	1,2,3,6,7,8-HxCDF-13C	2.00	46
2,3,4,7,8-PeCDF	3.7	-----	0.060	J	2,3,4,6,7,8-HxCDF-13C	2.00	72
Total PeCDF	56.0	-----	0.093		1,2,3,7,8,9-HxCDF-13C	2.00	63
					1,2,3,4,7,8-HxCDD-13C	2.00	81
1,2,3,7,8-PeCDD	5.9	-----	0.120	J	1,2,3,6,7,8-HxCDD-13C	2.00	64
Total PeCDD	60.0	-----	0.120		1,2,3,4,6,7,8-HpCDF-13C	2.00	61
					1,2,3,4,7,8,9-HpCDF-13C	2.00	68
1,2,3,4,7,8-HxCDF	15.0	-----	0.100		1,2,3,4,6,7,8-HpCDD-13C	2.00	86
1,2,3,6,7,8-HxCDF	24.0	-----	0.083		OCDD-13C	4.00	72
2,3,4,6,7,8-HxCDF	7.9	-----	0.130				
1,2,3,7,8,9-HxCDF	3.4	-----	0.130	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	780.0	-----	0.110		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	4.4	-----	0.260	J	2,3,7,8-TCDD-37Cl4	0.20	85
1,2,3,6,7,8-HxCDD	33.0	-----	0.300				
1,2,3,7,8,9-HxCDD	18.0	-----	0.045				
Total HxCDD	270.0	-----	0.200				
1,2,3,4,6,7,8-HpCDF	1200.0	-----	0.140		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	8.9	-----	0.240		Equivalence: 39 ng/Kg		
Total HpCDF	2300.0	-----	0.190		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	460.0	-----	0.910				
Total HpCDD	1000.0	-----	0.910				
OCDF	560.0	-----	0.650	Y			
OCDD	5200.0	-----	0.570				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-115-0.0-0.15		
Lab Sample ID	10365198002		
Filename	U161012A_15		
Injected By	SMT		
Total Amount Extracted	16.1 g	Matrix	Solid
% Moisture	52.1	Dilution	NA
Dry Weight Extracted	7.71 g	Collected	09/30/2016 12:20
ICAL ID	U161011	Received	10/06/2016 20:25
CCal Filename(s)	U161012A_01 & U161012A_17	Extracted	10/10/2016 16:10
Method Blank ID	BLANK-52316	Analyzed	10/12/2016 20:54

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	4.2	-----	0.130		2,3,7,8-TCDF-13C	2.00	71
Total TCDF	21.0	-----	0.130		2,3,7,8-TCDD-13C	2.00	94
					1,2,3,7,8-PeCDF-13C	2.00	83
2,3,7,8-TCDD	2.1	-----	0.067		2,3,4,7,8-PeCDF-13C	2.00	76
Total TCDD	21.0	-----	0.067		1,2,3,7,8-PeCDD-13C	2.00	100
					1,2,3,4,7,8-HxCDF-13C	2.00	73
1,2,3,7,8-PeCDF	1.7	-----	0.190	J	1,2,3,6,7,8-HxCDF-13C	2.00	67
2,3,4,7,8-PeCDF	3.4	-----	0.093	J	2,3,4,6,7,8-HxCDF-13C	2.00	72
Total PeCDF	54.0	-----	0.140		1,2,3,7,8,9-HxCDF-13C	2.00	64
					1,2,3,4,7,8-HxCDD-13C	2.00	84
1,2,3,7,8-PeCDD	5.3	-----	0.150	J	1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	56.0	-----	0.150		1,2,3,4,6,7,8-HpCDF-13C	2.00	62
					1,2,3,4,7,8,9-HpCDF-13C	2.00	67
1,2,3,4,7,8-HxCDF	12.0	-----	0.240		1,2,3,4,6,7,8-HpCDD-13C	2.00	86
1,2,3,6,7,8-HxCDF	21.0	-----	0.140		OCDD-13C	4.00	67
2,3,4,6,7,8-HxCDF	6.6	-----	0.120				
1,2,3,7,8,9-HxCDF	2.5	-----	0.140	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	580.0	-----	0.160		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	4.5	-----	1.600	J	2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	30.0	-----	0.550				
1,2,3,7,8,9-HxCDD	17.0	-----	1.300				
Total HxCDD	270.0	-----	1.200				
1,2,3,4,6,7,8-HpCDF	940.0	-----	1.700		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	7.5	-----	0.540		Equivalence: 32 ng/Kg		
Total HpCDF	1800.0	-----	1.100		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	350.0	-----	0.350				
Total HpCDD	710.0	-----	0.350				
OCDF	390.0	-----	0.130	Y			
OCDD	3100.0	-----	0.190				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52316	Matrix	Solid
Filename	U161012A_06	Dilution	NA
Total Amount Extracted	10.2 g	Extracted	10/10/2016 16:10
ICAL ID	U161011	Analyzed	10/12/2016 13:53
CCal Filename(s)	U161012A_01 & U161012A_17	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.046		2,3,7,8-TCDF-13C	2.00	64
Total TCDF	0.054	-----	0.046	J	2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	73
2,3,7,8-TCDD	ND	-----	0.064		2,3,4,7,8-PeCDF-13C	2.00	69
Total TCDD	ND	-----	0.064		1,2,3,7,8-PeCDD-13C	2.00	92
					1,2,3,4,7,8-HxCDF-13C	2.00	69
1,2,3,7,8-PeCDF	ND	-----	0.059		1,2,3,6,7,8-HxCDF-13C	2.00	66
2,3,4,7,8-PeCDF	ND	-----	0.040		2,3,4,6,7,8-HxCDF-13C	2.00	70
Total PeCDF	ND	-----	0.049		1,2,3,7,8,9-HxCDF-13C	2.00	64
					1,2,3,4,7,8-HxCDD-13C	2.00	77
1,2,3,7,8-PeCDD	ND	-----	0.044		1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	ND	-----	0.044		1,2,3,4,6,7,8-HpCDF-13C	2.00	68
					1,2,3,4,7,8,9-HpCDF-13C	2.00	69
1,2,3,4,7,8-HxCDF	ND	-----	0.063		1,2,3,4,6,7,8-HpCDD-13C	2.00	84
1,2,3,6,7,8-HxCDF	ND	-----	0.068		OCDD-13C	4.00	58
2,3,4,6,7,8-HxCDF	ND	-----	0.060				
1,2,3,7,8,9-HxCDF	ND	-----	0.063		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.063		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.074		2,3,7,8-TCDD-37Cl4	0.20	77
1,2,3,6,7,8-HxCDD	ND	-----	0.076				
1,2,3,7,8,9-HxCDD	ND	-----	0.079				
Total HxCDD	0.270	-----	0.076	J			
1,2,3,4,6,7,8-HpCDF	ND	-----	0.140		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.180		Equivalence: 0.00087 ng/Kg		
Total HpCDF	ND	-----	0.160		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	0.087	-----	0.086	J			
Total HpCDD	0.087	-----	0.086	J			
OCDF	ND	-----	0.170				
OCDD	ND	-----	0.290				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

Results reported on a total weight basis and are valid to no more than 2 significant figures.
J = Estimated value

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52317		
Filename	U161012A_04	Matrix	Solid
Total Amount Extracted	10.4 g	Dilution	NA
ICAL ID	U161011	Extracted	10/10/2016 16:10
CCal Filename(s)	U161012A_01 & U161012A_17	Analyzed	10/12/2016 12:20
Method Blank ID	BLANK-52316	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.21	103	2,3,7,8-TCDF-13C	2.0	67
Total TCDF				2,3,7,8-TCDD-13C	2.0	95
				1,2,3,7,8-PeCDF-13C	2.0	79
2,3,7,8-TCDD	0.20	0.16	82	2,3,4,7,8-PeCDF-13C	2.0	73
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	94
				1,2,3,4,7,8-HxCDF-13C	2.0	73
1,2,3,7,8-PeCDF	1.0	1.00	100	1,2,3,6,7,8-HxCDF-13C	2.0	69
2,3,4,7,8-PeCDF	1.0	1.1	108	2,3,4,6,7,8-HxCDF-13C	2.0	71
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	64
				1,2,3,4,7,8-HxCDD-13C	2.0	83
1,2,3,7,8-PeCDD	1.0	0.93	93	1,2,3,6,7,8-HxCDD-13C	2.0	69
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	70
				1,2,3,4,7,8,9-HpCDF-13C	2.0	72
1,2,3,4,7,8-HxCDF	1.0	1.1	112	1,2,3,4,6,7,8-HpCDD-13C	2.0	87
1,2,3,6,7,8-HxCDF	1.0	1.0	102	OCDD-13C	4.0	63
2,3,4,6,7,8-HxCDF	1.0	1.0	102			
1,2,3,7,8,9-HxCDF	1.0	1.0	100	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.0	101	2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	1.0	1.1	112			
1,2,3,7,8,9-HxCDD	1.0	1.1	108			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	106			
1,2,3,4,7,8,9-HpCDF	1.0	0.98	98			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	1.0	101			
Total HpCDD						
OCDF	2.0	2.3	117 Y			
OCDD	2.0	2.2	108			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range

Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365862

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/27/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☐	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☒	☐	☐	The response obtained for the native OCDF in the calibration standard analyses U161019C_19 was outside the target range. As specified in the Pace procedures, the

					average of the daily response factors for this compound was used in the calculations for the samples from this analytical run. The affected values were flagged "Y" on the results tables. No data were qualified.

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	Low-level concentrations of 2,3,4,7,8-PeCDF 1,2,3,4, 7,8-HxCDD, 2,3,4,6,7,8-HxCDF, Total HxCDF, 1,2,3,4,6,7,8-HpCDF Total HpCDD, and OCDD were detected in the method blank 52398.
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☒	☐	All sample results were > 10x the blank concentrations except for the 2,3,4,7,8-PeCDF result in sample BW16SR-009-0.0-0.15.The 2,3,4,7,8-PeCDF result in sample BW16SR-009-0.0-0.15 was qualified "U".

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☒	☐	☐	Dioxins/furans have internal standards instead of surrogates.
b.	Are the lab recovery limits specified on the report?		☒	☐	☐	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☒	☐	☐	
	i.	If yes, are the surrogates above the lab limits?	☐	☒	☐	
	ii.	Below the lab limits?	☒	☐	☐	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	Except for fifteen low values, which were flagged "R" on the sample results, the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. No qualifiers were applied because the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, so data were automatically corrected for variation in recovery and accurate values were obtained. No data were qualified.

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	
	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question				Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.			☐	☒	☐	
	i.	Have the required matrix spikes been prepared and reported?		☐	☐	☒	
	ii.	If no, is there and explanation in the report as to why?		☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?		☐	☐	☒	
	iv.	Are the lab limits specified on the report?		☐	☐	☒	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?		☐	☐	☒	
	vi.	Are there compounds outside the lab limits?		☐	☐	☒	
		1.	If yes, are the analytes above the lab limits?	☐	☐	☒	
		2.	Below the lab limits?	☐	☐	☒	
		3.	Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.			☐	☐	☒	
	i.	Is the RPD for the duplicate pair within the lab limits?		☐	☐	☒	
	ii.	If no, has the associated source sample been flagged?		☐	☐	☒	
c.	What is the impact of failed QC on this project?			☐	☐	☒	

7. Method Detection Limits/Report Limits

Question	Yes	No	N/A	Comments

a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Samples BW16SR-005-0.0-0.15 BW16SR-115-0.0-0.15 were collected as field duplicates. All RPDs were within the QC guideline of ≤ 50% except for the following. The RPD for OCDD (50.6%) was high and exceeded the QC guideline of ≤ 50%. Results for OCDD were qualified “J” as estimated in samples BW16SR-005-0.0-0.15 BW16SR-115-0.0-0.15.

(2) Interfering substances impacted the determinations of PCDD or PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where polychlorinated diphenyl ethers were present. All results flagged “I” or “J” were qualified “J” as estimated. concentrations below the calibration range were flagged “J” as estimated by the laboratory

(3) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

www.pacelabs.com

Report Prepared for:

Nancy McDonald
Bay West, Inc.

Saint Paul MN 55103

REPORT OF LABORATORY ANALYSIS FOR PCDD/PCDF

Report Prepared Date:
October 27, 2016

Report Information:

Pace Project #: 10365862
Sample Receipt Date: 10/12/2016
Client Project #: J160139 SLR Sediment AOCs
Client Sub PO #: 108002
State Cert #: 027-053-137

Invoicing & Reporting Options:

The report provided has been invoiced as a Level 2 PCDD/PCDF Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

This report has been reviewed by:

October 27, 2016

Carolynne Trout, Project Manager
(612) 607-6351
(612) 607-6444 (fax)
Carolynne.Trout@pacelabs.com

Report of Laboratory Analysis

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

DISCUSSION

This report presents the results from the analyses performed on eleven samples submitted by a representative of Bay West LLC. The samples were analyzed for the presence or absence of polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs) using a modified version of USEPA Method 8290. The reporting limits were based on signal-to-noise measurements. Estimated Maximum Possible Concentration (EMPC) values were treated as positives in the toxic equivalence calculations.

Second column confirmation analyses of 2,3,7,8-TCDF values obtained from the primary (DB5-MS) column are performed only when specifically requested for a project and only when the values are above the concentration of the lowest calibration standard. Typical resolution for this isomer using the DB5-MS column ranges from 25-30%.

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extracts ranged from 52-96%. Except for fifteen low values, which were flagged "R" on the results table, the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. Also, since the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained.

In some cases, interfering substances impacted the determinations of PCDD or PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where polychlorinated diphenyl ethers were present. Concentrations below the calibration range were flagged "J" and should be regarded as estimates.

A laboratory method blank was prepared and analyzed with each sample batch as part of our routine quality control procedures. The results show Blank-52398 to contain trace levels of selected congeners. These levels were below the calibration range of the method. The levels reported for the affected congeners in the field samples were higher than the corresponding blank levels by one or more orders of magnitude. These results indicate that the sample processing steps did not contribute significantly to the levels reported for the field samples.

A laboratory spike sample was also prepared with each sample batch using clean reference matrix that had been fortified with native standard materials. The results show that the spiked native compounds were recovered at 87-130%. These results were within the target range for the method. Matrix spikes were prepared with the sample batches using sample material from separate projects; results from these analyses will be provided upon request.

The response obtained for the labeled OCDD in calibration standard analyses Y161019C_19 was outside the target range. As specified in our procedures, the average of the daily response factors for this compound was used in the calculations for the samples from this runshift. The affected values were flagged "Y" on the results tables.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Montana	92
Alaska	MN00064	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN_00064_200
Arkansas	88-0680	New Jersey (NE	MN002
California	01155CA	New York (NEL	11647
Colorado	MN00064	North Carolina	27700
Connecticut	PH-0256	North Dakota	R-036
EPA Region 8	8TMS-Q	Ohio	4150
Florida (NELAP	E87605	Oklahoma	D9922
Georgia (DNR)	959	Oregon (ELAP)	MN200001-005
Guam	959	Oregon (OREL	MN300001-001
Hawaii	SLD	Pennsylvania	68-00563
Idaho	MN00064	Puerto Rico	MN00064
Illinois	200012	Saipan	MP0003
Indiana	C-MN-01	South Carolina	74003001
Indiana	C-MN-01	Tennessee	TN02818
Iowa	368	Texas	T104704192-08
Kansas	E-10167	Utah (NELAP)	MN00064
Kentucky	90062	Virginia	00251
Louisiana	03086	Washington	C755
Maine	2007029	West Virginia #	9952C
Maryland	322	West Virginia D	382
Michigan	9909	Wisconsin	999407970
Minnesota	027-053-137	Wyoming	8TMS-Q

REPORT OF LABORATORY ANALYSIS

Appendix A

Sample Management

Report No....10365862_8290

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365862

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-4
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0.15	SO	G	3/12/15	1204													
1	BW16SR-006	BW16SR-006-0.0-0.15	SO	G	10/11/16	1435	3	3								1	1	1	001
2	BW16SR-007	BW16SR-007-0.0-0.15	SO	G	10/11/16	1440	3	3								1	1	1	002
3	BW16SR-008	BW16SR-008-0.0-0.15	SO	G	10/11/16	1450	3	3								1	1	1	003
4	BW16SR-009	BW16SR-009-0.0-0.15	SO	G	10/11/16	1500	3	3								1	1	1	004
5	BW16SR-011	BW16SR-011-0.0-0.15	SO	G	10/11/16	1505	3	3								1	1	1	005
6	BW16SR-011	BW16SR-111-0.0-0.15	SO	G	10/11/16	1510	3	3								1	1	1	006
7	BW16SR-013	BW16SR-013-0.0-0.15	SO	G	10/11/16	1515	3	3								1	1	1	007
8	BW16SR-014	BW16SR-014-0.0-0.15	SO	G	10/11/16	1530	3	3								1	1	1	S. Dam Wall 008
9	BW16SR-015	BW16SR-015-0.0-0.15	SO	G	10/11/16	1540	3	3								1	1	1	Channel 009
10	BW16SR-001	BW16SR-001-0.0-0.15	SO	G	9/21/16	1347	2	2								1	1	0	Take moisture from dioxins/furans jar 010
11	BW16SR-003	BW16SR-003-0.0-0.15	SO	G	9/22/16	1234	2	2								1	1	0	Take moisture from dioxins/furans jar 011
12																			

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16	Chris Musson / Bay West	10/12/16	1445	Lauren Hood / Pace	10/12/16	14:45
	Lauren Hood	10/12/16	1600		10/12/16	1600
		10/12/16	1830	Trini / PACE	10/12/16	18:30

SAMPLE CONDITIONS			
Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
1.9	Y	N	Y
3.3	Y		

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: Chris Musson DATE Signed (MM/DD/YY): 10/12/16

Page 5 of 23

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project # WO#: 10365862

10365862

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ SpeeDee ☐ Other:_____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☑ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☑ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 3.3, 1.9 **Cooler Temp Corrected (°C):** 3.5, 2.1 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** BL 10/12/16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☑ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☑ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☑ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☐ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL	☑ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☑ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☑ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☑ N/A	Initial when completed:_____ Lot # of added preservative:_____
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):_____				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: Carolynne Trout **Date:** 10/13/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Reporting Flags

A = Reporting Limit based on signal to noise

B = Less than 10x higher than method blank level

C = Result obtained from confirmation analysis

D = Result obtained from analysis of diluted sample

E = Exceeds calibration range

I = Interference present

J = Estimated value

Nn = Value obtained from additional analysis

P = PCDE Interference

R = Recovery outside target range

S = Peak saturated

U = Analyte not detected

V = Result verified by confirmation analysis

X = %D Exceeds limits

Y = Calculated using average of daily RFs

* = See Discussion

Appendix B

Sample Analysis Summary

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-006-0.0-0.15		
Lab Sample ID	10365862001		
Filename	Y161019C_07		
Injected By	SMT		
Total Amount Extracted	17.0 g	Matrix	Solid
% Moisture	60.0	Dilution	NA
Dry Weight Extracted	6.80 g	Collected	10/11/2016 14:35
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 00:52

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**	
2,3,7,8-TCDF	1.9	-----	0.55		2,3,7,8-TCDF-13C	2.00	80	
Total TCDF	7.0	-----	0.55		2,3,7,8-TCDD-13C	2.00	82	
					1,2,3,7,8-PeCDF-13C	2.00	83	
2,3,7,8-TCDD	ND	-----	0.56		2,3,4,7,8-PeCDF-13C	2.00	84	
Total TCDD	6.6	-----	0.56		1,2,3,7,8-PeCDD-13C	2.00	87	
					1,2,3,4,7,8-HxCDF-13C	2.00	75	
1,2,3,7,8-PeCDF	-----	0.73	0.44	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	73	
2,3,4,7,8-PeCDF	-----	0.82	0.34	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	74	
Total PeCDF	12.0	-----	0.39		1,2,3,7,8,9-HxCDF-13C	2.00	72	
					1,2,3,4,7,8-HxCDD-13C	2.00	66	
1,2,3,7,8-PeCDD	2.4	-----	0.57	J	1,2,3,6,7,8-HxCDD-13C	2.00	67	
Total PeCDD	15.0	-----	0.57		1,2,3,4,6,7,8-HpCDF-13C	2.00	60	
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61	
1,2,3,4,7,8-HxCDF	3.7	-----	0.32	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	62	
1,2,3,6,7,8-HxCDF	5.9	-----	0.32	J	OCDD-13C	4.00	72	Y
2,3,4,6,7,8-HxCDF	2.7	-----	1.00	J				
1,2,3,7,8,9-HxCDF	1.2	-----	0.62	J	1,2,3,4-TCDD-13C	2.00	NA	
Total HxCDF	180.0	-----	0.57		1,2,3,7,8,9-HxCDD-13C	2.00	NA	
1,2,3,4,7,8-HxCDD	ND	-----	1.40		2,3,7,8-TCDD-37Cl4	0.20	82	
1,2,3,6,7,8-HxCDD	7.4	-----	1.50					
1,2,3,7,8,9-HxCDD	3.5	-----	1.20	J				
Total HxCDD	70.0	-----	1.30					
1,2,3,4,6,7,8-HpCDF	320.0	-----	0.43		Total 2,3,7,8-TCDD			
1,2,3,4,7,8,9-HpCDF	1.9	-----	0.92	J	Equivalence: 10 ng/Kg			
Total HpCDF	620.0	-----	0.67		(Using 2005 WHO Factors)			
1,2,3,4,6,7,8-HpCDD	110.0	-----	0.74					
Total HpCDD	250.0	-----	0.74					
OCDF	160.0	-----	1.30					
OCDD	1300.0	-----	1.30					

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-007-0.0-0.15		
Lab Sample ID	10365862002		
Filename	Y161019C_08		
Injected By	SMT		
Total Amount Extracted	13.2 g	Matrix	Solid
% Moisture	39.0	Dilution	NA
Dry Weight Extracted	8.05 g	Collected	10/11/2016 14:40
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 01:34

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery	
2,3,7,8-TCDF	1.10	-----	0.23	J	2,3,7,8-TCDF-13C	2.00	82	
Total TCDF	3.40	-----	0.23		2,3,7,8-TCDD-13C	2.00	88	
					1,2,3,7,8-PeCDF-13C	2.00	88	
2,3,7,8-TCDD	0.33	-----	0.27	J	2,3,4,7,8-PeCDF-13C	2.00	86	
Total TCDD	2.40	-----	0.27		1,2,3,7,8-PeCDD-13C	2.00	91	
					1,2,3,4,7,8-HxCDF-13C	2.00	82	
1,2,3,7,8-PeCDF	-----	0.27	0.11	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	74	
2,3,4,7,8-PeCDF	-----	0.52	0.12	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	78	
Total PeCDF	3.40	-----	0.11	J	1,2,3,7,8,9-HxCDF-13C	2.00	72	
					1,2,3,4,7,8-HxCDD-13C	2.00	78	
1,2,3,7,8-PeCDD	0.39	-----	0.22	J	1,2,3,6,7,8-HxCDD-13C	2.00	66	
Total PeCDD	3.00	-----	0.22	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	57	
					1,2,3,4,7,8,9-HpCDF-13C	2.00	63	
1,2,3,4,7,8-HxCDF	1.90	-----	0.35	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66	
1,2,3,6,7,8-HxCDF	3.00	-----	0.68	J	OCDD-13C	4.00	74	Y
2,3,4,6,7,8-HxCDF	1.10	-----	0.60	J				
1,2,3,7,8,9-HxCDF	0.64	-----	0.22	J	1,2,3,4-TCDD-13C	2.00	NA	
Total HxCDF	82.00	-----	0.46		1,2,3,7,8,9-HxCDD-13C	2.00	NA	
1,2,3,4,7,8-HxCDD	ND	-----	0.51		2,3,7,8-TCDD-37Cl4	0.20	83	
1,2,3,6,7,8-HxCDD	4.00	-----	0.40	J				
1,2,3,7,8,9-HxCDD	1.60	-----	0.51	J				
Total HxCDD	33.00	-----	0.47					
1,2,3,4,6,7,8-HpCDF	130.00	-----	0.44		Total 2,3,7,8-TCDD			
1,2,3,4,7,8,9-HpCDF	-----	1.40	0.86	IJ	Equivalence: 5.0 ng/Kg			
Total HpCDF	260.00	-----	0.65		(Using 2005 WHO Factors)			
1,2,3,4,6,7,8-HpCDD	120.00	-----	0.73					
Total HpCDD	250.00	-----	0.73					
OCDF	80.00	-----	0.59					
OCDD	1000.00	-----	0.73					

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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EDL = Estimated Detection Limit

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NC = Not Calculated

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I = Interference present
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REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-008-0.0-0.15		
Lab Sample ID	10365862003		
Filename	Y161019C_09		
Injected By	SMT		
Total Amount Extracted	15.0 g	Matrix	Solid
% Moisture	53.1	Dilution	NA
Dry Weight Extracted	7.04 g	Collected	10/11/2016 14:50
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 02:16

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**	
2,3,7,8-TCDF	2.40	-----	0.24		2,3,7,8-TCDF-13C	2.00	77	
Total TCDF	9.60	-----	0.24		2,3,7,8-TCDD-13C	2.00	80	
					1,2,3,7,8-PeCDF-13C	2.00	80	
2,3,7,8-TCDD	0.77	-----	0.33	J	2,3,4,7,8-PeCDF-13C	2.00	78	
Total TCDD	6.50	-----	0.33		1,2,3,7,8-PeCDD-13C	2.00	85	
					1,2,3,4,7,8-HxCDF-13C	2.00	74	
1,2,3,7,8-PeCDF	0.77	-----	0.35	J	1,2,3,6,7,8-HxCDF-13C	2.00	70	
2,3,4,7,8-PeCDF	0.94	-----	0.23	J	2,3,4,6,7,8-HxCDF-13C	2.00	72	
Total PeCDF	11.00	-----	0.29		1,2,3,7,8,9-HxCDF-13C	2.00	68	
					1,2,3,4,7,8-HxCDD-13C	2.00	69	
1,2,3,7,8-PeCDD	1.50	-----	0.38	J	1,2,3,6,7,8-HxCDD-13C	2.00	62	
Total PeCDD	13.00	-----	0.38		1,2,3,4,6,7,8-HpCDF-13C	2.00	53	
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59	
1,2,3,4,7,8-HxCDF	2.50	-----	0.71	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	58	
1,2,3,6,7,8-HxCDF	4.80	-----	0.94	J	OCDD-13C	4.00	70	Y
2,3,4,6,7,8-HxCDF	-----	1.5	1.20	IJ				
1,2,3,7,8,9-HxCDF	0.97	-----	0.59	J	1,2,3,4-TCDD-13C	2.00	NA	
Total HxCDF	89.00	-----	0.86		1,2,3,7,8,9-HxCDD-13C	2.00	NA	
1,2,3,4,7,8-HxCDD	ND	-----	0.99		2,3,7,8-TCDD-37Cl4	0.20	81	
1,2,3,6,7,8-HxCDD	5.60	-----	1.30	J				
1,2,3,7,8,9-HxCDD	2.30	-----	1.10	J				
Total HxCDD	56.00	-----	1.10					
1,2,3,4,6,7,8-HpCDF	120.00	-----	0.64		Total 2,3,7,8-TCDD			
1,2,3,4,7,8,9-HpCDF	-----	1.8	0.73	IJ	Equivalence: 7.5 ng/Kg			
Total HpCDF	270.00	-----	0.69		(Using 2005 WHO Factors)			
1,2,3,4,6,7,8-HpCDD	120.00	-----	0.82					
Total HpCDD	280.00	-----	0.82					
OCDF	76.00	-----	1.20					
OCDD	1600.00	-----	0.84					

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REPORT OF LABORATORY ANALYSIS

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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-009-0.0-0.15		
Lab Sample ID	10365862004		
Filename	Y161019C_10		
Injected By	SMT		
Total Amount Extracted	13.6 g	Matrix	Solid
% Moisture	37.5	Dilution	NA
Dry Weight Extracted	8.50 g	Collected	10/11/2016 15:00
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 02:58

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.41	-----	0.21 J	2,3,7,8-TCDF-13C	2.00	75
Total TCDF	1.80	-----	0.21	2,3,7,8-TCDD-13C	2.00	84
				1,2,3,7,8-PeCDF-13C	2.00	77
2,3,7,8-TCDD	ND	-----	0.25	2,3,4,7,8-PeCDF-13C	2.00	73
Total TCDD	1.90	-----	0.25	1,2,3,7,8-PeCDD-13C	2.00	84
				1,2,3,4,7,8-HxCDF-13C	2.00	74
1,2,3,7,8-PeCDF	ND	-----	0.17	1,2,3,6,7,8-HxCDF-13C	2.00	73
2,3,4,7,8-PeCDF	0.29	-----	0.16 J	2,3,4,6,7,8-HxCDF-13C	2.00	75
Total PeCDF	1.50	-----	0.16 J	1,2,3,7,8,9-HxCDF-13C	2.00	68
				1,2,3,4,7,8-HxCDD-13C	2.00	65
1,2,3,7,8-PeCDD	0.37	-----	0.25 J	1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	3.70	-----	0.25 J	1,2,3,4,6,7,8-HpCDF-13C	2.00	53
				1,2,3,4,7,8,9-HpCDF-13C	2.00	57
1,2,3,4,7,8-HxCDF	0.50	-----	0.30 J	1,2,3,4,6,7,8-HpCDD-13C	2.00	57
1,2,3,6,7,8-HxCDF	-----	0.76	0.27 IJ	OCDD-13C	4.00	63 Y
2,3,4,6,7,8-HxCDF	0.58	-----	0.39 J			
1,2,3,7,8,9-HxCDF	ND	-----	0.31	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	20.00	-----	0.32	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.38	2,3,7,8-TCDD-37Cl4	0.20	81
1,2,3,6,7,8-HxCDD	0.92	-----	0.30 J			
1,2,3,7,8,9-HxCDD	0.59	-----	0.27 J			
Total HxCDD	10.00	-----	0.32			
1,2,3,4,6,7,8-HpCDF	29.00	-----	0.38	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.61	Equivalence: 1.4 ng/Kg		
Total HpCDF	58.00	-----	0.49	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	23.00	-----	0.47			
Total HpCDD	53.00	-----	0.47			
OCDF	14.00	-----	0.97			
OCDD	240.00	-----	0.85			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
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Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-011-0.0-0.15		
Lab Sample ID	10365862005		
Filename	Y161019C_11		
Injected By	SMT		
Total Amount Extracted	14.8 g	Matrix	Solid
% Moisture	55.5	Dilution	NA
Dry Weight Extracted	6.59 g	Collected	10/11/2016 15:05
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 03:41

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	2.10	-----	0.25		2,3,7,8-TCDF-13C	2.00	81
Total TCDF	10.00	-----	0.25		2,3,7,8-TCDD-13C	2.00	87
					1,2,3,7,8-PeCDF-13C	2.00	83
2,3,7,8-TCDD	0.67	-----	0.24	J	2,3,4,7,8-PeCDF-13C	2.00	77
Total TCDD	7.50	-----	0.24		1,2,3,7,8-PeCDD-13C	2.00	85
					1,2,3,4,7,8-HxCDF-13C	2.00	82
1,2,3,7,8-PeCDF	-----	0.78	0.28	PJ	1,2,3,6,7,8-HxCDF-13C	2.00	79
2,3,4,7,8-PeCDF	1.40	-----	0.18	J	2,3,4,6,7,8-HxCDF-13C	2.00	80
Total PeCDF	16.00	-----	0.23		1,2,3,7,8,9-HxCDF-13C	2.00	74
					1,2,3,4,7,8-HxCDD-13C	2.00	73
1,2,3,7,8-PeCDD	1.70	-----	0.41	J	1,2,3,6,7,8-HxCDD-13C	2.00	70
Total PeCDD	16.00	-----	0.41		1,2,3,4,6,7,8-HpCDF-13C	2.00	59
					1,2,3,4,7,8,9-HpCDF-13C	2.00	62
1,2,3,4,7,8-HxCDF	4.50	-----	0.82	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	63
1,2,3,6,7,8-HxCDF	7.10	-----	1.60	J	OCDD-13C	4.00	75 Y
2,3,4,6,7,8-HxCDF	2.40	-----	0.76	J			
1,2,3,7,8,9-HxCDF	-----	1.00	0.40	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	190.00	-----	0.91		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.89	-----	0.60	J	2,3,7,8-TCDD-37Cl4	0.20	83
1,2,3,6,7,8-HxCDD	6.50	-----	0.73	J			
1,2,3,7,8,9-HxCDD	2.50	-----	0.42	J			
Total HxCDD	62.00	-----	0.58				
1,2,3,4,6,7,8-HpCDF	320.00	-----	0.52		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	2.20	-----	0.78	J	Equivalence: 10 ng/Kg		
Total HpCDF	610.00	-----	0.65		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	110.00	-----	0.88				
Total HpCDD	250.00	-----	0.88				
OCDF	160.00	-----	1.10				
OCDD	1400.00	-----	1.00				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

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J = Estimated value
P = PCDE Interference
I = Interference present
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-111-0.0-0.15		
Lab Sample ID	10365862006		
Filename	Y161019C_12		
Injected By	SMT		
Total Amount Extracted	14.7 g	Matrix	Solid
% Moisture	54.4	Dilution	NA
Dry Weight Extracted	6.70 g	Collected	10/11/2016 15:10
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 04:23

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery	
2,3,7,8-TCDF	2.30	-----	0.28		2,3,7,8-TCDF-13C	2.00	77	
Total TCDF	9.50	-----	0.28		2,3,7,8-TCDD-13C	2.00	81	
					1,2,3,7,8-PeCDF-13C	2.00	77	
2,3,7,8-TCDD	0.67	-----	0.20	J	2,3,4,7,8-PeCDF-13C	2.00	73	
Total TCDD	9.50	-----	0.20		1,2,3,7,8-PeCDD-13C	2.00	79	
					1,2,3,4,7,8-HxCDF-13C	2.00	76	
1,2,3,7,8-PeCDF	-----	0.63	0.44	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	71	
2,3,4,7,8-PeCDF	1.10	-----	0.17	J	2,3,4,6,7,8-HxCDF-13C	2.00	73	
Total PeCDF	14.00	-----	0.30		1,2,3,7,8,9-HxCDF-13C	2.00	71	
					1,2,3,4,7,8-HxCDD-13C	2.00	69	
1,2,3,7,8-PeCDD	1.20	-----	0.25	J	1,2,3,6,7,8-HxCDD-13C	2.00	61	
Total PeCDD	16.00	-----	0.25		1,2,3,4,6,7,8-HpCDF-13C	2.00	52	
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59	
1,2,3,4,7,8-HxCDF	3.30	-----	0.72	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	55	
1,2,3,6,7,8-HxCDF	6.20	-----	0.17	J	OCDD-13C	4.00	65	Y
2,3,4,6,7,8-HxCDF	2.10	-----	0.30	J				
1,2,3,7,8,9-HxCDF	-----	1.00	0.40	IJ	1,2,3,4-TCDD-13C	2.00	NA	
Total HxCDF	180.00	-----	0.40		1,2,3,7,8,9-HxCDD-13C	2.00	NA	
1,2,3,4,7,8-HxCDD	ND	-----	0.89		2,3,7,8-TCDD-37Cl4	0.20	78	
1,2,3,6,7,8-HxCDD	7.10	-----	0.75	J				
1,2,3,7,8,9-HxCDD	2.90	-----	0.47	J				
Total HxCDD	71.00	-----	0.71					
1,2,3,4,6,7,8-HpCDF	320.00	-----	0.68		Total 2,3,7,8-TCDD			
1,2,3,4,7,8,9-HpCDF	2.60	-----	0.90	J	Equivalence: 10.0 ng/Kg			
Total HpCDF	610.00	-----	0.79		(Using 2005 WHO Factors)			
1,2,3,4,6,7,8-HpCDD	150.00	-----	1.10					
Total HpCDD	350.00	-----	1.10					
OCDF	180.00	-----	0.85					
OCDD	1800.00	-----	1.20					

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
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NC = Not Calculated

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J = Estimated value
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REPORT OF LABORATORY ANALYSIS

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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-013-0.0-0.15		
Lab Sample ID	10365862007		
Filename	Y161019C_13		
Injected By	SMT		
Total Amount Extracted	14.4 g	Matrix	Solid
% Moisture	64.2	Dilution	NA
Dry Weight Extracted	5.16 g	Collected	10/11/2016 15:15
ICAL ID	Y160816A	Received	10/12/2016 18:30
CCal Filename(s)	Y161019B_12 & Y161019C_19	Extracted	10/17/2016 17:00
Method Blank ID	BLANK-52398	Analyzed	10/20/2016 05:05

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	10.0	-----	0.44		2,3,7,8-TCDF-13C	2.00	80
Total TCDF	51.0	-----	0.44		2,3,7,8-TCDD-13C	2.00	88
					1,2,3,7,8-PeCDF-13C	2.00	75
2,3,7,8-TCDD	5.6	-----	0.44		2,3,4,7,8-PeCDF-13C	2.00	65
Total TCDD	46.0	-----	0.44		1,2,3,7,8-PeCDD-13C	2.00	73
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	-----	2.7	0.20	PJ	1,2,3,6,7,8-HxCDF-13C	2.00	69
2,3,4,7,8-PeCDF	7.0	-----	0.22	J	2,3,4,6,7,8-HxCDF-13C	2.00	74
Total PeCDF	130.0	-----	0.21		1,2,3,7,8,9-HxCDF-13C	2.00	71
					1,2,3,4,7,8-HxCDD-13C	2.00	71
1,2,3,7,8-PeCDD	22.0	-----	0.72		1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	150.0	-----	0.72		1,2,3,4,6,7,8-HpCDF-13C	2.00	56
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	17.0	-----	3.50		1,2,3,4,6,7,8-HpCDD-13C	2.00	61
1,2,3,6,7,8-HxCDF	23.0	-----	4.40		OCDD-13C	4.00	79 Y
2,3,4,6,7,8-HxCDF	13.0	-----	5.20				
1,2,3,7,8,9-HxCDF	5.5	-----	0.18	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	1100.0	-----	3.30		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	9.4	-----	1.30	J	2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	59.0	-----	1.50				
1,2,3,7,8,9-HxCDD	33.0	-----	1.40				
Total HxCDD	590.0	-----	1.40				
1,2,3,4,6,7,8-HpCDF	1800.0	-----	0.51		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	13.0	-----	1.90		Equivalence: 71 ng/Kg		
Total HpCDF	3400.0	-----	1.20		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	560.0	-----	1.80				
Total HpCDD	1200.0	-----	1.80				
OCDF	730.0	-----	1.10				
OCDD	4200.0	-----	1.90				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
P = PCDE Interference
Y = Calculated using average of daily RFs

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-014-0.0-0.15		
Lab Sample ID	10365862008		
Filename	U161025B_14		
Injected By	SMT		
Total Amount Extracted	17.4 g	Matrix	Solid
% Moisture	44.7	Dilution	NA
Dry Weight Extracted	9.62 g	Collected	10/11/2016 15:30
ICAL ID	U161025	Received	10/12/2016 18:30
CCal Filename(s)	U161025B_02 & U161025B_19	Extracted	10/21/2016 19:15
Method Blank ID	BLANK-52467	Analyzed	10/26/2016 07:12

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.50	-----	0.150	J	2,3,7,8-TCDF-13C	2.00	74
Total TCDF	2.00	-----	0.150		2,3,7,8-TCDD-13C	2.00	86
					1,2,3,7,8-PeCDF-13C	2.00	75
2,3,7,8-TCDD	ND	-----	0.230		2,3,4,7,8-PeCDF-13C	2.00	70
Total TCDD	2.10	-----	0.230		1,2,3,7,8-PeCDD-13C	2.00	86
					1,2,3,4,7,8-HxCDF-13C	2.00	69
1,2,3,7,8-PeCDF	0.12	-----	0.097	J	1,2,3,6,7,8-HxCDF-13C	2.00	66
2,3,4,7,8-PeCDF	0.26	-----	0.060	J	2,3,4,6,7,8-HxCDF-13C	2.00	72
Total PeCDF	2.70	-----	0.078	J	1,2,3,7,8,9-HxCDF-13C	2.00	75
					1,2,3,4,7,8-HxCDD-13C	2.00	78
1,2,3,7,8-PeCDD	0.19	-----	0.150	J	1,2,3,6,7,8-HxCDD-13C	2.00	60
Total PeCDD	3.20	-----	0.150	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	65
					1,2,3,4,7,8,9-HpCDF-13C	2.00	73
1,2,3,4,7,8-HxCDF	-----	0.44	0.180	IJ	1,2,3,4,6,7,8-HpCDD-13C	2.00	79
1,2,3,6,7,8-HxCDF	0.97	-----	0.028	J	OCDD-13C	4.00	70
2,3,4,6,7,8-HxCDF	0.38	-----	0.048	J			
1,2,3,7,8,9-HxCDF	ND	-----	0.032		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	20.00	-----	0.073		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.23	-----	0.080	J	2,3,7,8-TCDD-37Cl4	0.20	90
1,2,3,6,7,8-HxCDD	-----	0.89	0.098	IJ			
1,2,3,7,8,9-HxCDD	0.44	-----	0.041	J			
Total HxCDD	5.20	-----	0.073	J			
1,2,3,4,6,7,8-HpCDF	41.00	-----	0.690		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	0.38	-----	0.170	J	Equivalence: 1.3 ng/Kg		
Total HpCDF	76.00	-----	0.430		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	14.00	-----	0.250				
Total HpCDD	31.00	-----	0.250				
OCDF	19.00	-----	0.280				
OCDD	160.00	-----	0.230				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-015-0.0-0.15		
Lab Sample ID	10365862009		
Filename	U161025B_15		
Injected By	SMT		
Total Amount Extracted	16.6 g	Matrix	Solid
% Moisture	40.3	Dilution	NA
Dry Weight Extracted	9.91 g	Collected	10/11/2016 15:40
ICAL ID	U161025	Received	10/12/2016 18:30
CCal Filename(s)	U161025B_02 & U161025B_19	Extracted	10/21/2016 19:15
Method Blank ID	BLANK-52467	Analyzed	10/26/2016 08:00

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	0.37	-----	0.120	J	2,3,7,8-TCDF-13C	2.00	75
Total TCDF	2.30	-----	0.120		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	76
2,3,7,8-TCDD	ND	-----	0.086		2,3,4,7,8-PeCDF-13C	2.00	71
Total TCDD	1.40	-----	0.086		1,2,3,7,8-PeCDD-13C	2.00	87
					1,2,3,4,7,8-HxCDF-13C	2.00	73
1,2,3,7,8-PeCDF	0.23	-----	0.160	J	1,2,3,6,7,8-HxCDF-13C	2.00	70
2,3,4,7,8-PeCDF	0.28	-----	0.070	J	2,3,4,6,7,8-HxCDF-13C	2.00	74
Total PeCDF	4.60	-----	0.110	J	1,2,3,7,8,9-HxCDF-13C	2.00	80
					1,2,3,4,7,8-HxCDD-13C	2.00	78
1,2,3,7,8-PeCDD	0.20	-----	0.130	J	1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	2.20	-----	0.130	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	70
					1,2,3,4,7,8,9-HpCDF-13C	2.00	79
1,2,3,4,7,8-HxCDF	ND	-----	0.970		1,2,3,4,6,7,8-HpCDD-13C	2.00	84
1,2,3,6,7,8-HxCDF	1.80	-----	0.940	J	OCDD-13C	4.00	71
2,3,4,6,7,8-HxCDF	0.47	-----	0.110	J			
1,2,3,7,8,9-HxCDF	ND	-----	0.063		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	39.00	-----	0.520		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.120		2,3,7,8-TCDD-37Cl4	0.20	91
1,2,3,6,7,8-HxCDD	0.94	-----	0.120	J			
1,2,3,7,8,9-HxCDD	-----	0.35	0.120	IJ			
Total HxCDD	9.10	-----	0.120				
1,2,3,4,6,7,8-HpCDF	89.00	-----	0.170		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	0.44	0.220	IJ	Equivalence: 1.8 ng/Kg		
Total HpCDF	160.00	-----	0.200		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	15.00	-----	0.110				
Total HpCDD	37.00	-----	0.110				
OCDF	34.00	-----	0.410				
OCDD	180.00	-----	0.560				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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EDL = Estimated Detection Limit

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I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-001-0.0-0.15		
Lab Sample ID	10365862010		
Filename	U161025B_16		
Injected By	SMT		
Total Amount Extracted	18.3 g	Matrix	Solid
% Moisture	58.1	Dilution	NA
Dry Weight Extracted	7.67 g	Collected	09/21/2016 13:47
ICAL ID	U161025	Received	10/12/2016 18:30
CCal Filename(s)	U161025B_02 & U161025B_19	Extracted	10/21/2016 19:15
Method Blank ID	BLANK-52467	Analyzed	10/26/2016 08:47

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	7.6	-----	0.170		2,3,7,8-TCDF-13C	2.00	80
Total TCDF	35.0	-----	0.170		2,3,7,8-TCDD-13C	2.00	93
					1,2,3,7,8-PeCDF-13C	2.00	78
2,3,7,8-TCDD	4.0	-----	0.190		2,3,4,7,8-PeCDF-13C	2.00	74
Total TCDD	33.0	-----	0.190		1,2,3,7,8-PeCDD-13C	2.00	88
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	2.3	-----	0.130	J	1,2,3,6,7,8-HxCDF-13C	2.00	66
2,3,4,7,8-PeCDF	4.8	-----	0.086	J	2,3,4,6,7,8-HxCDF-13C	2.00	76
Total PeCDF	100.0	-----	0.110		1,2,3,7,8,9-HxCDF-13C	2.00	77
					1,2,3,4,7,8-HxCDD-13C	2.00	79
1,2,3,7,8-PeCDD	10.0	-----	0.410		1,2,3,6,7,8-HxCDD-13C	2.00	65
Total PeCDD	87.0	-----	0.410		1,2,3,4,6,7,8-HpCDF-13C	2.00	68
					1,2,3,4,7,8,9-HpCDF-13C	2.00	76
1,2,3,4,7,8-HxCDF	15.0	-----	0.420		1,2,3,4,6,7,8-HpCDD-13C	2.00	83
1,2,3,6,7,8-HxCDF	27.0	-----	0.270		OCDD-13C	4.00	72
2,3,4,6,7,8-HxCDF	11.0	-----	0.250				
1,2,3,7,8,9-HxCDF	3.9	-----	0.190	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	710.0	-----	0.280		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	6.7	-----	0.310		2,3,7,8-TCDD-37Cl4	0.20	94
1,2,3,6,7,8-HxCDD	45.0	-----	0.320				
1,2,3,7,8,9-HxCDD	28.0	-----	0.670				
Total HxCDD	370.0	-----	0.440				
1,2,3,4,6,7,8-HpCDF	1400.0	-----	0.840		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	9.1	-----	0.720		Equivalence: 49 ng/Kg		
Total HpCDF	2500.0	-----	0.780		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	440.0	-----	0.640				
Total HpCDD	930.0	-----	0.640				
OCDF	550.0	-----	0.450				
OCDD	4100.0	-----	0.170				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
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NA = Not Applicable
NC = Not Calculated

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J = Estimated value

REPORT OF LABORATORY ANALYSIS

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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-003-0.0-0.15		
Lab Sample ID	10365862011		
Filename	U161025B_17		
Injected By	SMT		
Total Amount Extracted	18.9 g	Matrix	Solid
% Moisture	55.1	Dilution	NA
Dry Weight Extracted	8.49 g	Collected	09/22/2016 12:34
ICAL ID	U161025	Received	10/12/2016 18:30
CCal Filename(s)	U161025B_02 & U161025B_19	Extracted	10/21/2016 19:15
Method Blank ID	BLANK-52467	Analyzed	10/26/2016 09:34

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	4.2	-----	0.240		2,3,7,8-TCDF-13C	2.00	82
Total TCDF	17.0	-----	0.240		2,3,7,8-TCDD-13C	2.00	96
					1,2,3,7,8-PeCDF-13C	2.00	82
2,3,7,8-TCDD	2.4	-----	0.190		2,3,4,7,8-PeCDF-13C	2.00	78
Total TCDD	19.0	-----	0.190		1,2,3,7,8-PeCDD-13C	2.00	93
					1,2,3,4,7,8-HxCDF-13C	2.00	80
1,2,3,7,8-PeCDF	1.7	-----	0.200	J	1,2,3,6,7,8-HxCDF-13C	2.00	74
2,3,4,7,8-PeCDF	3.8	-----	0.088	J	2,3,4,6,7,8-HxCDF-13C	2.00	79
Total PeCDF	64.0	-----	0.140		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	84
1,2,3,7,8-PeCDD	5.9	-----	0.360	J	1,2,3,6,7,8-HxCDD-13C	2.00	67
Total PeCDD	55.0	-----	0.360		1,2,3,4,6,7,8-HpCDF-13C	2.00	72
					1,2,3,4,7,8,9-HpCDF-13C	2.00	78
1,2,3,4,7,8-HxCDF	12.0	-----	0.250		1,2,3,4,6,7,8-HpCDD-13C	2.00	88
1,2,3,6,7,8-HxCDF	22.0	-----	0.240		OCDD-13C	4.00	78
2,3,4,6,7,8-HxCDF	7.5	-----	0.250				
1,2,3,7,8,9-HxCDF	3.3	-----	0.170	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	540.0	-----	0.230		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	4.4	-----	0.570	J	2,3,7,8-TCDD-37Cl4	0.20	92
1,2,3,6,7,8-HxCDD	34.0	-----	1.200				
1,2,3,7,8,9-HxCDD	18.0	-----	0.590				
Total HxCDD	260.0	-----	0.800				
1,2,3,4,6,7,8-HpCDF	1100.0	-----	0.330		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	9.2	-----	0.520		Equivalence: 36 ng/Kg		
Total HpCDF	2100.0	-----	0.430		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	360.0	-----	0.940				
Total HpCDD	700.0	-----	0.940				
OCDF	470.0	-----	0.340				
OCDD	2900.0	-----	0.260				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

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J = Estimated value

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52398	Matrix	Solid
Filename	F161019A_10	Dilution	NA
Total Amount Extracted	20.6 g	Extracted	10/17/2016 17:00
ICAL ID	F161011	Analyzed	10/19/2016 21:29
CCal Filename(s)	F161019A_03 & F161020A_02	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.049		2,3,7,8-TCDF-13C	2.00	69
Total TCDF	ND	-----	0.049		2,3,7,8-TCDD-13C	2.00	81
					1,2,3,7,8-PeCDF-13C	2.00	64
2,3,7,8-TCDD	ND	-----	0.060		2,3,4,7,8-PeCDF-13C	2.00	58
Total TCDD	ND	-----	0.060		1,2,3,7,8-PeCDD-13C	2.00	64
					1,2,3,4,7,8-HxCDF-13C	2.00	76
1,2,3,7,8-PeCDF	ND	-----	0.027		1,2,3,6,7,8-HxCDF-13C	2.00	75
2,3,4,7,8-PeCDF	-----	0.036	0.026	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	79
Total PeCDF	ND	-----	0.027		1,2,3,7,8,9-HxCDF-13C	2.00	76
					1,2,3,4,7,8-HxCDD-13C	2.00	68
1,2,3,7,8-PeCDD	ND	-----	0.032		1,2,3,6,7,8-HxCDD-13C	2.00	62
Total PeCDD	ND	-----	0.032		1,2,3,4,6,7,8-HpCDF-13C	2.00	52
					1,2,3,4,7,8,9-HpCDF-13C	2.00	49
1,2,3,4,7,8-HxCDF	-----	0.047	0.030	IJ	1,2,3,4,6,7,8-HpCDD-13C	2.00	58
1,2,3,6,7,8-HxCDF	ND	-----	0.039		OCDD-13C	4.00	43
2,3,4,6,7,8-HxCDF	0.041	-----	0.036	J			
1,2,3,7,8,9-HxCDF	ND	-----	0.046		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	0.041	-----	0.038	J	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.042		2,3,7,8-TCDD-37Cl4	0.20	73
1,2,3,6,7,8-HxCDD	ND	-----	0.037				
1,2,3,7,8,9-HxCDD	ND	-----	0.048				
Total HxCDD	ND	-----	0.042				
1,2,3,4,6,7,8-HpCDF	0.058	-----	0.049	J	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.066		Equivalence: 0.020 ng/Kg		
Total HpCDF	0.058	-----	0.057	J	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	ND	-----	0.053				
Total HpCDD	ND	-----	0.053				
OCDF	ND	-----	0.120				
OCDD	0.210	-----	0.160	J			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

Results reported on a total weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52467	Matrix	Solid
Filename	U161026A_02	Dilution	NA
Total Amount Extracted	10.5 g	Extracted	10/21/2016 19:15
ICAL ID	U161025	Analyzed	10/26/2016 12:54
CCal Filename(s)	U161025B_19 & U161026A_05	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.73	2,3,7,8-TCDF-13C	2.00	9 R
Total TCDF	ND	-----	0.73	2,3,7,8-TCDD-13C	2.00	10 R
				1,2,3,7,8-PeCDF-13C	2.00	10 R
2,3,7,8-TCDD	ND	-----	0.73	2,3,4,7,8-PeCDF-13C	2.00	10 R
Total TCDD	ND	-----	0.73	1,2,3,7,8-PeCDD-13C	2.00	12 R
				1,2,3,4,7,8-HxCDF-13C	2.00	11 R
1,2,3,7,8-PeCDF	ND	-----	0.65	1,2,3,6,7,8-HxCDF-13C	2.00	11 R
2,3,4,7,8-PeCDF	ND	-----	0.42	2,3,4,6,7,8-HxCDF-13C	2.00	14 R
Total PeCDF	ND	-----	0.53	1,2,3,7,8,9-HxCDF-13C	2.00	15 R
				1,2,3,4,7,8-HxCDD-13C	2.00	12 R
1,2,3,7,8-PeCDD	ND	-----	0.55	1,2,3,6,7,8-HxCDD-13C	2.00	11 R
Total PeCDD	ND	-----	0.55	1,2,3,4,6,7,8-HpCDF-13C	2.00	15 R
				1,2,3,4,7,8,9-HpCDF-13C	2.00	19 R
1,2,3,4,7,8-HxCDF	ND	-----	0.34	1,2,3,4,6,7,8-HpCDD-13C	2.00	19 R
1,2,3,6,7,8-HxCDF	ND	-----	0.29	OCDD-13C	4.00	20 R
2,3,4,6,7,8-HxCDF	ND	-----	0.33			
1,2,3,7,8,9-HxCDF	ND	-----	0.31	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.32	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.35	2,3,7,8-TCDD-37Cl4	0.20	80
1,2,3,6,7,8-HxCDD	ND	-----	0.30			
1,2,3,7,8,9-HxCDD	ND	-----	0.37			
Total HxCDD	ND	-----	0.34			
1,2,3,4,6,7,8-HpCDF	ND	-----	0.28	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.33	Equivalence: 0.00 ng/Kg		
Total HpCDF	ND	-----	0.31	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	ND	-----	0.29			
Total HpCDD	ND	-----	0.29			
OCDF	ND	-----	0.38			
OCDD	ND	-----	0.55			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

Results reported on a total weight basis and are valid to no more than 2 significant figures.
R = Recovery outside target range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52399		
Filename	F161019A_05	Matrix	Solid
Total Amount Extracted	20.2 g	Dilution	NA
ICAL ID	F161011	Extracted	10/17/2016 17:00
CCal Filename(s)	F161019A_03 & F161020A_02	Analyzed	10/19/2016 17:26
Method Blank ID	BLANK-52398	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.25	124	2,3,7,8-TCDF-13C	2.0	74
Total TCDF				2,3,7,8-TCDD-13C	2.0	87
				1,2,3,7,8-PeCDF-13C	2.0	71
2,3,7,8-TCDD	0.20	0.18	89	2,3,4,7,8-PeCDF-13C	2.0	63
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	71
				1,2,3,4,7,8-HxCDF-13C	2.0	80
1,2,3,7,8-PeCDF	1.0	1.2	118	1,2,3,6,7,8-HxCDF-13C	2.0	85
2,3,4,7,8-PeCDF	1.0	1.3	128	2,3,4,6,7,8-HxCDF-13C	2.0	83
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	79
				1,2,3,4,7,8-HxCDD-13C	2.0	75
1,2,3,7,8-PeCDD	1.0	0.99	99	1,2,3,6,7,8-HxCDD-13C	2.0	67
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	58
				1,2,3,4,7,8,9-HpCDF-13C	2.0	52
1,2,3,4,7,8-HxCDF	1.0	1.3	130	1,2,3,4,6,7,8-HpCDD-13C	2.0	64
1,2,3,6,7,8-HxCDF	1.0	1.2	117	OCDD-13C	4.0	45
2,3,4,6,7,8-HxCDF	1.0	1.2	118			
1,2,3,7,8,9-HxCDF	1.0	1.2	118	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.2	123	2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	1.0	1.2	119			
1,2,3,7,8,9-HxCDD	1.0	1.1	112			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	106			
1,2,3,4,7,8,9-HpCDF	1.0	1.0	104			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	0.94	94			
Total HpCDD						
OCDF	2.0	2.3	114			
OCDD	2.0	2.2	108			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range

Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52468		
Filename	Y161025A_04	Matrix	Solid
Total Amount Extracted	11.0 g	Dilution	NA
ICAL ID	Y160816A	Extracted	10/21/2016 19:15
CCal Filename(s)	Y161025A_03 & Y161025A_21	Analyzed	10/25/2016 14:39
Method Blank ID	BLANK-52467	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.21	107	2,3,7,8-TCDF-13C	2.0	68
Total TCDF				2,3,7,8-TCDD-13C	2.0	83
				1,2,3,7,8-PeCDF-13C	2.0	68
2,3,7,8-TCDD	0.20	0.17	87	2,3,4,7,8-PeCDF-13C	2.0	63
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	78
				1,2,3,4,7,8-HxCDF-13C	2.0	67
1,2,3,7,8-PeCDF	1.0	1.0	105	1,2,3,6,7,8-HxCDF-13C	2.0	69
2,3,4,7,8-PeCDF	1.0	1.1	112	2,3,4,6,7,8-HxCDF-13C	2.0	71
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	73
				1,2,3,4,7,8-HxCDD-13C	2.0	67
1,2,3,7,8-PeCDD	1.0	0.94	94	1,2,3,6,7,8-HxCDD-13C	2.0	71
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	75
				1,2,3,4,7,8,9-HpCDF-13C	2.0	80
1,2,3,4,7,8-HxCDF	1.0	1.1	115	1,2,3,4,6,7,8-HpCDD-13C	2.0	88
1,2,3,6,7,8-HxCDF	1.0	1.1	108	OCDD-13C	4.0	78
2,3,4,6,7,8-HxCDF	1.0	1.0	103			
1,2,3,7,8,9-HxCDF	1.0	1.1	106	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.2	115	2,3,7,8-TCDD-37Cl4	0.20	78
1,2,3,6,7,8-HxCDD	1.0	1.1	112			
1,2,3,7,8,9-HxCDD	1.0	1.2	116			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	105			
1,2,3,4,7,8,9-HpCDF	1.0	1.0	102			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	1.0	101			
Total HpCDD						
OCDF	2.0	2.1	104			
OCDD	2.0	2.1	106			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range
Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10365945

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/28/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☐	☒	☐	

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☐	☒	

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☐	☒	☐	
b.	Are the lab recovery limits specified on the report?		☐	☐	☒	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☐	☐	☒	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☐	☒	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	

	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question			Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.		☒	☐	☐	
	i.	Have the required matrix spikes been prepared and reported?	☒	☐	☐	Batch QC was reported with the samples in this SDG.
	ii.	If no, is there and explanation in the report as to why?	☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?	☐	☐	☒	
	iv.	Are the lab limits specified on the report?	☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
	vi.	Are there compounds outside the lab limits?	☒	☐	☐	
		1. If yes, are the analytes above the lab limits?	☐	☒	☐	
		2. Below the lab limits?	☒	☐	☐	
		3. Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	The MS recovery for TOC was biased low and outside QC limits in the batch QC reported with the samples in this SDG.
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.		☐	☒	☐	RPDs discussed apply to MS/MSDs.
	i.	Is the RPD for the duplicate pair within the lab limits?	☒	☐	☐	
	ii.	If no, has the associated source sample been flagged?	☐	☐	☒	
c.	What is the impact of failed QC on this project?		☐	☐	☒	No qualifiers were applied based on batch QC.

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

October 28, 2016

Nancy McDonald
Bay West
5 Empire Drive
Saint Paul, MN 55103

RE: Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Dear Nancy McDonald:
Enclosed are the analytical results for sample(s) received by the laboratory on October 12, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Lori Castille
lori.castille@pacelabs.com
Project Manager

Enclosures

cc: Paul Raymaker, Bay West
Jeff Smith, Pace Analytical Services, Inc

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

CERTIFICATIONS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792
Alaska Certification UST-107
Alaska Certification UST-107
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445
North Dakota Certification: # R-203
Wisconsin DNR Certification # : 998027470
WA Department of Ecology Lab ID# C1007
Nevada DNR #MN010842015-1
Oklahoma Department of Environmental Quality

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

SAMPLE SUMMARY

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10365945001	**BW16SR-006-0.0-0.15**	Solid	10/11/16 14:35	10/12/16 18:30
10365945002	**BW16SR-007-0.0-0.15**	Solid	10/11/16 14:40	10/12/16 18:30
10365945003	**BW16SR-008-0.0-0.15**	Solid	10/11/16 14:50	10/12/16 18:30
10365945004	**BW16SR-009-0.0-0.15**	Solid	10/11/16 15:00	10/12/16 18:30
10365945005	**BW16SR-011-0.0-0.15**	Solid	10/11/16 15:05	10/12/16 18:30
10365945006	**BW16SR-013-0.0-0.15**	Solid	10/11/16 15:15	10/12/16 18:30
10365945007	**BW16SR-014-0.0-0.15**	Solid	10/11/16 15:30	10/12/16 18:30
10365945008	**BW16SR-015-0.0-0.15**	Solid	10/11/16 15:40	10/12/16 18:30
10365945009	**BW16SR-001-0.0-0.15**	Solid	09/21/16 13:47	10/12/16 18:30
10365945010	**BW16SR-003-0.0-0.15**	Solid	09/22/16 12:34	10/12/16 18:30

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

SAMPLE ANALYTE COUNT

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10365945001	**BW16SR-006-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945002	**BW16SR-007-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945003	**BW16SR-008-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945004	**BW16SR-009-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945005	**BW16SR-011-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945006	**BW16SR-013-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945007	**BW16SR-014-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945008	**BW16SR-015-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945009	**BW16SR-001-0.0-0.15**	EPA 9060A	KRV	5	PASI-V
10365945010	**BW16SR-003-0.0-0.15**	EPA 9060A	KRV	5	PASI-V

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-006-0.0-0.15 **Lab ID: 10365945001** Collected: 10/11/16 14:35 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**22500**	mg/kg	2910	466	1		10/18/16 18:57	7440-44-0	
Total Organic Carbon	**28400**	mg/kg	2540	406	1		10/18/16 19:07	7440-44-0	
Total Organic Carbon	**27100**	mg/kg	2590	414	1		10/18/16 19:14	7440-44-0	
Total Organic Carbon	**29900**	mg/kg	2600	416	1		10/18/16 19:22	7440-44-0	
Mean Total Organic Carbon	**27000**	mg/kg	2660	425	1		10/18/16 19:22	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-007-0.0-0.15 **Lab ID: 10365945002** Collected: 10/11/16 14:40 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**20000**	mg/kg	2610	417	1		10/18/16 19:32	7440-44-0	
Total Organic Carbon	**19500**	mg/kg	2290	367	1		10/18/16 19:41	7440-44-0	
Total Organic Carbon	**18200**	mg/kg	2220	356	1		10/18/16 19:48	7440-44-0	
Total Organic Carbon	**21100**	mg/kg	2220	355	1		10/18/16 19:56	7440-44-0	
Mean Total Organic Carbon	**19700**	mg/kg	2340	374	1		10/18/16 19:56	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-008-0.0-0.15 **Lab ID: 10365945003** Collected: 10/11/16 14:50 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**33900**	mg/kg	3590	574	1		10/21/16 09:40	7440-44-0	
Total Organic Carbon	**33500**	mg/kg	3690	590	1		10/21/16 09:47	7440-44-0	
Total Organic Carbon	**20500**	mg/kg	3260	522	1		10/21/16 09:54	7440-44-0	
Total Organic Carbon	**15000**	mg/kg	3460	554	1		10/21/16 10:02	7440-44-0	
Mean Total Organic Carbon	**25700**	mg/kg	3500	560	1		10/21/16 10:02	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-009-0.0-0.15 **Lab ID: 10365945004** Collected: 10/11/16 15:00 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**9290**	mg/kg	3370	539	1		10/21/16 11:07	7440-44-0	
Total Organic Carbon	**12500**	mg/kg	1180	188	1		10/21/16 11:18	7440-44-0	
Total Organic Carbon	**11200**	mg/kg	1190	191	1		10/21/16 11:25	7440-44-0	
Total Organic Carbon	**13100**	mg/kg	1150	185	1		10/21/16 11:32	7440-44-0	
Mean Total Organic Carbon	**11500**	mg/kg	1720	276	1		10/21/16 11:32	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-011-0.0-0.15 **Lab ID: 10365945005** Collected: 10/11/16 15:05 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**27100**	mg/kg	1460	234	1		10/21/16 11:39	7440-44-0	
Total Organic Carbon	**33500**	mg/kg	1450	232	1		10/21/16 11:47	7440-44-0	
Total Organic Carbon	**13000**	mg/kg	1480	237	1		10/21/16 11:54	7440-44-0	
Total Organic Carbon	**18100**	mg/kg	1460	233	1		10/21/16 12:01	7440-44-0	
Mean Total Organic Carbon	**22900**	mg/kg	1460	234	1		10/21/16 12:01	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-013-0.0-0.15 **Lab ID: 10365945006** Collected: 10/11/16 15:15 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**56700**	mg/kg	3470	555	1		10/21/16 12:09	7440-44-0	
Total Organic Carbon	**60200**	mg/kg	3350	536	1		10/21/16 12:16	7440-44-0	
Total Organic Carbon	**51500**	mg/kg	3390	543	1		10/21/16 12:24	7440-44-0	
Total Organic Carbon	**49600**	mg/kg	3450	552	1		10/21/16 12:31	7440-44-0	
Mean Total Organic Carbon	**54500**	mg/kg	3420	547	1		10/21/16 12:31	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-014-0.0-0.15 **Lab ID: 10365945007** Collected: 10/11/16 15:30 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**12700**	mg/kg	2810	450	1		10/21/16 12:38	7440-44-0	
Total Organic Carbon	**17300**	mg/kg	1470	235	1		10/21/16 12:45	7440-44-0	
Total Organic Carbon	**15400**	mg/kg	1490	239	1		10/21/16 12:53	7440-44-0	
Total Organic Carbon	**11900**	mg/kg	1420	227	1		10/21/16 13:00	7440-44-0	
Mean Total Organic Carbon	**14300**	mg/kg	1800	288	1		10/21/16 13:00	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-015-0.0-0.15 **Lab ID: 10365945008** Collected: 10/11/16 15:40 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**17300**	mg/kg	1820	291	1		10/21/16 13:22	7440-44-0	
Total Organic Carbon	**18600**	mg/kg	1700	271	1		10/21/16 13:30	7440-44-0	
Total Organic Carbon	**8870**	mg/kg	1690	270	1		10/21/16 13:37	7440-44-0	
Total Organic Carbon	**11000**	mg/kg	1730	277	1		10/21/16 13:44	7440-44-0	
Mean Total Organic Carbon	**13900**	mg/kg	1730	278	1		10/21/16 13:44	7440-44-0	

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-001-0.0-0.15 **Lab ID: 10365945009** Collected: 09/21/16 13:47 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**39500**	mg/kg	1960	314	1		10/18/16 11:20	7440-44-0	
Total Organic Carbon	**45900**	mg/kg	2220	356	1		10/18/16 11:27	7440-44-0	
Total Organic Carbon	**33200**	mg/kg	2140	342	1		10/18/16 11:34	7440-44-0	
Total Organic Carbon	**39300**	mg/kg	2190	350	1		10/18/16 11:41	7440-44-0	
Mean Total Organic Carbon	**39500**	mg/kg	2130	340	1		10/18/16 11:41	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Sample: BW16SR-003-0.0-0.15 **Lab ID: 10365945010** Collected: 09/22/16 12:34 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
Total Organic Carbon Quad	Analytical Method: EPA 9060A								
Total Organic Carbon	**24600**	mg/kg	2260	362	1		10/18/16 11:49	7440-44-0	
Total Organic Carbon	**34300**	mg/kg	2360	378	1		10/18/16 11:57	7440-44-0	
Total Organic Carbon	**33900**	mg/kg	2620	418	1		10/18/16 12:04	7440-44-0	
Total Organic Carbon	**32900**	mg/kg	2500	401	1		10/18/16 12:11	7440-44-0	
Mean Total Organic Carbon	**31400**	mg/kg	2440	390	1		10/18/16 12:11	7440-44-0	

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

QC Batch: 97524 Analysis Method: EPA 9060A
QC Batch Method: EPA 9060A Analysis Description: 9060 TOC Average
Associated Lab Samples: 10365945001, 10365945002, 10365945009, 10365945010

METHOD BLANK: 385805 Matrix: Solid
Associated Lab Samples: 10365945001, 10365945002, 10365945009, 10365945010

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mean Total Organic Carbon	mg/kg	ND	300	48.0	10/18/16 07:29	

LABORATORY CONTROL SAMPLE: 385806

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mean Total Organic Carbon	mg/kg	5820	4730	81	49-151	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 385809 385810

Parameter	Units	10364112010 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mean Total Organic Carbon	mg/kg	5100	10100	10100	15000	15100	98	99	70-130	1	25	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

QC Batch: 97885 Analysis Method: EPA 9060A
QC Batch Method: EPA 9060A Analysis Description: 9060 TOC Average
Associated Lab Samples: 10365945003, 10365945004, 10365945005, 10365945006, 10365945007, 10365945008

METHOD BLANK: 387929 Matrix: Solid
Associated Lab Samples: 10365945003, 10365945004, 10365945005, 10365945006, 10365945007, 10365945008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mean Total Organic Carbon	mg/kg	ND	302	48.3	10/21/16 08:33	

LABORATORY CONTROL SAMPLE: 387930

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mean Total Organic Carbon	mg/kg	5820	4930	85	49-151	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 387931 387932

Parameter	Units	10365945003 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mean Total Organic Carbon	mg/kg	25700	37600	36100	65200	62600	105	102	70-130	4	25	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 387933 387934

Parameter	Units	10365379003 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mean Total Organic Carbon	mg/kg	21300	21800	22500	30700	39500	43	81	70-130	25	25	M1

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALIFIERS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-V	Pace Analytical Services - Virginia

ANALYTE QUALIFIERS

M1	Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
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(612)607-1700

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10365945

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10365945001	**BW16SR-006-0.0-0.15**	EPA 9060A	97524		
10365945001	**BW16SR-006-0.0-0.15**	EPA 9060A	97558		
10365945002	**BW16SR-007-0.0-0.15**	EPA 9060A	97524		
10365945002	**BW16SR-007-0.0-0.15**	EPA 9060A	97558		
10365945003	**BW16SR-008-0.0-0.15**	EPA 9060A	97885		
10365945003	**BW16SR-008-0.0-0.15**	EPA 9060A	97886		
10365945004	**BW16SR-009-0.0-0.15**	EPA 9060A	97885		
10365945004	**BW16SR-009-0.0-0.15**	EPA 9060A	97886		
10365945005	**BW16SR-011-0.0-0.15**	EPA 9060A	97885		
10365945005	**BW16SR-011-0.0-0.15**	EPA 9060A	97886		
10365945006	**BW16SR-013-0.0-0.15**	EPA 9060A	97885		
10365945006	**BW16SR-013-0.0-0.15**	EPA 9060A	97886		
10365945007	**BW16SR-014-0.0-0.15**	EPA 9060A	97885		
10365945007	**BW16SR-014-0.0-0.15**	EPA 9060A	97886		
10365945008	**BW16SR-015-0.0-0.15**	EPA 9060A	97885		
10365945008	**BW16SR-015-0.0-0.15**	EPA 9060A	97886		
10365945009	**BW16SR-001-0.0-0.15**	EPA 9060A	97524		
10365945009	**BW16SR-001-0.0-0.15**	EPA 9060A	97558		
10365945010	**BW16SR-003-0.0-0.15**	EPA 9060A	97524		
10365945010	**BW16SR-003-0.0-0.15**	EPA 9060A	97558		

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

10365945

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-5
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ITEM #	Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: TOC (SW-846 9060A Quad Burn)	Grain Size (ASTM D422 w/ hydrometer)	Comments
Ex.	BW15MLW-005	BW14MLW-005-0-0.15	SO	G	3/12/15	1204												
1	BW16SR-006	BW16SR-006-0.0-0.15	SO	G	10/11/16	1435	2	2								1	1	001
2	BW16SR-007	BW16SR-007-0.0-0.15	SO	G	10/11/16	1440	2	2								1	1	002
3	BW16SR-008	BW16SR-008-0.0-0.15	SO	G	10/11/16	1450	2	2								1	1	003
4	BW16SR-009	BW16SR-009-0.0-0.15	SO	G	10/11/16	1500	2	2								1	1	~~004~~
5	BW16SR-011	BW16SR-011-0.0-0.15	SO	G	10/11/16	1505	2	2								1	1	005
6	BW16SR-013	BW16SR-013-0.0-0.15	SO	G	10/11/16	1515	2	2								1	1	006 ~~007~~
7	BW16SR-014	BW16SR-014-0.0-0.15	SO	G	10/11/16	1530	2	2								1	1	007 ~~008~~
8	BW16SR-015	BW16SR-015-0.0-0.15	SO	G	10/11/16	1540	2	2								1	1	008 ~~009~~
9	BW16SR-001	BW16SR-001-0.0-0.15	SO	G	9/21/16	1347	2	2								1	1	009 ~~010~~
10	BW16SR-003	BW16SR-003-0.0-0.15	SO	G	9/22/16	1234	2	2								1	1	010 ~~011~~
11																		
12																		

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
Reference Subcontractor Goods and/or Services Purchase Order Form signed by Bay West on 9/19/16	Chris Musson / Bay West	10/12/16	1445	Lauren Hud / Pace	10-12-16	14:40	1.9	Y	N	Y
	Lauren Hud	10-12-16	1600		10/12/16	1600				
		10/12/16	1830	Erin Pace	10/12/16	1830	3.3 1.7			
							Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE	
PRINT Name of SAMPLER:	Chris Musson
SIGNATURE of SAMPLER:	DATE Signed (MM/DD/YY): 10/12/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: **WO# : 10365945**

10365945

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ SpeeDee ☐ Other:_____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☑ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☑ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 3.3, 1.5 **Cooler Temp Corrected (°C):** 3.5, 1.7 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** BC 10/12/16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☑ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☑ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☑ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: WT/SO	☑ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked? All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide) Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☑ Yes	☐ No	☐ N/A	13. ☑ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample # 1-5 Initial when completed: DC 10/11/16 Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Project Manager Review: Lori Peterson **Date:** 10/13/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Intra-Regional Chain of Custody

Pace Analytical®
www.pacelabs.com

Workorder: 10365945 **Workorder Name:** J160139 SLR Sediment AOCs **Owner Received Date:** 10/12/2016 **Due Date:** 10/26/2016

Received at:	Send To Lab:
Pace Analytical Minnesota 1700 Elm Street Suite 200 Minneapolis, MN 55414 Phone (612)607-1700	Pace Analytical Billings MT 150 N Ninth Street Billings, MT 59101 Phone (406)254-7226

Report To:
Lori Castille

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: Other	Requested Analysis: ASTM D422	LAB USE ONLY
1	BW16SR-006-0.0-0.15	PS	10/11/2016 14:35	10365945001	Solid		X	
2	BW16SR-007-0.0-0.15	PS	10/11/2016 14:40	10365945002	Solid		X	
3	BW16SR-008-0.0-0.15	PS	10/11/2016 14:50	10365945003	Solid		X	
4	BW16SR-009-0.0-0.15	PS	10/11/2016 15:00	10365945004	Solid		X	
5	BW16SR-011-0.0-0.15	PS	10/11/2016 15:05	10365945005	Solid		X	
6	BW16SR-013-0.0-0.15	PS	10/11/2016 15:15	10365945006	Solid		X	
7	BW16SR-014-0.0-0.15	PS	10/11/2016 15:30	10365945007	Solid		X	
8	BW16SR-015-0.0-0.15	PS	10/11/2016 15:40	10365945008	Solid		X	
9	BW16SR-001-0.0-0.15	PS	9/21/2016 13:47	10365945009	Solid	✓	X	
10	BW16SR-003-0.0-0.15	PS	9/22/2016 12:34	10365945010	Solid		X	

Transfers	Released By	Date/Time	Received By	Date/Time
1	[signature]	10/13/16 17:00		
2	FedEx		[signature]/Pace	10/14/16 09:45
3				
4				

Comments

Cooler Temperature on Receipt 3.4 °C | Custody Seal (Y) or N | Received on Ice (Y) or N | Samples Intact (Y) or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.*

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 04Aug2016 Page 1 of 1
	Document No.: **F-MT-C-184-Rev.10**	Issuing Authority: Pace Montana Quality Office

Sample Condition Upon Receipt

Client Name: Pace MN **Project #:** 1036594/5

Courier: ☒ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other:

Tracking Number: 6752 5820 6496

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☒ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

Thermometer Used: ☒ 160285052 ☐ 140279186 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun ☐ NA

Cooler Temp Read: 3.9 **Date and Initials of Person Examining Contents:** 10/14 NH

Cooler Temp Corrected: 3.4 **Biological Tissue Frozen?** ☐ Yes ☒ No

Temp should be above freezing to 6°C

				Comments:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☐ Yes	☐ No	☒ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved container.
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: SL	☒ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☒ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample # NA
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☒ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, WI-DRO (water)	☐ Yes	☒ No		Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased): NA				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: Lori Peterson **Date:** 10/14/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

Chain of Custody

WO#: 1277022

PM: CLJ Due Date: 10/28/16

CLIENT: PACE MPLS

www.pacelabs.com

Workorder: 10365945 **Workorder Name:** J160139 SLR Sediment AOCs **Owner Received Date:** 10/12/2016 **Results Requested By:** 10/26/2016

Report To	Subcontract To
Lori Castille Pace Analytical Minnesota 1700 Elm Street Suite 200 Minneapolis, MN 55414 Phone (612)607-1700	Pace Analytical Virginia MN 315 Chestnut Street Virginia, MN 55792 Phone (218)742-1042

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: Unpreserved	TOC 9060 Grab	LAB USE ONLY
1	BW16SR-006-0.0-0.15	PS	10/11/2016 14:35	10365945001	Solid	1	X	
2	BW16SR-007-0.0-0.15	PS	10/11/2016 14:40	10365945002	Solid	1	X	
3	BW16SR-008-0.0-0.15	PS	10/11/2016 14:50	10365945003	Solid	1	X	
4	BW16SR-009-0.0-0.15	PS	10/11/2016 15:00	10365945004	Solid	1	X	
5	BW16SR-011-0.0-0.15	PS	10/11/2016 15:05	10365945005	Solid	1	X	
6	BW16SR-013-0.0-0.15	PS	10/11/2016 15:15	10365945006	Solid	1	X	
7	BW16SR-014-0.0-0.15	PS	10/11/2016 15:30	10365945007	Solid	1	X	
8	BW16SR-015-0.0-0.15	PS	10/11/2016 15:40	10365945008	Solid	1	X	
9	BW16SR-001-0.0-0.15	PS	9/21/2016 13:47	10365945009	Solid	1	X	
10	BW16SR-003-0.0-0.15	PS	9/22/2016 12:34	10365945010	Solid	1	X	

Transfers	Released By	Date/Time	Received By	Date/Time
1		10/13/16 1700		10/13/16 0800
2		10/13/16 2315		10/14/16 0800
3				

Comments

Cooler Temperature on Receipt 3.1 °C | Custody Seal (Y) or N | Received on Ice (Y) or N | Samples Intact (Y) or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.*

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Pace - IN IV

Project #: WO#: 1277022 PM: CLJ Due Date: 10/28/16 CLIENT: PACE MPLS

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☑ Pace ☐ Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No Seals Intact? ☑ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☑ Other: Temp Blank? ☑ Yes ☐ No

Thermometer Used: ☑ 140792808 Type of Ice: ☑ Wet ☐ Blue ☐ None ☑ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.8 Cooler Temp Corrected °C: 3.1 Biological Tissue Frozen? ☐ Yes ☐ No ☑ NA

Temp should be above freezing to 6°C Correction Factor: +0.3 Date and Initials of Person Examining Contents: JPL 10/13/16

				Comments:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☐ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☐ Yes	☐ No	☑ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	☑ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	☐ No	☑ N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐ Yes	☐ No	☑ N/A	13.
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION Field Data Required? ☐ Yes ☐ No

Person Contacted: Date/Time:

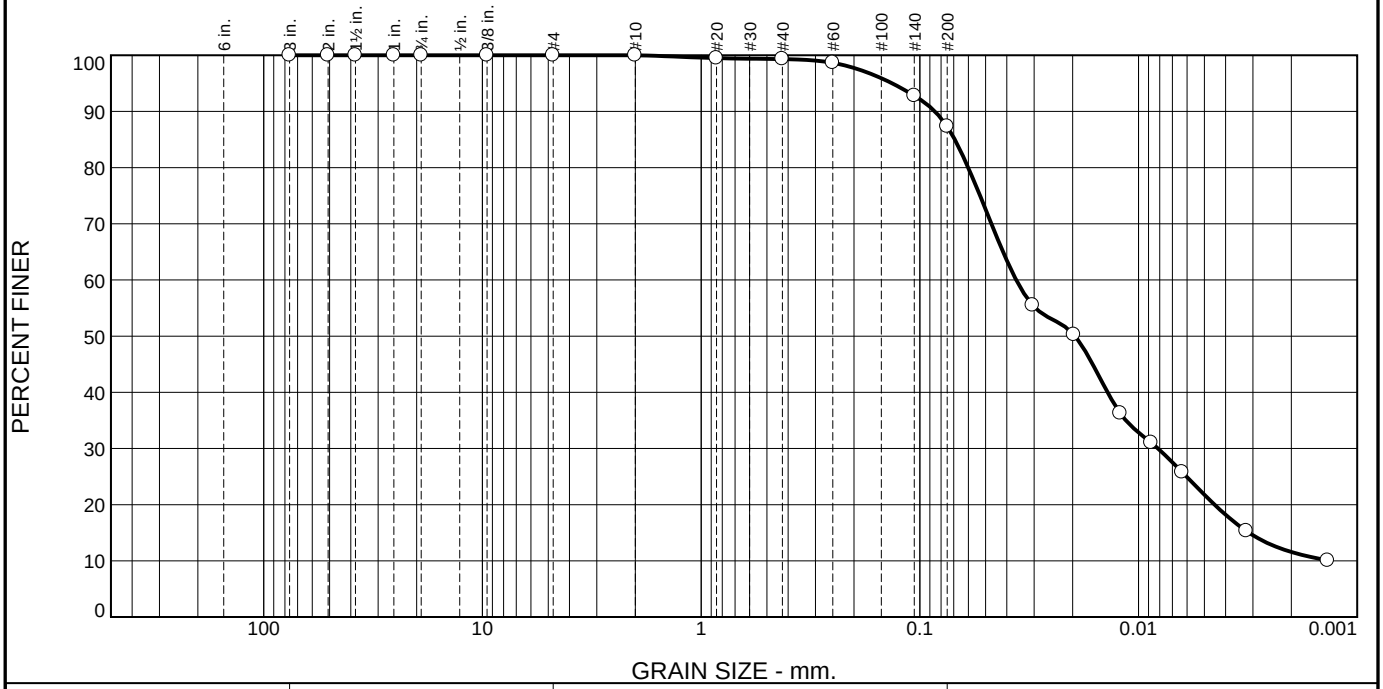
Comments/Resolution:

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: Date: 10/17/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	12	65	22

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	99		
#60	99		
#140	93		
#200	87		
0.0305 mm.	55		
0.0198 mm.	50		
0.0121 mm.	36		
0.0088 mm.	31		
0.0063 mm.	26		
0.0032 mm.	15		
0.0014 mm.	10		

* (no specification provided)

Material Description

silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0853 D_{85}= 0.0692 D_{60}= 0.0362
D_{50}= 0.0195 D_{30}= 0.0082 D_{15}= 0.0031
D_{10}= C_u= C_c=

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-006-0.0-0.15
Sample Number: 10365945-1

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-006-0.0-0.15
Sample Number: 10365945-1
Material Description: silt
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
858.04	589.13	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
57.21	0.00	#20	0.29	0.00	99
		#40	0.10	0.00	99
		#60	0.38	0.00	99
		#140	3.35	0.00	93
		#200	3.14	0.00	87

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =57.21
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	40.0	31.8	0.0138	40.0	9.7	0.0305	55.5
5.00	19.0	37.0	28.8	0.0138	37.0	10.2	0.0198	50.3
15.00	19.0	29.0	20.8	0.0138	29.0	11.5	0.0121	36.3
30.00	19.0	26.0	17.8	0.0138	26.0	12.0	0.0088	31.0
60.00	19.0	23.0	14.8	0.0138	23.0	12.5	0.0063	25.8
250.00	19.0	17.0	8.8	0.0138	17.0	13.5	0.0032	15.3
1440.00	19.0	14.0	5.8	0.0138	14.0	14.0	0.0014	10.1

Pace Analytical Services, Inc.

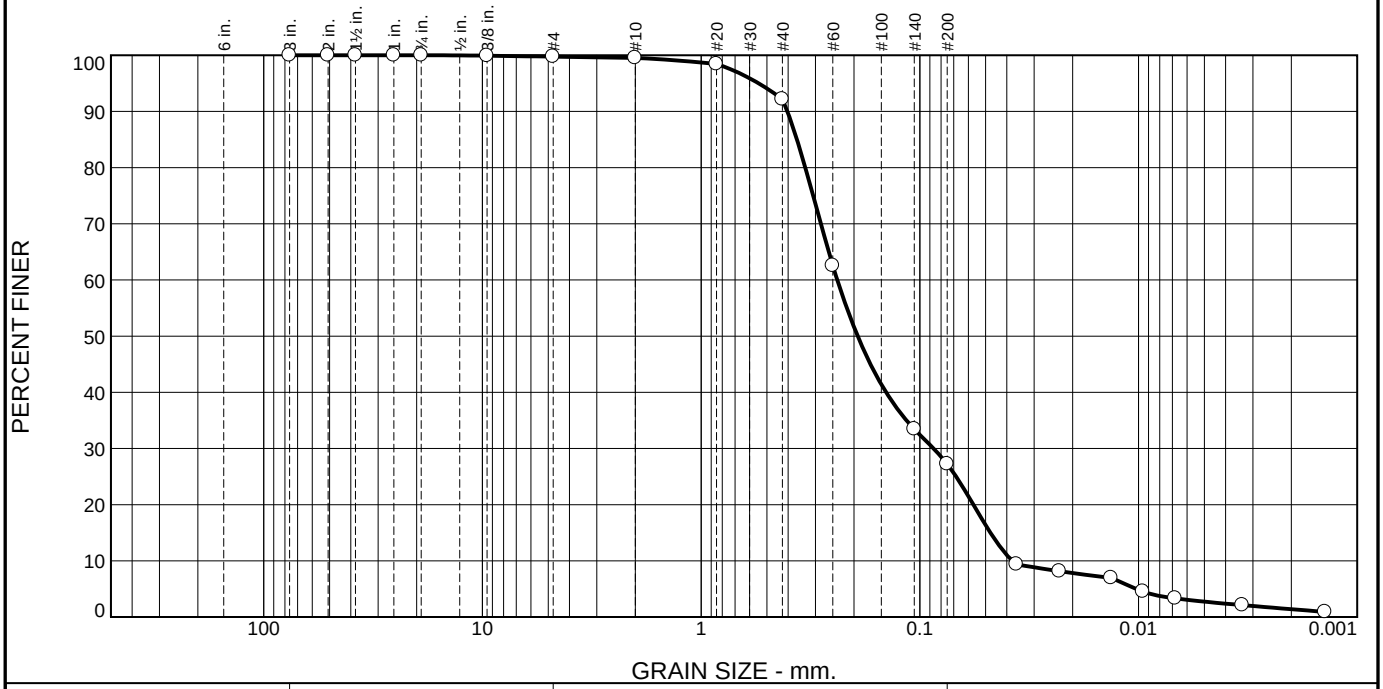
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	12	13	65	22	87

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
		0.0031	0.0045	0.0082	0.0139	0.0195	0.0362	0.0600	0.0692	0.0853	0.1338

Fineness Modulus
0.06

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	8	65	24	3

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	98		
#40	92		
#60	63		
#140	33		
#200	27		
0.0361 mm.	9.4		
0.0230 mm.	8.2		
0.0133 mm.	7.0		
0.0095 mm.	4.5		
0.0068 mm.	3.3		
0.0033 mm.	2.1		
0.0014 mm.	0.9		

* (no specification provided)

Material Description

silty sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= SM **AASHTO (M 145)=** A-2-4(0)

Coefficients

D_{90}= 0.4031 D_{85}= 0.3645 D_{60}= 0.2385
D_{50}= 0.1924 D_{30}= 0.0865 D_{15}= 0.0475
D_{10}= 0.0377 C_u= 6.32 C_c= 0.83

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-007-0.0-0.15
Sample Number: 10365945-2

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-007-0.0-0.15
Sample Number: 10365945-2
Material Description: silty sand
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: SM **AASHTO Classification:** A-2-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1089.46	569.32	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.43	0.00	100
		#4	0.84	0.00	100
		#10	1.17	0.00	100
82.29	0.00	#20	0.90	0.00	98
		#40	5.17	0.00	92
		#60	24.49	0.00	63
		#140	24.08	0.00	33
		#200	5.12	0.00	27

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =82.29
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	16.0	7.8	0.0138	16.0	13.7	0.0361	9.4
5.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0230	8.2
15.00	19.0	14.0	5.8	0.0138	14.0	14.0	0.0133	7.0
30.00	19.0	12.0	3.8	0.0138	12.0	14.3	0.0095	4.5
60.00	19.0	11.0	2.8	0.0138	11.0	14.5	0.0068	3.3
250.00	19.0	10.0	1.8	0.0138	10.0	14.7	0.0033	2.1
1440.00	19.0	9.0	0.8	0.0138	9.0	14.8	0.0014	0.9

Pace Analytical Services, Inc.

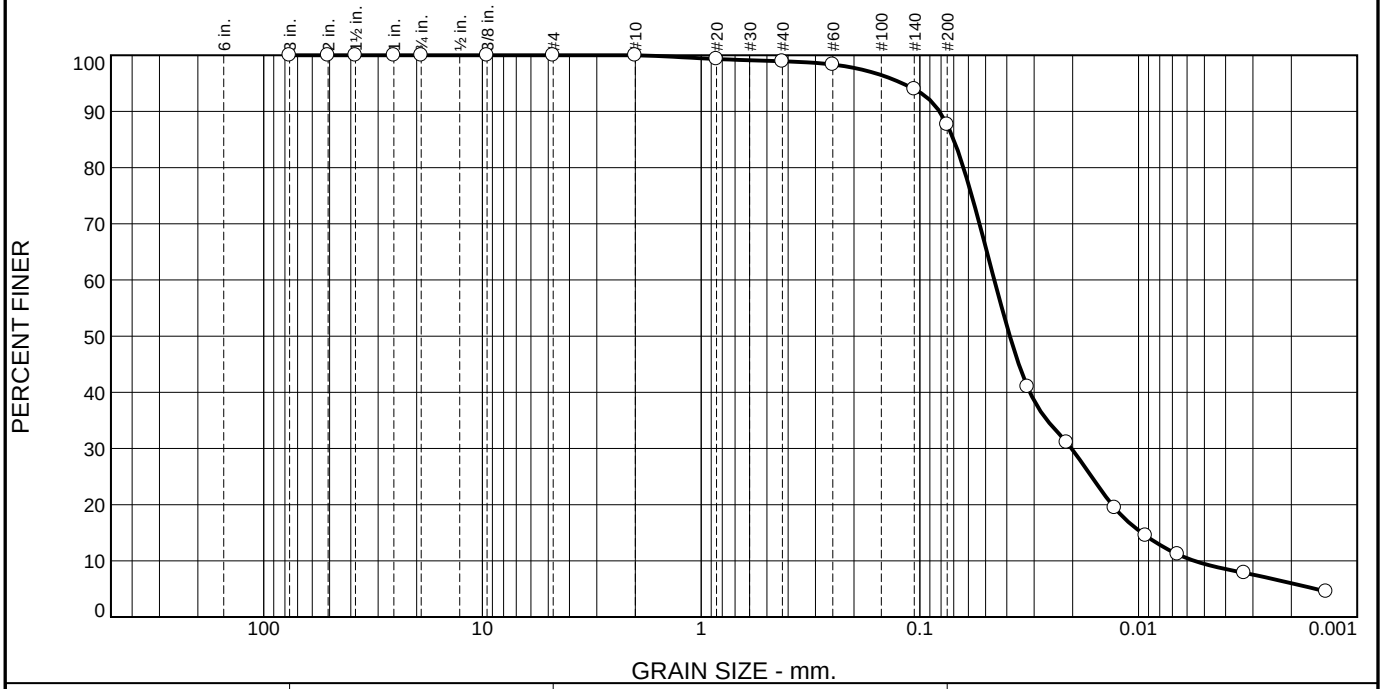
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	8	65	73	24	3	27

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0102	0.0377	0.0475	0.0569	0.0865	0.1426	0.1924	0.2385	0.3338	0.3645	0.4031	0.5458

Fineness Modulus	C_u	C_c
0.91	6.32	0.83

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	11	79	9

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	99		
#60	98		
#140	94		
#200	88		
0.0322 mm.	41		
0.0213 mm.	31		
0.0129 mm.	19		
0.0093 mm.	14		
0.0066 mm.	11		
0.0033 mm.	7.9		
0.0014 mm.	4.6		

* (no specification provided)

Material Description

silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0812 D_{85}= 0.0701 D_{60}= 0.0456
D_{50}= 0.0388 D_{30}= 0.0202 D_{15}= 0.0097
D_{10}= 0.0056 C_u= 8.22 C_c= 1.62

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-008-0.0-0.15
Sample Number: 10365945-3

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-008-0.0-0.15
Sample Number: 10365945-3
Material Description: silt
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1013.51	566.14	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
60.38	0.00	#20	0.40	0.00	99
		#40	0.25	0.00	99
		#60	0.35	0.00	98
		#140	2.65	0.00	94
		#200	3.80	0.00	88

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =60.38
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	33.0	24.8	0.0138	33.0	10.9	0.0322	41.0
5.00	19.0	27.0	18.8	0.0138	27.0	11.9	0.0213	31.1
15.00	19.0	20.0	11.8	0.0138	20.0	13.0	0.0129	19.5
30.00	19.0	17.0	8.8	0.0138	17.0	13.5	0.0093	14.5
60.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0066	11.2
250.00	19.0	13.0	4.8	0.0138	13.0	14.2	0.0033	7.9
1440.00	19.0	11.0	2.8	0.0138	11.0	14.5	0.0014	4.6

Pace Analytical Services, Inc.

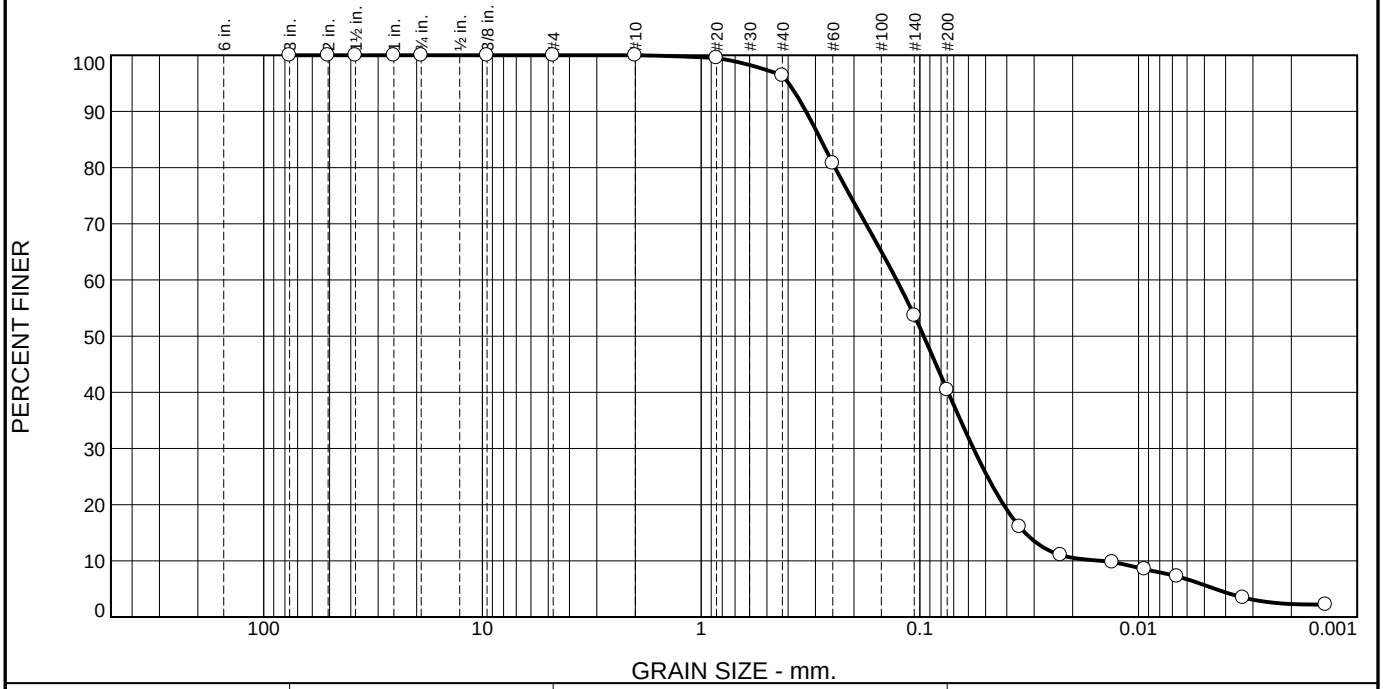
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	11	12	79	9	88

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0015	0.0056	0.0097	0.0132	0.0202	0.0314	0.0388	0.0456	0.0632	0.0701	0.0812	0.1201

Fineness Modulus	C_u	C_c
0.06	8.22	1.62

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	4	56	34	6

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	100		
#40	96		
#60	81		
#140	54		
#200	40		
0.0350 mm.	16		
0.0227 mm.	11		
0.0132 mm.	9.8		
0.0094 mm.	8.5		
0.0067 mm.	7.2		
0.0033 mm.	3.5		
0.0014 mm.	2.2		

* (no specification provided)

Material Description

silty sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= SM **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.3313 D_{85}= 0.2840 D_{60}= 0.1276
D_{50}= 0.0960 D_{30}= 0.0569 D_{15}= 0.0331
D_{10}= 0.0148 C_u= 8.64 C_c= 1.71

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-009-0.0-0.15
Sample Number: 10365945-4

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-009-0.0-0.15
Sample Number: 10365945-4
Material Description: silty sand
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: SM **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1202.44	574.38	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
79.33	0.00	#20	0.38	0.00	100
		#40	2.47	0.00	96
		#60	12.41	0.00	81
		#140	21.49	0.00	54
		#200	10.51	0.00	40

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =79.33
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	21.0	12.8	0.0138	21.0	12.9	0.0350	16.1
5.00	19.0	17.0	8.8	0.0138	17.0	13.5	0.0227	11.0
15.00	19.0	16.0	7.8	0.0138	16.0	13.7	0.0132	9.8
30.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0094	8.5
60.00	19.0	14.0	5.8	0.0138	14.0	14.0	0.0067	7.2
250.00	19.0	11.0	2.8	0.0138	11.0	14.5	0.0033	3.5
1440.00	19.0	10.0	1.8	0.0138	10.0	14.7	0.0014	2.2

Pace Analytical Services, Inc.

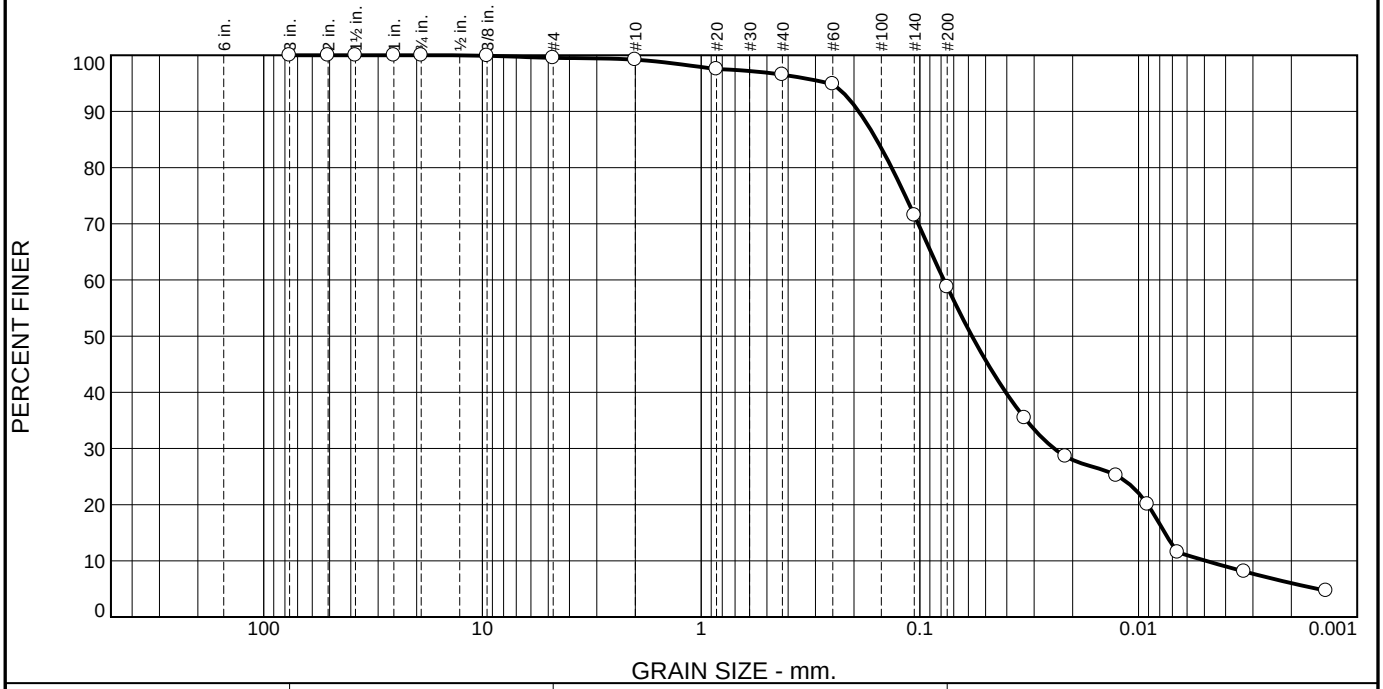
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	4	56	60	34	6	40

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0044	0.0148	0.0331	0.0414	0.0569	0.0742	0.0960	0.1276	0.2442	0.2840	0.3313	0.3976

Fineness Modulus	C_u	C_c
0.50	8.64	1.71

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	1	2	38	49	10

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	99		
#20	98		
#40	97		
#60	95		
#140	72		
#200	59		
0.0332 mm.	35		
0.0216 mm.	29		
0.0126 mm.	25		
0.0091 mm.	20		
0.0066 mm.	12		
0.0033 mm.	8.1		
0.0014 mm.	4.7		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.1902 D_{85}= 0.1577 D_{60}= 0.0776
D_{50}= 0.0577 D_{30}= 0.0243 D_{15}= 0.0076
D_{10}= 0.0049 C_u= 15.79 C_c= 1.55

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-011-0.0-0.15
Sample Number: 10365945-5

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-011-0.0-0.15
Sample Number: 10365945-5
Material Description: sandy silt
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
775.45	558.62	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.21	0.00	100
		#4	0.78	0.00	100
		#10	0.74	0.00	99
58.09	0.00	#20	0.98	0.00	98
		#40	0.58	0.00	97
		#60	0.98	0.00	95
		#140	13.66	0.00	72
		#200	7.48	0.00	59

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 99
Weight of hydrometer sample =58.09
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	29.0	20.8	0.0138	29.0	11.5	0.0332	35.4
5.00	19.0	25.0	16.8	0.0138	25.0	12.2	0.0216	28.6
15.00	19.0	23.0	14.8	0.0138	23.0	12.5	0.0126	25.2
30.00	19.0	20.0	11.8	0.0138	20.0	13.0	0.0091	20.1
60.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0066	11.5
250.00	19.0	13.0	4.8	0.0138	13.0	14.2	0.0033	8.1
1440.00	19.0	11.0	2.8	0.0138	11.0	14.5	0.0014	4.7

Pace Analytical Services, Inc.

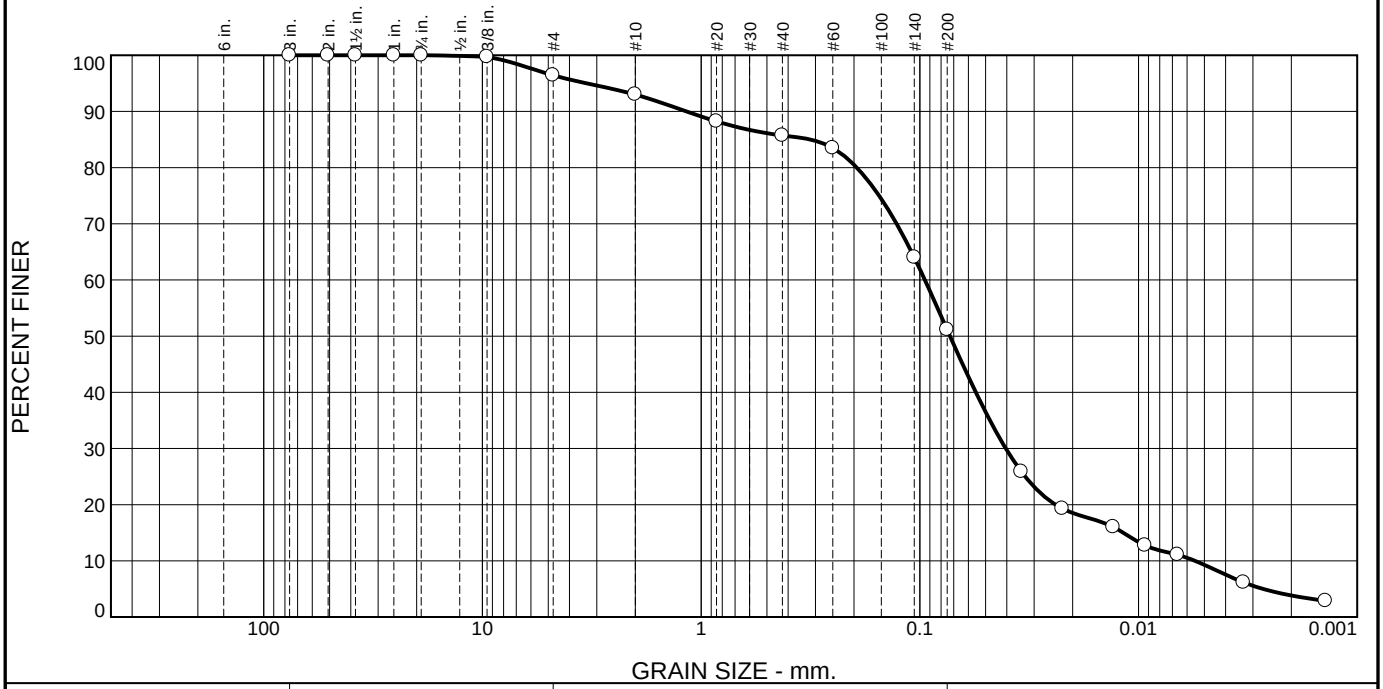
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	1	2	38	41	49	10	59

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0015	0.0049	0.0076	0.0091	0.0243	0.0405	0.0577	0.0776	0.1347	0.1577	0.1902	0.2589

Fineness Modulus	C_u	C_c
0.27	15.79	1.55

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	4	3	7	35	42	9

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	96		
#10	93		
#20	88		
#40	86		
#60	83		
#140	64		
#200	51		
0.0343 mm.	26		
0.0223 mm.	19		
0.0130 mm.	16		
0.0093 mm.	13		
0.0066 mm.	11		
0.0033 mm.	6.2		
0.0014 mm.	2.9		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 1.1678 D_{85}= 0.3169 D_{60}= 0.0948
D_{50}= 0.0728 D_{30}= 0.0405 D_{15}= 0.0117
D_{10}= 0.0055 C_u= 17.19 C_c= 3.13

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-013-0.0-0.15
Sample Number: 10365945-6

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-013-0.0-0.15
Sample Number: 10365945-6
Material Description: sandy silt
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
739.64	565.75	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.49	0.00	100
		#4	5.70	0.00	96
		#10	5.98	0.00	93
56.61	0.00	#20	2.91	0.00	88
		#40	1.52	0.00	86
		#60	1.37	0.00	83
		#140	11.85	0.00	64
		#200	7.83	0.00	51

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 93
Weight of hydrometer sample =56.61
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	24.0	15.8	0.0138	24.0	12.4	0.0343	25.9
5.00	19.0	20.0	11.8	0.0138	20.0	13.0	0.0223	19.3
15.00	19.0	18.0	9.8	0.0138	18.0	13.3	0.0130	16.0
30.00	19.0	16.0	7.8	0.0138	16.0	13.7	0.0093	12.7
60.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0066	11.1
250.00	19.0	12.0	3.8	0.0138	12.0	14.3	0.0033	6.2
1440.00	19.0	10.0	1.8	0.0138	10.0	14.7	0.0014	2.9

Pace Analytical Services, Inc.

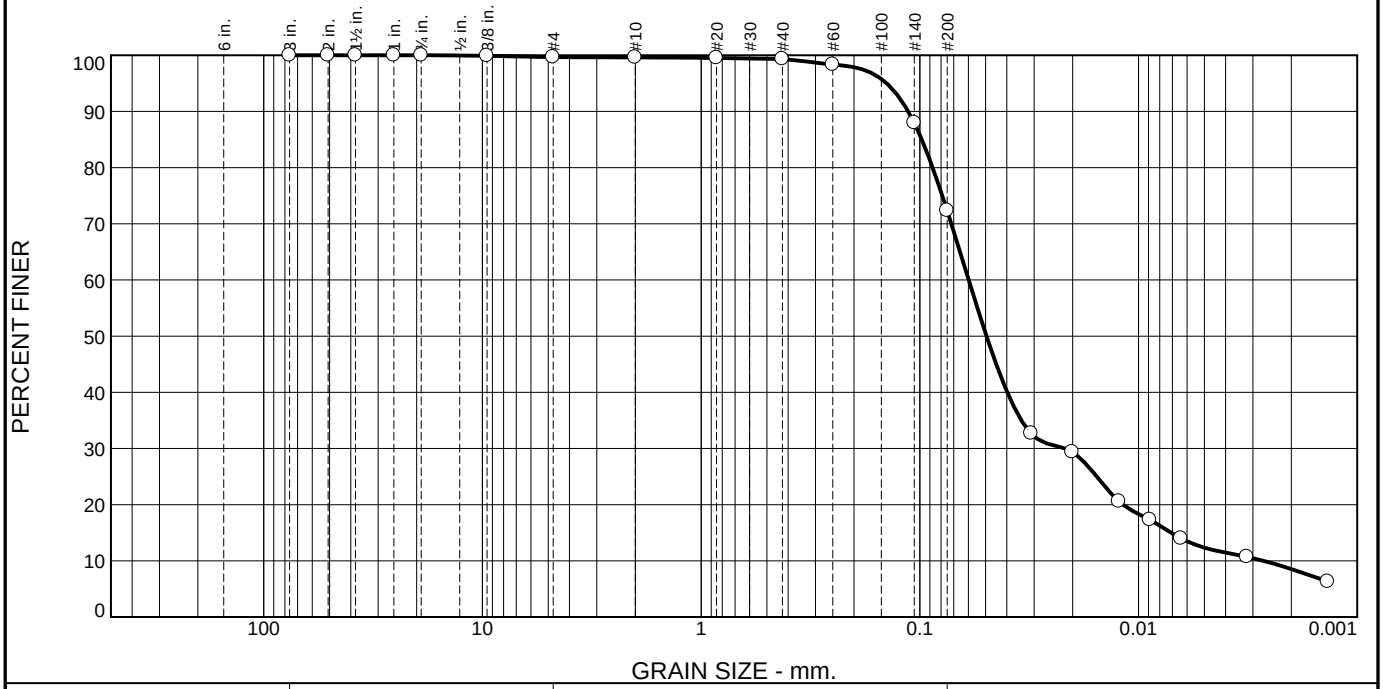
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	4	4	3	7	35	45	42	9	51

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
0.0027	0.0055	0.0117	0.0240	0.0405	0.0554	0.0728	0.0948	0.1942	0.3169	1.1678	3.3371

Fineness Modulus	C_u	C_c
0.74	17.19	3.13

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	27	60	12

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	100		
#40	99		
#60	98		
#140	88		
#200	72		
0.0310 mm.	33		
0.0201 mm.	29		
0.0123 mm.	21		
0.0089 mm.	17		
0.0064 mm.	14		
0.0032 mm.	11		
0.0014 mm.	6.3		

* (no specification provided)

Material Description

silt with sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.1130 D_{85}= 0.0979 D_{60}= 0.0598
D_{50}= 0.0495 D_{30}= 0.0217 D_{15}= 0.0071
D_{10}= 0.0027 C_u= 22.29 C_c= 2.93

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-014-0.0-0.15
Sample Number: 10365945-7

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-014-0.0-0.15
Sample Number: 10365945-7
Material Description: silt with sand
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
914.92	573.10	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.36	0.00	100
		#4	0.73	0.00	100
		#10	0.22	0.00	100
90.71	0.00	#20	0.09	0.00	100
		#40	0.17	0.00	99
		#60	0.90	0.00	98
		#140	9.45	0.00	88
		#200	14.22	0.00	72

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =90.71
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	38.0	29.8	0.0138	38.0	10.1	0.0310	32.7
5.00	19.0	35.0	26.8	0.0138	35.0	10.6	0.0201	29.4
15.00	19.0	27.0	18.8	0.0138	27.0	11.9	0.0123	20.6
30.00	19.0	24.0	15.8	0.0138	24.0	12.4	0.0089	17.3
60.00	19.0	21.0	12.8	0.0138	21.0	12.9	0.0064	14.0
250.00	19.0	18.0	9.8	0.0138	18.0	13.3	0.0032	10.7
1440.00	19.0	14.0	5.8	0.0138	14.0	14.0	0.0014	6.3

Pace Analytical Services, Inc.

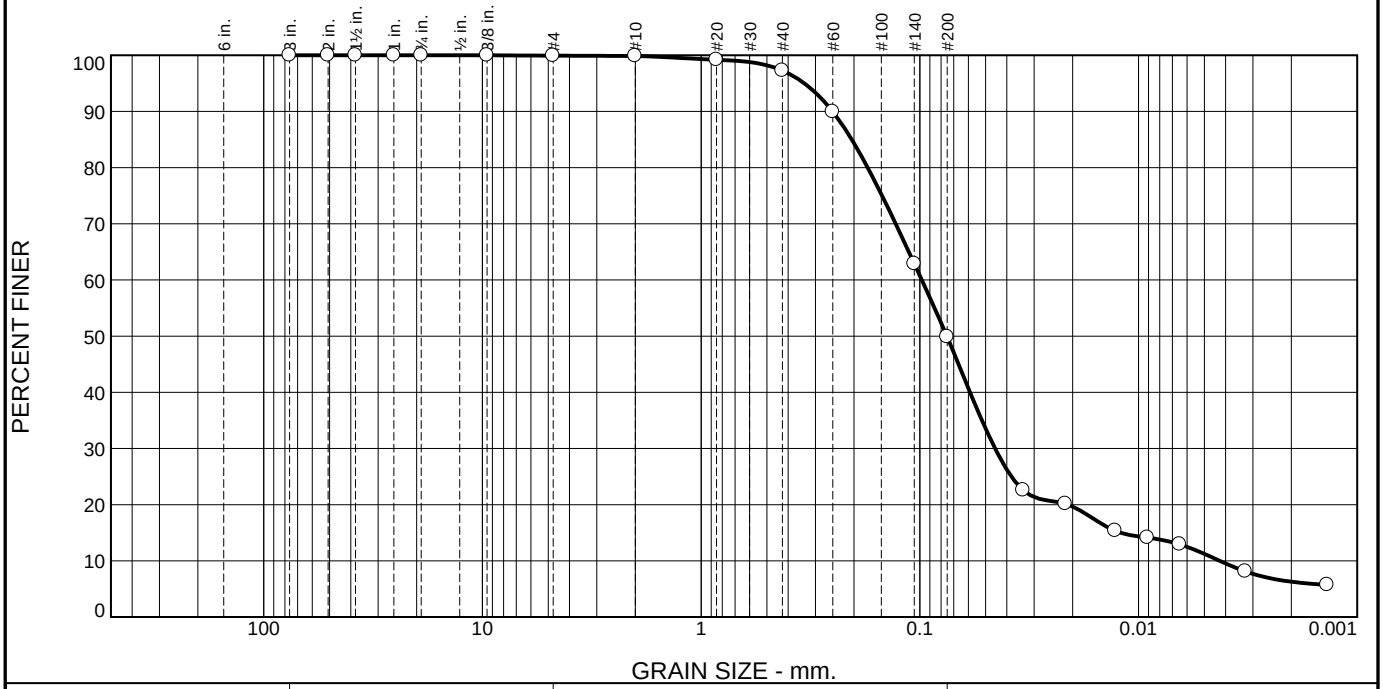
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	27	28	60	12	72

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0027	0.0071	0.0118	0.0217	0.0398	0.0495	0.0598	0.0874	0.0979	0.1130	0.1418

Fineness Modulus	C_u	C_c
0.07	22.29	2.93

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	3	47	39	11

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	97		
#60	90		
#140	63		
#200	50		
0.0337 mm.	23		
0.0216 mm.	20		
0.0128 mm.	15		
0.0091 mm.	14		
0.0065 mm.	13		
0.0033 mm.	8.1		
0.0014 mm.	5.7		

* (no specification provided)

Material Description

sandy silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.2507 D_{85}= 0.2049 D_{60}= 0.0979
D_{50}= 0.0752 D_{30}= 0.0453 D_{15}= 0.0121
D_{10}= 0.0042 C_u= 23.04 C_c= 4.93

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-015-0.0-0.15
Sample Number: 10365945-8

Date Sampled: 10/11/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-015-0.0-0.15
Sample Number: 10365945-8
Material Description: sandy silt
Sample Date: 10/11/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1047.60	577.96	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.33	0.00	100
		#10	0.23	0.00	100
83.01	0.00	#20	0.57	0.00	99
		#40	1.60	0.00	97
		#60	6.09	0.00	90
		#140	22.48	0.00	63
		#200	10.82	0.00	50

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =83.01
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	27.0	18.8	0.0138	27.0	11.9	0.0337	22.6
5.00	19.0	25.0	16.8	0.0138	25.0	12.2	0.0216	20.2
15.00	19.0	21.0	12.8	0.0138	21.0	12.9	0.0128	15.3
30.00	19.0	20.0	11.8	0.0138	20.0	13.0	0.0091	14.1
60.00	19.0	19.0	10.8	0.0138	19.0	13.2	0.0065	12.9
250.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0033	8.1
1440.00	19.0	13.0	4.8	0.0138	13.0	14.2	0.0014	5.7

Pace Analytical Services, Inc.

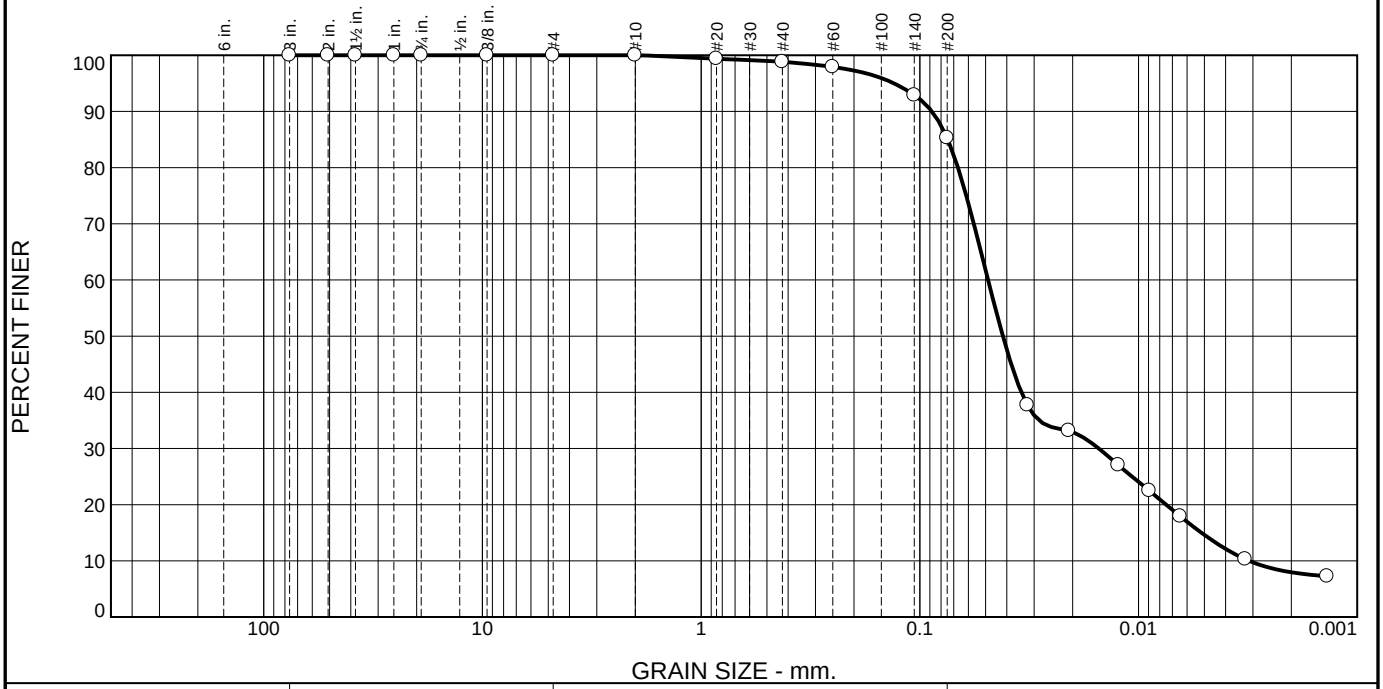
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	3	47	50	39	11	50

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0042	0.0121	0.0210	0.0453	0.0589	0.0752	0.0979	0.1732	0.2049	0.2507	0.3371

Fineness Modulus	C_u	C_c
0.33	23.04	4.93

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	14	70	15

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	99		
#40	99		
#60	98		
#140	93		
#200	85		
0.0322 mm.	38		
0.0208 mm.	33		
0.0124 mm.	27		
0.0089 mm.	22		
0.0064 mm.	18		
0.0033 mm.	10		
0.0014 mm.	7.2		

* (no specification provided)

Material Description

silt

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0880 D_{85}= 0.0745 D_{60}= 0.0487
D_{50}= 0.0417 D_{30}= 0.0152 D_{15}= 0.0052
D_{10}= 0.0031 C_u= 15.60 C_c= 1.52

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-001-0.0-0.15
Sample Number: 10365945-9

Date Sampled: 9/21/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-001-0.0-0.15
Sample Number: 10365945-9
Material Description: silt
Sample Date: 9/21/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1097.45	584.59	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
65.61	0.00	#20	0.41	0.00	99
		#40	0.36	0.00	99
		#60	0.60	0.00	98
		#140	3.29	0.00	93
		#200	5.00	0.00	85

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =65.61
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	33.0	24.8	0.0138	33.0	10.9	0.0322	37.7
5.00	19.0	30.0	21.8	0.0138	30.0	11.4	0.0208	33.2
15.00	19.0	26.0	17.8	0.0138	26.0	12.0	0.0124	27.1
30.00	19.0	23.0	14.8	0.0138	23.0	12.5	0.0089	22.5
60.00	19.0	20.0	11.8	0.0138	20.0	13.0	0.0064	17.9
250.00	19.0	15.0	6.8	0.0138	15.0	13.8	0.0033	10.3
1440.00	19.0	13.0	4.8	0.0138	13.0	14.2	0.0014	7.2

Pace Analytical Services, Inc.

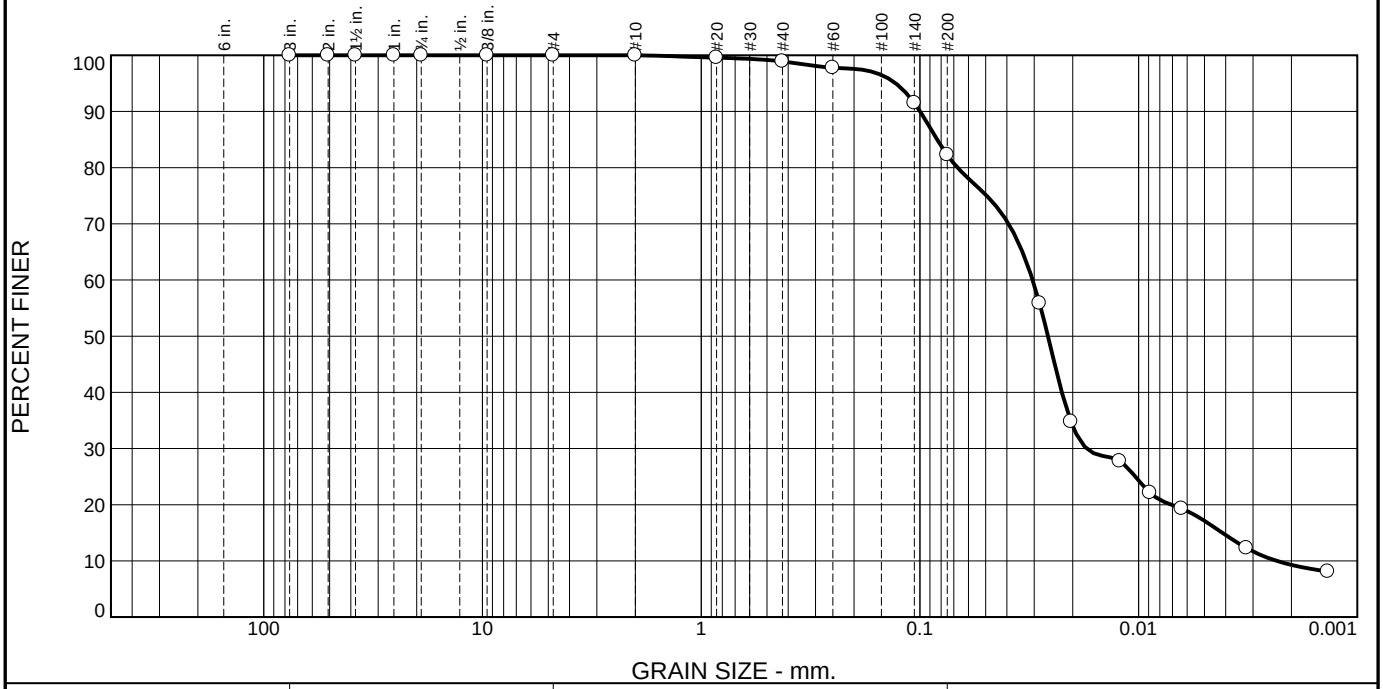
Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	14	15	70	15	85

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0031	0.0052	0.0075	0.0152	0.0344	0.0417	0.0487	0.0670	0.0745	0.0880	0.1314

Fineness Modulus	C_u	C_c
0.07	15.60	1.52

Particle Size Distribution Report



% +3"	% Gravel		% Sand			% Fines	
	Coarse	Fine	Coarse	Medium	Fine	Silt	Clay
0	0	0	0	1	17	65	17

TEST RESULTS (ASTM D422)

Opening Size	Percent Finer	Spec.* (Percent)	Pass? (X=Fail)
3	100		
2	100		
1.5	100		
1	100		
.75	100		
.375	100		
#4	100		
#10	100		
#20	100		
#40	99		
#60	98		
#140	92		
#200	82		
0.0284 mm.	56		
0.0204 mm.	35		
0.0122 mm.	28		
0.0089 mm.	22		
0.0064 mm.	19		
0.0032 mm.	12		
0.0014 mm.	8.1		

* (no specification provided)

Material Description

silt with sand

Atterberg Limits (ASTM D 4318)

PL= NP **LL=** NV **PI=**

Classification

USCS (D 2487)= ML **AASHTO (M 145)=** A-4(0)

Coefficients

D_{90}= 0.0998 D_{85}= 0.0833 D_{60}= 0.0305
D_{50}= 0.0260 D_{30}= 0.0173 D_{15}= 0.0041
D_{10}= 0.0023 C_u= 13.07 C_c= 4.20

Remarks

Date Received: 10/12/16 **Date Tested:** 10/26/16

Tested By: Will Thomas

Checked By: Rhonda Johnson

Title: Lab Manager

Location: BW16SR-003-0.0-0.15
Sample Number: 10365945-10

Date Sampled: 9/22/16

Pace Analytical Services, Inc.

Billings, MT

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's

Project No: **Figure**

GRAIN SIZE DISTRIBUTION TEST DATA

10/27/2016

Client: Bay West, Inc
Project: J160139 SLR Sediment AOC's
Location: BW16SR-003-0.0-0.15
Sample Number: 10365945-10
Material Description: silt with sand
Sample Date: 9/22/16
Date Received: 10/12/16 **PL:** NP **LL:** NV
USCS Classification: ML **AASHTO Classification:** A-4(0)
Grain Size Test Method: ASTM D422
Tested By: Will Thomas **Test Date:** 10/26/16
Checked By: Rhonda Johnson **Title:** Lab Manager

Sieve Test Data

Dry Sample and Tare (grams)	Tare (grams)	Sieve Opening Size	Weight Retained (grams)	Sieve Weight (grams)	Percent Finer
1179.88	581.09	3	0.00	0.00	100
		2	0.00	0.00	100
		1.5	0.00	0.00	100
		1	0.00	0.00	100
		.75	0.00	0.00	100
		.375	0.00	0.00	100
		#4	0.00	0.00	100
		#10	0.00	0.00	100
71.18	0.00	#20	0.31	0.00	100
		#40	0.47	0.00	99
		#60	0.80	0.00	98
		#140	4.47	0.00	92
		#200	6.57	0.00	82

Hydrometer Test Data

Hydrometer test uses material passing #10
Percent passing #10 based upon complete sample = 100
Weight of hydrometer sample =71.18
Automatic temperature correction
Composite correction (fluid density and meniscus height) at 20 deg. C = -8
Meniscus correction only = 0.0
Specific gravity of solids = 2.65
Hydrometer type = 152H
Hydrometer effective depth equation: L = 16.294964 **-** 0.164 **x Rm**

Elapsed Time (min.)	Temp. (deg. C.)	Actual Reading	Corrected Reading	K	Rm	Eff. Depth	Diameter (mm.)	Percent Finer
2.00	19.0	48.0	39.8	0.0138	48.0	8.4	0.0284	55.8
5.00	19.0	33.0	24.8	0.0138	33.0	10.9	0.0204	34.8
15.00	19.0	28.0	19.8	0.0138	28.0	11.7	0.0122	27.7
30.00	19.0	24.0	15.8	0.0138	24.0	12.4	0.0089	22.1
60.00	19.0	22.0	13.8	0.0138	22.0	12.7	0.0064	19.3
250.00	19.0	17.0	8.8	0.0138	17.0	13.5	0.0032	12.3
1440.00	19.0	14.0	5.8	0.0138	14.0	14.0	0.0014	8.1

Pace Analytical Services, Inc.

Fractional Components

Cobbles	Gravel			Sand				Fines		
	Coarse	Fine	Total	Coarse	Medium	Fine	Total	Silt	Clay	Total
0	0	0	0	0	1	17	18	65	17	82

D_5	D_{10}	D_{15}	D_{20}	D_{30}	D_{40}	D_{50}	D_{60}	D_{80}	D_{85}	D_{90}	D_{95}
	0.0023	0.0041	0.0071	0.0173	0.0224	0.0260	0.0305	0.0673	0.0833	0.0998	0.1290

Fineness Modulus	C_u	C_c
0.06	13.07	4.20

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10366077

Work order number: 3000017136 Report date (mm/dd/yyyy): 10/26/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.	Is there a chain of custody (COC) with the report?		☒	☐	☐	
b.	Is there a sample condition form with the report?		☒	☐	☐	
c.	Were there samples requiring preservation?		☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.	Were samples received in the correct containers?		☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.	Were samples received with adequate holding time for sample prep for all requested analyses?		☒	☐	☐	
f.	Are there notes about sample condition or holding time issues on the COC? Explain impact.		☐	☒	☐	
g.	Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.		☐	☒	☐	

2. Calibration

Question		Yes	No	N/A	Comments
a.	Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact.	☐	☒	☐	

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☐	☒	

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☐	☒	☐	
b.	Are the lab recovery limits specified on the report?		☐	☐	☒	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☐	☐	☒	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☐	☒	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	

	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question				Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.			☒	☐	☐	
	i.	Have the required matrix spikes been prepared and reported?		☒	☐	☐	Batch QC was reported with the mercury samples in this SDG.
	ii.	If no, is there an explanation in the report as to why?		☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?		☐	☐	☒	
	iv.	Are the lab limits specified on the report?		☒	☐	☐	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?		☒	☐	☐	
	vi.	Are there compounds outside the lab limits?		☐	☒	☐	
		1.	If yes, are the analytes above the lab limits?	☐	☐	☒	
		2.	Below the lab limits?	☐	☐	☒	
		3.	Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.			☐	☒	☐	RPDs discussed apply to MS/MSDs.
	i.	Is the RPD for the duplicate pair within the lab limits?		☒	☐	☐	
	ii.	If no, has the associated source sample been flagged?		☐	☐	☒	
c.	What is the impact of failed QC on this project?			☐	☐	☒	

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Samples BW16SR-011-0.0-0.15 and BW16SR-111-0.0-0.15 were collected as blind field duplicates and analyzed for mercury. The RPD for mercury (17.2%) was within the QC guideline of ≤ 50%.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

October 26, 2016

Nancy McDonald
Bay West
5 Empire Drive
Saint Paul, MN 55103

RE: Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Dear Nancy McDonald:
Enclosed are the analytical results for sample(s) received by the laboratory on October 12, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current, applicable TNI/NELAC standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Lori Castille
lori.castille@pacelabs.com
Project Manager

Enclosures

cc: Paul Raymaker, Bay West
Jeff Smith, Pace Analytical Services, Inc

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

CERTIFICATIONS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Minnesota Certification IDs

1700 Elm Street SE Suite 200, Minneapolis, MN 55414
525 N 8th Street, Salina, KS 67401
Alaska Certification UST-107
A2LA Certification #: 2926.01
Alaska Certification #: UST-078
Alaska Certification #MN00064
Alabama Certification #40770
Arizona Certification #: AZ-0014
Arkansas Certification #: 88-0680
California Certification #: 01155CA
Colorado Certification #Pace
Connecticut Certification #: PH-0256
EPA Region 8 Certification #: 8TMS-L
Florida/NELAP Certification #: E87605
Guam Certification #:14-008r
Georgia Certification #: 959
Georgia EPD #: Pace
Idaho Certification #: MN00064
Hawaii Certification #MN00064
Illinois Certification #: 200011
Indiana Certification#C-MN-01
Iowa Certification #: 368
Kansas Certification #: E-10167
Kentucky Dept of Envi. Protection - DW #90062
Kentucky Dept of Envi. Protection - WW #:90062
Louisiana DEQ Certification #: 3086
Louisiana DHH #: LA140001
Maine Certification #: 2013011
Maryland Certification #: 322
Michigan DEPH Certification #: 9909
Minnesota Certification #: 027-053-137
Mississippi Certification #: Pace
Montana Certification #: MT0092
Nevada Certification #: MN_00064
Nebraska Certification #: Pace
New Jersey Certification #: MN-002
New York Certification #: 11647
North Carolina Certification #: 530
North Carolina State Public Health #: 27700
North Dakota Certification #: R-036
Ohio EPA #: 4150
Ohio VAP Certification #: CL101
Oklahoma Certification #: 9507
Oregon Certification #: MN200001
Oregon Certification #: MN300001
Pennsylvania Certification #: 68-00563
Puerto Rico Certification
Saipan (CNMI) #:MP0003
South Carolina #:74003001
Texas Certification #: T104704192
Tennessee Certification #: 02818
Utah Certification #: MN000642013-4
Virginia DGS Certification #: 251
Virginia/VELAP Certification #: Pace
Washington Certification #: C486
West Virginia Certification #: 382
West Virginia DHHR #:9952C
Wisconsin Certification #: 999407970

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

SAMPLE SUMMARY

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Lab ID	Sample ID	Matrix	Date Collected	Date Received
10366077001	**BW16SR-006-0.0-0.15**	Solid	10/11/16 14:35	10/12/16 18:30
10366077002	**BW16SR-007-0.0-0.15**	Solid	10/11/16 14:40	10/12/16 18:30
10366077003	**BW16SR-008-0.0-0.15**	Solid	10/11/16 14:50	10/12/16 18:30
10366077004	**BW16SR-009-0.0-0.15**	Solid	10/11/16 15:00	10/12/16 18:30
10366077005	**BW16SR-011-0.0-0.15**	Solid	10/11/16 15:05	10/12/16 18:30
10366077006	**BW16SR-111-0.0-0.15**	Solid	10/11/16 15:10	10/12/16 18:30
10366077007	**BW16SR-013-0.0-0.15**	Solid	10/11/16 15:15	10/12/16 18:30
10366077008	**BW16SR-014-0.0-0.15**	Solid	10/11/16 15:30	10/12/16 18:30
10366077009	**BW16SR-015-0.0-0.15**	Solid	10/11/16 15:40	10/12/16 18:30
10366077010	**BW16SR-001-0.0-0.15**	Solid	09/21/16 13:47	10/12/16 18:30
10366077011	**BW16SR-003-0.0-0.15**	Solid	09/22/16 12:34	10/12/16 18:30

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SAMPLE ANALYTE COUNT

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
10366077001	**BW16SR-006-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077002	**BW16SR-007-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077003	**BW16SR-008-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077004	**BW16SR-009-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077005	**BW16SR-011-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077006	**BW16SR-111-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077007	**BW16SR-013-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077008	**BW16SR-014-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077009	**BW16SR-015-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077010	**BW16SR-001-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M
10366077011	**BW16SR-003-0.0-0.15**	EPA 7471B	LMW	1	PASI-M
		ASTM D2974	JDL	1	PASI-M

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-006-0.0-0.15 **Lab ID: 10366077001** Collected: 10/11/16 14:35 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.086**	mg/kg	0.041	0.011	1	10/19/16 09:34	10/19/16 16:41	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**57.9**	%	0.10	0.10	1		10/25/16 10:36		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-007-0.0-0.15 **Lab ID: 10366077002** Collected: 10/11/16 14:40 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.052**	mg/kg	0.028	0.0073	1	10/19/16 09:34	10/19/16 16:43	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**38.7**	%	0.10	0.10	1		10/25/16 10:37		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-008-0.0-0.15 **Lab ID: 10366077003** Collected: 10/11/16 14:50 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.072**	mg/kg	0.032	0.0084	1	10/19/16 09:34	10/19/16 16:45	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**48.5**	%	0.10	0.10	1		10/25/16 10:37		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-009-0.0-0.15 **Lab ID: 10366077004** Collected: 10/11/16 15:00 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.033**	mg/kg	0.029	0.0074	1	10/19/16 09:34	10/19/16 16:51	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**34.4**	%	0.10	0.10	1		10/25/16 10:37		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-011-0.0-0.15 **Lab ID: 10366077005** Collected: 10/11/16 15:05 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.082**	mg/kg	0.037	0.0096	1	10/19/16 09:34	10/19/16 16:53	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**52.0**	%	0.10	0.10	1		10/25/16 10:37		

REPORT OF LABORATORY ANALYSIS

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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-111-0.0-0.15 **Lab ID: 10366077006** Collected: 10/11/16 15:10 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.069**	mg/kg	0.035	0.0091	1	10/19/16 09:34	10/19/16 16:55	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**52.4**	%	0.10	0.10	1		10/25/16 10:38		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-013-0.0-0.15 **Lab ID: 10366077007** Collected: 10/11/16 15:15 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.16**	mg/kg	0.043	0.011	1	10/19/16 09:34	10/19/16 16:58	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**58.2**	%	0.10	0.10	1		10/25/16 10:38		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-014-0.0-0.15 **Lab ID: 10366077008** Collected: 10/11/16 15:30 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.045**	mg/kg	0.034	0.0089	1	10/19/16 09:34	10/19/16 17:00	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**41.6**	%	0.10	0.10	1		10/25/16 10:38		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-015-0.0-0.15 **Lab ID: 10366077009** Collected: 10/11/16 15:40 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.047**	mg/kg	0.031	0.0082	1	10/19/16 09:34	10/19/16 17:02	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**43.7**	%	0.10	0.10	1		10/25/16 10:39		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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(612)607-1700

ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-001-0.0-0.15 **Lab ID: 10366077010** Collected: 09/21/16 13:47 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.16**	mg/kg	0.038	0.0098	1	10/19/16 09:34	10/19/16 17:04	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**52.9**	%	0.10	0.10	1		10/25/16 10:39		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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ANALYTICAL RESULTS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Sample: BW16SR-003-0.0-0.15 **Lab ID: 10366077011** Collected: 09/22/16 12:34 Received: 10/12/16 18:30 Matrix: Solid

Results reported on a "dry weight" basis and are adjusted for percent moisture, sample size and any dilutions.

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
7471B Mercury	Analytical Method: EPA 7471B Preparation Method: EPA 7471B								
Mercury	**0.10**	mg/kg	0.042	0.011	1	10/19/16 09:34	10/19/16 17:06	7439-97-6	
Dry Weight	Analytical Method: ASTM D2974								
Percent Moisture	**58.2**	%	0.10	0.10	1		10/25/16 10:39		

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

QC Batch: 441316 Analysis Method: EPA 7471B
QC Batch Method: EPA 7471B Analysis Description: 7471B Mercury Solids
Associated Lab Samples: 10366077001, 10366077002, 10366077003, 10366077004, 10366077005, 10366077006, 10366077007, 10366077008, 10366077009, 10366077010, 10366077011

METHOD BLANK: 2402428 Matrix: Solid
Associated Lab Samples: 10366077001, 10366077002, 10366077003, 10366077004, 10366077005, 10366077006, 10366077007, 10366077008, 10366077009, 10366077010, 10366077011

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/kg	ND	0.017	0.0045	10/19/16 16:09	

LABORATORY CONTROL SAMPLE: 2402429

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/kg	.5	0.52	104	80-120	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 2402430 2402431

Parameter	Units	10366072001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/kg	ND	.53	.46	0.58	0.48	107	99	75-125	20	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

QC Batch: 443074 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 10366077001, 10366077002, 10366077003, 10366077004, 10366077005, 10366077006, 10366077007, 10366077008, 10366077009, 10366077010, 10366077011

SAMPLE DUPLICATE: 2414709

Parameter	Units	10367206003 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	15.1	15.5	3	30	

SAMPLE DUPLICATE: 2414743

Parameter	Units	10366077001 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	57.9	58.8	1	30	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, LLC
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QUALIFIERS

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-M Pace Analytical Services - Minneapolis

Pace Analytical Services, LLC
1700 Elm Street - Suite 200
Minneapolis, MN 55414
(612)607-1700

QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: J160139 SLR Sediment AOCs
Pace Project No.: 10366077

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
10366077001	**BW16SR-006-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077002	**BW16SR-007-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077003	**BW16SR-008-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077004	**BW16SR-009-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077005	**BW16SR-011-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077006	**BW16SR-111-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077007	**BW16SR-013-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077008	**BW16SR-014-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077009	**BW16SR-015-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077010	**BW16SR-001-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077011	**BW16SR-003-0.0-0.15**	EPA 7471B	441316	EPA 7471B	442057
10366077001	**BW16SR-006-0.0-0.15**	ASTM D2974	443074		
10366077002	**BW16SR-007-0.0-0.15**	ASTM D2974	443074		
10366077003	**BW16SR-008-0.0-0.15**	ASTM D2974	443074		
10366077004	**BW16SR-009-0.0-0.15**	ASTM D2974	443074		
10366077005	**BW16SR-011-0.0-0.15**	ASTM D2974	443074		
10366077006	**BW16SR-111-0.0-0.15**	ASTM D2974	443074		
10366077007	**BW16SR-013-0.0-0.15**	ASTM D2974	443074		
10366077008	**BW16SR-014-0.0-0.15**	ASTM D2974	443074		
10366077009	**BW16SR-015-0.0-0.15**	ASTM D2974	443074		
10366077010	**BW16SR-001-0.0-0.15**	ASTM D2974	443074		
10366077011	**BW16SR-003-0.0-0.15**	ASTM D2974	443074		

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

~~10365863~~ 10366077

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Nancy McDonald	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103		Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-SR-4
Email To: nmcdonald@baywest.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 651-291-3483	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (EPA 7471B)	% Moisture	Comments
Ex.	*BW15MLW-005*	*BW14MLW-005-0-0.15*	*SO*	*G*	*3/12/15*	*1204*													
1	BW16SR-006	BW16SR-006-0.0-0.15	SO	G	10/11/16	1435	3	3								1	1	1	001
2	BW16SR-007	BW16SR-007-0.0-0.15	SO	G	10/11/16	1440	3	3								1	1	1	002
3	BW16SR-008	BW16SR-008-0.0-0.15	SO	G	10/11/16	1450	3	3								1	1	1	003
4	BW16SR-009	BW16SR-009-0.0-0.15	SO	G	10/11/16	1500	3	3								1	1	1	004
5	BW16SR-011	BW16SR-011-0.0-0.15	SO	G	10/11/16	1505	3	3								1	1	1	005
6	BW16SR-011	BW16SR-111-0.0-0.15	SO	G	10/11/16	1510	3	3								1	1	1	006
7	BW16SR-013	BW16SR-013-0.0-0.15	SO	G	10/11/16	1515	3	3								1	1	1	007
8	BW16SR-014	BW16SR-014-0.0-0.15	SO	G	10/11/16	1530	3	3								1	1	1	S. Dam Wall 008
9	BW16SR-015	BW16SR-015-0.0-0.15	SO	G	10/11/16	1540	3	3								1	1	1	Channel 009
10	BW16SR-001	BW16SR-001-0.0-0.15	SO	G	9/21/16	1347	2	2								1	1	0	Take moisture from dioxins/furans jar 010
11	BW16SR-003	BW16SR-003-0.0-0.15	SO	G	9/22/16	1234	2	2								1	1	0	Take moisture from dioxins/furans jar 011
12																			

Valid Matrix Codes: Drinking Water DW; Waste Water W; Product WW; Soil/Solid P; Oil SO; Wipe OL; Air WP; Tissue AR; Other TS; OT

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME
	Chris Musson / Bay West	10/12/16	1445	Lan Tod / Pace	10/13/16	14:45
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16	Lance Flood	10/13/16	1600		10/12/16	1600
		10/12/16	1830		10/12/16	1830

SAMPLE CONDITIONS			
Temp (°C)	Received on ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
1.9	Y	N	Y
3.3			
1.7			

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Chris Musson

SIGNATURE of SAMPLER: Chris Musson DATE Signed (MM/DD/YY): 10/12/16

Pace Analytical	Document Name: **Sample Condition Upon Receipt Form**	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: **F-MN-L-213-rev.17**	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: Bay West LLC

Project #: WO# : 10366077

10366077

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ SpeeDee ☐ Other:_____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☑ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other:_____ **Temp Blank?** ☑ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ 151401164 ☐ B88A912167504 ☐ B88A0143310098 **Type of Ice:** ☑ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 3.3, 1.5 **Cooler Temp Corrected (°C):** 3.5, 1.7 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** BC 10/12/16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☑ No

Did samples originate from a foreign source (internationally including Hawaii and Puerto Rico)? ☐ Yes ☑ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☑ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: WT/ST	☑ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation have been checked? All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide) Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☑ Yes ☑ Yes ☑ Yes	☐ No ☐ No ☐ No	☐ N/A ☐ N/A ☐ N/A	13. ☑ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample # 1-5 BC 10/12/16 Initial when completed:_____ Lot # of added preservative:_____
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):_____				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

Dioxin on separate WO

Project Manager Review: Lori Cattle **Date:** 10/14/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155-4194

Laboratory Data Review Checklist

Doc Type: Data Review

Instructions: The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. This checklist should be used in conjunction with the *Laboratory Data Checklist Guidance* (p-eao-11a): http://www.pca.state.mn.us/index.php/view-document.html?gid=16113. Also see the MPCA Laboratory Quality Control (QC) and Data Policy: http://www.pca.state.mn.us/index.php/view-document.html?gid=16288.

Project Information

Project name: SLR Sediments AOCs – Scanlon Reservoir Laboratory: Pace - 10367136

Work order number: 3000017136 Report date (mm/dd/yyyy): 11/04/2016

1. Preservation

For help with this section on holding times, containers and preservatives, refer to the Minnesota Department of Health's website at: http://www.health.state.mn.us/divs/phl/environmental/handbook/internet/envhandbook.html.

Questions			Yes	No	N/A	Comments
a.		Is there a chain of custody (COC) with the report?	☒	☐	☐	COC includes samples for Scanlon Reservoir, Thomson and Boulder Lake. This data review checklist only applies to Scanlon Reservoir samples.
b.		Is there a sample condition form with the report?	☒	☐	☐	
c.		Were there samples requiring preservation?	☐	☒	☐	
	i.	If so, were they properly preserved?	☐	☐	☒	
	ii.	Were they received on ice?	☒	☐	☐	
d.		Were samples received in the correct containers?	☒	☐	☐	
	i.	Was there enough sample volume/weight to complete all requested analyses?	☒	☐	☐	
	ii.	Was there enough extra sample collected to complete method required batch QC?	☒	☐	☐	
e.		Were samples received with adequate holding time for sample prep for all requested analyses?	☒	☐	☐	
f.		Are there notes about sample condition or holding time issues on the COC? Explain impact.	☐	☒	☐	
g.		Is there narration or data qualifiers within the report about sample condition or holding time issues? Explain impact.	☐	☒	☐	

2. Calibration

Question			Yes	No	N/A	Comments
a.		Do the report narrative or data qualifiers indicate	☒	☐	☐	

	calibration problems for any analyses? If yes, explain the data impact.				

3. Blanks

Question			Yes	No	N/A	Comments
a.	Do any of the analyses contain samples for field or trip blanks?		☐	☒	☐	
	i.	If yes, are there target analytes present above the reporting limit?	☐	☐	☒	
	ii.	If yes, are the same compounds also present in the samples? Explain possible impact.	☐	☐	☒	
b.	Do method blanks for any analyses contain target analytes above the reporting limit?		☐	☒	☐	Low-level concentrations of Total TCDD, 1,2,3,4,6,7,8-HpCDD, Total HpCDD, and OCDD were detected in the method blank 52558.
	i.	If yes, are the same compounds present in the samples?	☐	☐	☒	
	ii.	Is the amount of target analyte in the blank more than 1/10th of that in the sample(s)? Explain the possible impact on sample results.	☐	☒	☐	All sample results were > 10x the blank concentrations.

4. Surrogates

Question			Yes	No	N/A	Comments
a.	Are there organic analyses that contain surrogate compounds?		☒	☐	☐	Dioxins/furans have internal standards instead of surrogates.
b.	Are the lab recovery limits specified on the report?		☒	☐	☐	
	i.	Do the lab limits seem reasonable when compared with the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
c.	Are there surrogates outside lab limits? (These should have a data qualifier)		☐	☒	☐	
	i.	If yes, are the surrogates above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	
	iii.	Explain what this could mean for the affected samples.	☐	☐	☒	

5. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Question			Yes	No	N/A	Comments
a.	Are there LCS/LCSD samples present for the reported analyses? (An LCS alone is acceptable if there is an Matrix Spike/Matrix Spike Duplicate [MS/MSD] or sample/sample dup for precision.)		☒	☐	☐	
	i.	If so, do the lab limits seem reasonable compared to the suggested guidelines in the MPCA QC Policy?	☒	☐	☐	
b.	Are there LCS/LCSD compounds outside lab limits? (These should have a data qualifier.)		☐	☒	☐	
	i.	If yes, are the analytes above the lab limits?	☐	☐	☒	
	ii.	Below the lab limits?	☐	☐	☒	

	iii.	Are all samples in the preparation batch also flagged for the same analyte(s)?	☐	☐	☒	
	iv.	Explain what this could mean for the affected samples.	☐	☐	☒	

6. Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/Dup)

Question				Yes	No	N/A	Comments
a.	Do the analytical methods used require an MS and/or MSD? If no, skip to 6.b.			☐	☒	☐	
	i.	Have the required matrix spikes been prepared and reported?		☐	☐	☒	
	ii.	If no, is there and explanation in the report as to why?		☐	☐	☒	
	iii.	Did the lab process an alternate spiked sample (such as LCSD) instead?		☐	☐	☒	
	iv.	Are the lab limits specified on the report?		☐	☐	☒	
	v.	Do the limits seem reasonable when compared to the suggested guidelines in the MPCA QC Policy?		☐	☐	☒	
	vi.	Are there compounds outside the lab limits?		☐	☐	☒	
		1.	If yes, are the analytes above the lab limits?	☐	☐	☒	
		2.	Below the lab limits?	☐	☐	☒	
		3.	Is the source sample also flagged for compounds outside lab limits?	☐	☐	☒	
b.	Is a sample duplicate reported for the analytical method(s)? If no, skip to 6.c.			☐	☐	☒	
	i.	Is the RPD for the duplicate pair within the lab limits?		☐	☐	☒	
	ii.	If no, has the associated source sample been flagged?		☐	☐	☒	
c.	What is the impact of failed QC on this project?			☐	☐	☒	

7. Method Detection Limits/Report Limits

Question		Yes	No	N/A	Comments
a.	Are reporting and/or method detection limits clearly listed on the report for all analyses? (may also be called quantitation limits)	☒	☐	☐	

Additional comments on report:

(1) Interfering substances impacted the determinations of PCDF congeners; the affected values were flagged "I" where incorrect isotope ratios were obtained or "P" where diphenyl ethers were present. All results flagged “I” or “P” were qualified “J” as estimated by the reviewer. The laboratory flagged concentrations > the calibration range “E”. These results were qualified “J” as estimated by the reviewer. Concentrations below the calibration range were flagged “J” as estimated by the laboratory.

(2) Level II reports were reviewed, so calibrations and raw data were not reviewed.

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

www.pacelabs.com

Report Prepared for:

Nancy McDonald
Bay West, Inc.
5 Empire Drive
Saint Paul MN 55103

REPORT OF LABORATORY ANALYSIS FOR PCDD/PCDF

Report Prepared Date:
November 3, 2016

Report Information:

Pace Project #: 10367136
Sample Receipt Date: 10/21/2016
Client Project #: J160139 SLR Sediment AOCs
Client Sub PO #: 108002
State Cert #: 027-053-137

Invoicing & Reporting Options:

The report provided has been invoiced as a Level 2 PCDD/PCDF Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

This report has been reviewed by:

November 04, 2016

Carolynne Trout, Project Manager
(612) 607-6351
(612) 607-6444 (fax)
Carolynne.Trout@pacelabs.com

Report of Laboratory Analysis

Pace Analytical Services, Inc.
1700 Elm Street
Minneapolis, MN 55414
Phone: 612.607.1700
Fax: 612.607.6444

DISCUSSION

This report presents the results from the analyses performed on seven samples submitted by a representative of BayWest, Inc. The samples were analyzed for the presence or absence of polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs) using a modified version of USEPA Method 8290. The reporting limits were based on signal-to-noise measurements. Estimated Maximum Possible Concentration (EMPC) values were treated as positives in the toxic equivalence calculations.

Second column confirmation analyses of 2,3,7,8-TCDF values obtained from the primary (DB5-MS) column are performed only when specifically requested for a project and only when the values are above the concentration of the lowest calibration standard. Typical resolution for this isomer using the DB5-MS column ranges from 25-30%.

The recoveries of the isotopically-labeled PCDD/PCDF internal standards in the sample extracts ranged from 49-99%. All of the labeled standard recoveries obtained for this project were within the 40-135% target range specified in Method 8290. Also, since the quantification of the native 2,3,7,8-substituted congeners was based on isotope dilution, the data were automatically corrected for variation in recovery and accurate values were obtained.

Values were flagged "I" where incorrect isotope ratios were obtained and "P" where diphenylethers were present at the elution times of PCDFs. Concentrations below the calibration range were flagged "J" and should be regarded as estimates. Levels above the calibration range were flagged "E" and should be regarded as estimated concentrations.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to contain trace levels of selected congeners. These levels were below the calibration range of the method. The levels reported for the affected congeners in the field samples were higher than the corresponding blank levels by one or more orders of magnitude. These results indicate that the sample processing steps did not contribute significantly to the levels reported for the field samples.

A laboratory spike sample was also prepared with the sample batch using clean reference matrix that had been fortified with native standard materials. The results show that the spiked native compounds were recovered at 85-114%. These values were within the target range for this method. Matrix spikes were prepared using sample material from a separate project. Results are available upon request.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Montana	92
Alaska	MN00064	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN_00064_200
Arkansas	88-0680	New Jersey (NE	MN002
California	01155CA	New York (NEL	11647
Colorado	MN00064	North Carolina	27700
Connecticut	PH-0256	North Dakota	R-036
EPA Region 8	8TMS-Q	Ohio	4150
Florida (NELAP	E87605	Oklahoma	D9922
Georgia (DNR)	959	Oregon (ELAP)	MN200001-005
Guam	959	Oregon (OREL	MN300001-001
Hawaii	SLD	Pennsylvania	68-00563
Idaho	MN00064	Puerto Rico	MN00064
Illinois	200012	Saipan	MP0003
Indiana	C-MN-01	South Carolina	74003001
Indiana	C-MN-01	Tennessee	TN02818
Iowa	368	Texas	T104704192-08
Kansas	E-10167	Utah (NELAP)	MN00064
Kentucky	90062	Virginia	00251
Louisiana	03086	Washington	C755
Maine	2007029	West Virginia #	9952C
Maryland	322	West Virginia D	382
Michigan	9909	Wisconsin	999407970
Minnesota	027-053-137	Wyoming	8TMS-Q

REPORT OF LABORATORY ANALYSIS

Report No.....In-House

Appendix A

Sample Management

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:	Section D EQuIS Information:	
Company: Bay West, LLC	Report To: Mailee Garton - Great Lake Environmental Center	Attention: Accounts Payable	Facility_Name: St. Louis River Sediment Areas of Concern	Page 1 of 1
Address: 5 Empire Drive	Copy To: Paul Raymaker - Bay West	Company Name: Bay West, LLC	Facility_Code: St Louis River Sed	
St. Paul, MN 55103	Nancy McDonald - Bay West	Address: 5 Empire Drive	Facility_ID: 547023	COC# SLR-ToxBio-02
Email To: mgarton@glec.com	Purchase Order No.: 108002	Lab Quote Reference: 3000017136	Subfacility_code:	
Phone: 231-941-2230	Project Name: SLR Sediment AOCs	Lab Project Manager: Oyeyemi Odujole		Site Location STATE: MN
Requested Due Date/TAT: Standard	Project Number: J160139			

Valid Matrix Codes: Drinking Water DW; Water W; Waste Water WW; Product P; Soil/Solid SO; Oil OL; Wipe WP; Air AR; Tissue TS; Other OT

ITEM #	Section E Required Client Information: Sample Location ID (sys_loc_code)	Sample ID (sys_sample_code)	MATRIX CODE	SAMPLE TYPE (G=GRAB C=COMP)	Collection DATE	Collection Time	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis: Dioxins and furans (SW-846 8290A)	Mercury (7471B)	% Moisture	TOC (SW-846 9060A Quad Burn)	Comments
Ex	BW16MLW-005	BW14MLW-005-0-0.15	SO	G		1204														
1	BW16SR-004	BW16SR-004-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	001
2	BW16SR-016	BW16SR-016-0.15-0.60	SO	G	10/20/16	10:00	4									x	x	x	x	002
3	BW16TR-008	BW16TR-008-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	003
4	BW16TR-013	BW16TR-013-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	004
5	BW16TR-017	BW16TR-017-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	005
6	BW16TR-018	BW16TR-018-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	006
7	BW16BLR-001	BW16BLR-001-0.0-0.15	SO	G	10/20/16	10:00	4									x	x	x	x	007
8																				
9																				
10																				
11																				
12																				

* 5 pm 10/20/16

Separate Cooler

Ref: pace.tox lab
Dep:
Date: 20Oct16
Wgt: 5.00 LBS
DV: 0.00
SHIPPING: 6.13
SPECIAL: 0.00
HANDLING: 0.00
TOTAL: 6.13
Svcs: PRIORITY OVERNIGHT
TRCK: 9802 5316 5172

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS
Reference Pace Subcontractor Order Form signed by Pace on 9/16/16	Mailee Garton/GLEC	10/20/16	14:00	PACE	10/21/16	9:45	0.7 / 0.6

Temp (°C)	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
	Y	N	Y

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Mailee Garton

SIGNATURE of SAMPLER: Mailee Garton DATE Signed (MM/DD/YY): 10/20/16

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 02Aug2016 Page 1 of 2
	Document No.: F-MN-L-213-rev.17	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt **Client Name:** Bay West LLC **Project #:** WO#: 10367136

Courier: ☑ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ SpeeDee ☐ Other:

Tracking Number: 9802 5318 5161 9802 5318 5172

Custody Seal on Cooler/Box Present? ☑ Yes ☐ No **Seals Intact?** ☑ Yes ☐ No **Optional:** Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☑ Yes ☐ No

Thermometer Used: ☑ 151401163 ☐ B88A912167504 ☐ 151401164 ☐ B88A0143310098 **Type of Ice:** ☑ Wet ☐ Blue ☐ None ☐ Samples on ice, cooling process has begun

Cooler Temp Read (°C): 0.6, 0.7 **Cooler Temp Corrected (°C):** 0.8, 0.9 **Biological Tissue Frozen?** ☐ Yes ☐ No ☑ N/A

Temp should be above freezing to 6°C **Correction Factor:** +0.2 **Date and Initials of Person Examining Contents:** BC 10/21/16

USDA Regulated Soil (☐ N/A, water sample)

Did samples originate in a quarantine zone within the United States: AL, AR, AZ, CA, FL, GA, ID, LA. MS, NC, NM, NY, OK, OR, SC, TN, TX or VA (check maps)? ☐ Yes ☑ No

Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)? ☐ Yes ☑ No

If Yes to either question, fill out a Regulated Soil Checklist (F-MN-Q-338) and include with SCUR/COC paperwork.

				COMMENTS:
Chain of Custody Present?	☑ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☑ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☑ Yes	☐ No	☐ N/A	3.
Sampler Name and/or Signature on COC?	☑ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☑ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☑ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☑ No	☐ N/A	7.
Sufficient Volume?	☑ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☑ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☑ Yes	☐ No	☐ N/A	
Containers Intact?	☑ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☑ N/A	11. Note if sediment is visible in the dissolved container
Sample Labels Match COC?	☑ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: SL				
All containers needing acid/base preservation have been checked?	☐ Yes	☐ No	☑ N/A	13. ☐ HNO_3 ☐ H_2SO_4 ☐ NaOH ☐ HCl Sample #
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO_3, H_2SO_4, HCl<2; NaOH >9 Sulfide, NaOH>12 Cyanide)	☐ Yes	☐ No	☑ N/A	
Exceptions: VOA, Coliform, TOC, Oil and Grease, DRO/8015 (water) DOC	☐ Yes	☐ No	☑ N/A	Initial when completed: Lot # of added preservative:
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☑ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☑ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☑ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

Project Manager Review: Carolynne Trout **Date:** 10/24/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers).

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Reporting Flags

A = Reporting Limit based on signal to noise

B = Less than 10x higher than method blank level

C = Result obtained from confirmation analysis

D = Result obtained from analysis of diluted sample

E = Exceeds calibration range

I = Interference present

J = Estimated value

Nn = Value obtained from additional analysis

P = PCDE Interference

R = Recovery outside target range

S = Peak saturated

U = Analyte not detected

V = Result verified by confirmation analysis

X = %D Exceeds limits

Y = Calculated using average of daily RFs

* = See Discussion

Appendix B

Sample Analysis Summary

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-004-0.0-0.15		
Lab Sample ID	10367136001		
Filename	F161101B_11		
Injected By	SMT		
Total Amount Extracted	18.6 g	Matrix	Solid
% Moisture	58.7	Dilution	NA
Dry Weight Extracted	7.68 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 21:43

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	15.0	-----	0.29		2,3,7,8-TCDF-13C	2.00	80
Total TCDF	43.0	-----	0.29		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	80
2,3,7,8-TCDD	3.5	-----	0.21		2,3,4,7,8-PeCDF-13C	2.00	73
Total TCDD	22.0	-----	0.21		1,2,3,7,8-PeCDD-13C	2.00	79
					1,2,3,4,7,8-HxCDF-13C	2.00	93
1,2,3,7,8-PeCDF	-----	1.2	0.13	IJ	1,2,3,6,7,8-HxCDF-13C	2.00	77
2,3,4,7,8-PeCDF	3.6	-----	0.21	J	2,3,4,6,7,8-HxCDF-13C	2.00	86
Total PeCDF	58.0	-----	0.17		1,2,3,7,8,9-HxCDF-13C	2.00	81
					1,2,3,4,7,8-HxCDD-13C	2.00	80
1,2,3,7,8-PeCDD	4.2	-----	0.22	J	1,2,3,6,7,8-HxCDD-13C	2.00	66
Total PeCDD	51.0	-----	0.22		1,2,3,4,6,7,8-HpCDF-13C	2.00	60
					1,2,3,4,7,8,9-HpCDF-13C	2.00	61
1,2,3,4,7,8-HxCDF	-----	15.0	4.70	P	1,2,3,4,6,7,8-HpCDD-13C	2.00	72
1,2,3,6,7,8-HxCDF	19.0	-----	0.82		OCDD-13C	4.00	67
2,3,4,6,7,8-HxCDF	7.9	-----	0.29				
1,2,3,7,8,9-HxCDF	3.8	-----	0.37	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	560.0	-----	1.60		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	7.4	-----	0.37		2,3,7,8-TCDD-37Cl4	0.20	87
1,2,3,6,7,8-HxCDD	55.0	-----	0.72				
1,2,3,7,8,9-HxCDD	16.0	-----	0.44				
Total HxCDD	350.0	-----	0.51				
1,2,3,4,6,7,8-HpCDF	870.0	-----	0.74		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	15.0	-----	0.84		Equivalence: 45 ng/Kg		
Total HpCDF	1900.0	-----	0.79		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	990.0	-----	2.40				
Total HpCDD	2000.0	-----	2.40				
OCDF	860.0	-----	0.56				
OCDD	11000.0	-----	0.39	E			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
P = PCDE Interference
E = Exceeds calibration range
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16SR-016-0.15-0.60		
Lab Sample ID	10367136002		
Filename	F161101B_12		
Injected By	SMT		
Total Amount Extracted	17.5 g	Matrix	Solid
% Moisture	44.5	Dilution	NA
Dry Weight Extracted	9.71 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 22:31

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	12.0	-----	0.70		2,3,7,8-TCDF-13C	2.00	79
Total TCDF	68.0	-----	0.70		2,3,7,8-TCDD-13C	2.00	86
					1,2,3,7,8-PeCDF-13C	2.00	74
2,3,7,8-TCDD	6.1	-----	0.34		2,3,4,7,8-PeCDF-13C	2.00	63
Total TCDD	53.0	-----	0.34		1,2,3,7,8-PeCDD-13C	2.00	70
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	-----	7.2	0.24	P	1,2,3,6,7,8-HxCDF-13C	2.00	78
2,3,4,7,8-PeCDF	17.0	-----	0.40		2,3,4,6,7,8-HxCDF-13C	2.00	85
Total PeCDF	240.0	-----	0.32		1,2,3,7,8,9-HxCDF-13C	2.00	82
					1,2,3,4,7,8-HxCDD-13C	2.00	81
1,2,3,7,8-PeCDD	23.0	-----	0.13		1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	190.0	-----	0.13		1,2,3,4,6,7,8-HpCDF-13C	2.00	62
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	72.0	-----	0.58		1,2,3,4,6,7,8-HpCDD-13C	2.00	74
1,2,3,6,7,8-HxCDF	110.0	-----	0.80		OCDD-13C	4.00	61
2,3,4,6,7,8-HxCDF	19.0	-----	0.53				
1,2,3,7,8,9-HxCDF	11.0	-----	0.66		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	2500.0	-----	0.64		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	17.0	-----	0.82		2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	100.0	-----	0.84				
1,2,3,7,8,9-HxCDD	67.0	-----	0.71				
Total HxCDD	900.0	-----	0.79				
1,2,3,4,6,7,8-HpCDF	4300.0	-----	0.37	E	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	34.0	-----	2.90		Equivalence: 130 ng/Kg		
Total HpCDF	8300.0	-----	1.70	E	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	850.0	-----	1.40				
Total HpCDD	1700.0	-----	1.40				
OCDF	2000.0	-----	0.48				
OCDD	6700.0	-----	0.28				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
P = PCDE Interference
E = Exceeds calibration range

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-008-0.0-0.15		
Lab Sample ID	10367136003		
Filename	F161101B_13		
Injected By	SMT		
Total Amount Extracted	18.2 g	Matrix	Solid
% Moisture	42.4	Dilution	NA
Dry Weight Extracted	10.5 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/01/2016 23:19

Native Isomers	**Conc** ng/Kg	**EMPC** ng/Kg	**EDL** ng/Kg		**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.74	-----	0.49	J	2,3,7,8-TCDF-13C	2.00	74
Total TCDF	2.50	-----	0.49		2,3,7,8-TCDD-13C	2.00	82
					1,2,3,7,8-PeCDF-13C	2.00	78
2,3,7,8-TCDD	ND	-----	0.54		2,3,4,7,8-PeCDF-13C	2.00	71
Total TCDD	2.20	-----	0.54		1,2,3,7,8-PeCDD-13C	2.00	74
					1,2,3,4,7,8-HxCDF-13C	2.00	84
1,2,3,7,8-PeCDF	ND	-----	0.44		1,2,3,6,7,8-HxCDF-13C	2.00	76
2,3,4,7,8-PeCDF	0.97	-----	0.35	J	2,3,4,6,7,8-HxCDF-13C	2.00	83
Total PeCDF	9.40	-----	0.40		1,2,3,7,8,9-HxCDF-13C	2.00	77
					1,2,3,4,7,8-HxCDD-13C	2.00	79
1,2,3,7,8-PeCDD	0.35	-----	0.31	J	1,2,3,6,7,8-HxCDD-13C	2.00	59
Total PeCDD	26.00	-----	0.31		1,2,3,4,6,7,8-HpCDF-13C	2.00	58
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	3.30	-----	0.51	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66
1,2,3,6,7,8-HxCDF	3.30	-----	0.26	J	OCDD-13C	4.00	55
2,3,4,6,7,8-HxCDF	2.20	-----	0.28	J			
1,2,3,7,8,9-HxCDF	-----	0.82	0.25	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	150.00	-----	0.32		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.50		2,3,7,8-TCDD-37Cl4	0.20	78
1,2,3,6,7,8-HxCDD	75.00	-----	0.60				
1,2,3,7,8,9-HxCDD	26.00	-----	0.37				
Total HxCDD	520.00	-----	0.49				
1,2,3,4,6,7,8-HpCDF	260.00	-----	0.33		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	2.00	-----	0.31	J	Equivalence: 16 ng/Kg		
Total HpCDF	470.00	-----	0.32		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	91.00	-----	0.39				
Total HpCDD	190.00	-----	0.39				
OCDF	87.00	-----	0.20				
OCDD	320.00	-----	0.21				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-008-0.0-0.15		
Lab Sample ID	10367136003		
Filename	F161102A_11		
Injected By	SMT		
Total Amount Extracted	18.2 g	Matrix	Solid
% Moisture	42.4	Dilution	NA
Dry Weight Extracted	10.5 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_19 & F161102A_15	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 12:58

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	-----	0.52	0.130	IJ	2,3,7,8-TCDF-13C	2.00	78
Total TCDF	2.60	-----	0.130		2,3,7,8-TCDD-13C	2.00	83
					1,2,3,7,8-PeCDF-13C	2.00	83
2,3,7,8-TCDD	ND	-----	0.130		2,3,4,7,8-PeCDF-13C	2.00	76
Total TCDD	3.80	-----	0.130		1,2,3,7,8-PeCDD-13C	2.00	78
					1,2,3,4,7,8-HxCDF-13C	2.00	81
1,2,3,7,8-PeCDF	0.30	-----	0.160	J	1,2,3,6,7,8-HxCDF-13C	2.00	81
2,3,4,7,8-PeCDF	-----	0.96	0.079	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	83
Total PeCDF	8.90	-----	0.120		1,2,3,7,8,9-HxCDF-13C	2.00	76
					1,2,3,4,7,8-HxCDD-13C	2.00	75
1,2,3,7,8-PeCDD	ND	-----	0.380		1,2,3,6,7,8-HxCDD-13C	2.00	63
Total PeCDD	27.00	-----	0.380		1,2,3,4,6,7,8-HpCDF-13C	2.00	56
					1,2,3,4,7,8,9-HpCDF-13C	2.00	55
1,2,3,4,7,8-HxCDF	3.90	-----	1.600	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	62
1,2,3,6,7,8-HxCDF	3.60	-----	0.370	J	OCDD-13C	4.00	49
2,3,4,6,7,8-HxCDF	2.20	-----	1.500	J			
1,2,3,7,8,9-HxCDF	1.10	-----	0.130	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	150.00	-----	0.900		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.720		2,3,7,8-TCDD-37Cl4	0.20	81
1,2,3,6,7,8-HxCDD	72.00	-----	0.710				
1,2,3,7,8,9-HxCDD	29.00	-----	0.700				
Total HxCDD	530.00	-----	0.710				
1,2,3,4,6,7,8-HpCDF	260.00	-----	0.570		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	-----	1.60	0.690	IJ	Equivalence: 15 ng/Kg		
Total HpCDF	470.00	-----	0.630		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	94.00	-----	0.720				
Total HpCDD	190.00	-----	0.720				
OCDF	86.00	-----	0.530				
OCDD	310.00	-----	0.380				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-013-0.0-0.15		
Lab Sample ID	10367136004		
Filename	F161101B_14		
Injected By	SMT		
Total Amount Extracted	18.9 g	Matrix	Solid
% Moisture	53.5	Dilution	NA
Dry Weight Extracted	8.79 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 00:07

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	1.40	-----	0.42		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	5.60	-----	0.42		2,3,7,8-TCDD-13C	2.00	83
					1,2,3,7,8-PeCDF-13C	2.00	79
2,3,7,8-TCDD	ND	-----	0.31		2,3,4,7,8-PeCDF-13C	2.00	74
Total TCDD	6.40	-----	0.31		1,2,3,7,8-PeCDD-13C	2.00	74
					1,2,3,4,7,8-HxCDF-13C	2.00	83
1,2,3,7,8-PeCDF	0.78	-----	0.32	J	1,2,3,6,7,8-HxCDF-13C	2.00	79
2,3,4,7,8-PeCDF	1.20	-----	0.39	J	2,3,4,6,7,8-HxCDF-13C	2.00	85
Total PeCDF	16.00	-----	0.35		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	75
1,2,3,7,8-PeCDD	-----	0.80	0.53	IJ	1,2,3,6,7,8-HxCDD-13C	2.00	62
Total PeCDD	9.70	-----	0.53		1,2,3,4,6,7,8-HpCDF-13C	2.00	58
					1,2,3,4,7,8,9-HpCDF-13C	2.00	59
1,2,3,4,7,8-HxCDF	4.00	-----	0.98	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	66
1,2,3,6,7,8-HxCDF	8.90	-----	0.36		OCDD-13C	4.00	57
2,3,4,6,7,8-HxCDF	2.80	-----	0.36	J			
1,2,3,7,8,9-HxCDF	-----	0.86	0.65	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	190.00	-----	0.59		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.73	-----	0.29	J	2,3,7,8-TCDD-37Cl4	0.20	77
1,2,3,6,7,8-HxCDD	6.10	-----	0.26				
1,2,3,7,8,9-HxCDD	2.30	-----	0.34	J			
Total HxCDD	55.00	-----	0.30				
1,2,3,4,6,7,8-HpCDF	320.00	-----	0.53		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	2.30	-----	0.50	J	Equivalence: 8.4 ng/Kg		
Total HpCDF	600.00	-----	0.51		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	85.00	-----	0.83				
Total HpCDD	190.00	-----	0.83				
OCDF	160.00	-----	0.19				
OCDD	1100.00	-----	0.28				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

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NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-017-0.0-0.15		
Lab Sample ID	10367136005		
Filename	F161101B_15		
Injected By	SMT		
Total Amount Extracted	18.8 g	Matrix	Solid
% Moisture	58.9	Dilution	NA
Dry Weight Extracted	7.73 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 00:56

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	2.10	-----	0.30		2,3,7,8-TCDF-13C	2.00	79
Total TCDF	9.70	-----	0.30		2,3,7,8-TCDD-13C	2.00	89
					1,2,3,7,8-PeCDF-13C	2.00	85
2,3,7,8-TCDD	-----	0.35	0.20	IJ	2,3,4,7,8-PeCDF-13C	2.00	81
Total TCDD	5.10	-----	0.20		1,2,3,7,8-PeCDD-13C	2.00	83
					1,2,3,4,7,8-HxCDF-13C	2.00	89
1,2,3,7,8-PeCDF	0.57	-----	0.30	J	1,2,3,6,7,8-HxCDF-13C	2.00	85
2,3,4,7,8-PeCDF	0.84	-----	0.22	J	2,3,4,6,7,8-HxCDF-13C	2.00	91
Total PeCDF	14.00	-----	0.26		1,2,3,7,8,9-HxCDF-13C	2.00	85
					1,2,3,4,7,8-HxCDD-13C	2.00	81
1,2,3,7,8-PeCDD	0.65	-----	0.37	J	1,2,3,6,7,8-HxCDD-13C	2.00	65
Total PeCDD	12.00	-----	0.37		1,2,3,4,6,7,8-HpCDF-13C	2.00	60
					1,2,3,4,7,8,9-HpCDF-13C	2.00	62
1,2,3,4,7,8-HxCDF	2.80	-----	0.41	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	69
1,2,3,6,7,8-HxCDF	4.40	-----	0.35	J	OCDD-13C	4.00	59
2,3,4,6,7,8-HxCDF	1.80	-----	0.50	J			
1,2,3,7,8,9-HxCDF	0.88	-----	0.39	J	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	90.00	-----	0.41		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.67	-----	0.33	J	2,3,7,8-TCDD-37Cl4	0.20	82
1,2,3,6,7,8-HxCDD	5.20	-----	0.30	J			
1,2,3,7,8,9-HxCDD	2.30	-----	0.26	J			
Total HxCDD	47.00	-----	0.30				
1,2,3,4,6,7,8-HpCDF	140.00	-----	0.48		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	1.80	-----	0.33	J	Equivalence: 6.1 ng/Kg		
Total HpCDF	280.00	-----	0.40		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	95.00	-----	0.66				
Total HpCDD	220.00	-----	0.66				
OCDF	100.00	-----	0.50				
OCDD	1300.00	-----	0.30				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
Fax: 612- 607-6444

Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16TR-018-0.0-0.15		
Lab Sample ID	10367136006		
Filename	F161101B_16		
Injected By	SMT		
Total Amount Extracted	18.6 g	Matrix	Solid
% Moisture	49.9	Dilution	NA
Dry Weight Extracted	9.32 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 01:44

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	1.20	-----	0.26		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	5.00	-----	0.26		2,3,7,8-TCDD-13C	2.00	83
					1,2,3,7,8-PeCDF-13C	2.00	78
2,3,7,8-TCDD	-----	0.30	0.27	IJ	2,3,4,7,8-PeCDF-13C	2.00	71
Total TCDD	5.60	-----	0.27		1,2,3,7,8-PeCDD-13C	2.00	76
					1,2,3,4,7,8-HxCDF-13C	2.00	85
1,2,3,7,8-PeCDF	0.49	-----	0.29	J	1,2,3,6,7,8-HxCDF-13C	2.00	74
2,3,4,7,8-PeCDF	0.91	-----	0.25	J	2,3,4,6,7,8-HxCDF-13C	2.00	83
Total PeCDF	12.00	-----	0.27		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	72
1,2,3,7,8-PeCDD	-----	0.62	0.26	IJ	1,2,3,6,7,8-HxCDD-13C	2.00	61
Total PeCDD	8.70	-----	0.26		1,2,3,4,6,7,8-HpCDF-13C	2.00	55
					1,2,3,4,7,8,9-HpCDF-13C	2.00	55
1,2,3,4,7,8-HxCDF	2.60	-----	0.42	J	1,2,3,4,6,7,8-HpCDD-13C	2.00	64
1,2,3,6,7,8-HxCDF	5.60	-----	0.60		OCDD-13C	4.00	53
2,3,4,6,7,8-HxCDF	1.70	-----	0.50	J			
1,2,3,7,8,9-HxCDF	-----	0.62	0.35	IJ	1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	140.00	-----	0.47		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	0.53	-----	0.26	J	2,3,7,8-TCDD-37Cl4	0.20	76
1,2,3,6,7,8-HxCDD	5.30	-----	0.27	J			
1,2,3,7,8,9-HxCDD	2.20	-----	0.30	J			
Total HxCDD	44.00	-----	0.28				
1,2,3,4,6,7,8-HpCDF	230.00	-----	0.32		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	1.60	-----	0.40	J	Equivalence: 6.5 ng/Kg		
Total HpCDF	440.00	-----	0.36		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	74.00	-----	0.40				
Total HpCDD	160.00	-----	0.40				
OCDF	130.00	-----	0.51				
OCDD	910.00	-----	0.38				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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EDL = Estimated Detection Limit

ND = Not Detected
NA = Not Applicable
NC = Not Calculated

Results reported on a dry weight basis and are valid to no more than 2 significant figures.
J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
Minneapolis, MN 55414

Tel: 612-607-1700
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Method 8290 Sample Analysis Results

Client - Bay West, Inc.

Client's Sample ID	BW16BLR-001-0.0-0.15		
Lab Sample ID	10367136007		
Filename	F161101B_17		
Injected By	SMT		
Total Amount Extracted	21.4 g	Matrix	Solid
% Moisture	82.6	Dilution	NA
Dry Weight Extracted	3.72 g	Collected	10/20/2016 10:00
ICAL ID	F161011	Received	10/21/2016 09:45
CCal Filename(s)	F161101B_03 & F161101B_19	Extracted	10/27/2016 16:25
Method Blank ID	BLANK-52558	Analyzed	11/02/2016 02:32

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	1.70	-----	0.59	J	2,3,7,8-TCDF-13C	2.00	87
Total TCDF	14.00	-----	0.59		2,3,7,8-TCDD-13C	2.00	94
					1,2,3,7,8-PeCDF-13C	2.00	91
2,3,7,8-TCDD	ND	-----	0.47		2,3,4,7,8-PeCDF-13C	2.00	84
Total TCDD	0.82	-----	0.47	J	1,2,3,7,8-PeCDD-13C	2.00	89
					1,2,3,4,7,8-HxCDF-13C	2.00	95
1,2,3,7,8-PeCDF	0.75	-----	0.49	J	1,2,3,6,7,8-HxCDF-13C	2.00	93
2,3,4,7,8-PeCDF	-----	0.97	0.34	IJ	2,3,4,6,7,8-HxCDF-13C	2.00	99
Total PeCDF	9.00	-----	0.41	J	1,2,3,7,8,9-HxCDF-13C	2.00	92
					1,2,3,4,7,8-HxCDD-13C	2.00	85
1,2,3,7,8-PeCDD	0.47	-----	0.43	J	1,2,3,6,7,8-HxCDD-13C	2.00	74
Total PeCDD	1.80	-----	0.43	J	1,2,3,4,6,7,8-HpCDF-13C	2.00	65
					1,2,3,4,7,8,9-HpCDF-13C	2.00	68
1,2,3,4,7,8-HxCDF	-----	0.69	0.41	IJ	1,2,3,4,6,7,8-HpCDD-13C	2.00	75
1,2,3,6,7,8-HxCDF	0.83	-----	0.42	J	OCDD-13C	4.00	59
2,3,4,6,7,8-HxCDF	-----	0.68	0.41	IJ			
1,2,3,7,8,9-HxCDF	ND	-----	0.70		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	6.60	-----	0.48	J	1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	-----	0.46	0.45	IJ	2,3,7,8-TCDD-37Cl4	0.20	87
1,2,3,6,7,8-HxCDD	-----	1.00	0.50	IJ			
1,2,3,7,8,9-HxCDD	1.10	-----	0.42	J			
Total HxCDD	12.00	-----	0.46	J			
1,2,3,4,6,7,8-HpCDF	3.50	-----	0.50	J	Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.64		Equivalence: 1.6 ng/Kg		
Total HpCDF	5.50	-----	0.57	J	(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	14.00	-----	0.37				
Total HpCDD	28.00	-----	0.37				
OCDF	5.40	-----	0.71	J			
OCDD	89.00	-----	0.74				

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
1700 Elm Street - Suite 200
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Tel: 612-607-1700
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Method 8290 Blank Analysis Results

Lab Sample ID	BLANK-52558	Matrix	Solid
Filename	U161101B_15	Dilution	NA
Total Amount Extracted	20.4 g	Extracted	10/27/2016 16:25
ICAL ID	U161025	Analyzed	11/02/2016 01:42
CCal Filename(s)	U161101B_03 & U161101B_19	Injected By	SMT

Native Isomers	Conc ng/Kg	EMPC ng/Kg	EDL ng/Kg		Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDF	ND	-----	0.031		2,3,7,8-TCDF-13C	2.00	75
Total TCDF	ND	-----	0.031		2,3,7,8-TCDD-13C	2.00	92
					1,2,3,7,8-PeCDF-13C	2.00	85
2,3,7,8-TCDD	ND	-----	0.033		2,3,4,7,8-PeCDF-13C	2.00	80
Total TCDD	0.042	-----	0.033	J	1,2,3,7,8-PeCDD-13C	2.00	99
					1,2,3,4,7,8-HxCDF-13C	2.00	76
1,2,3,7,8-PeCDF	ND	-----	0.039		1,2,3,6,7,8-HxCDF-13C	2.00	74
2,3,4,7,8-PeCDF	ND	-----	0.023		2,3,4,6,7,8-HxCDF-13C	2.00	78
Total PeCDF	ND	-----	0.031		1,2,3,7,8,9-HxCDF-13C	2.00	78
					1,2,3,4,7,8-HxCDD-13C	2.00	84
1,2,3,7,8-PeCDD	ND	-----	0.029		1,2,3,6,7,8-HxCDD-13C	2.00	70
Total PeCDD	ND	-----	0.029		1,2,3,4,6,7,8-HpCDF-13C	2.00	75
					1,2,3,4,7,8,9-HpCDF-13C	2.00	79
1,2,3,4,7,8-HxCDF	ND	-----	0.027		1,2,3,4,6,7,8-HpCDD-13C	2.00	90
1,2,3,6,7,8-HxCDF	ND	-----	0.023		OCDD-13C	4.00	75
2,3,4,6,7,8-HxCDF	ND	-----	0.021				
1,2,3,7,8,9-HxCDF	ND	-----	0.026		1,2,3,4-TCDD-13C	2.00	NA
Total HxCDF	ND	-----	0.024		1,2,3,7,8,9-HxCDD-13C	2.00	NA
1,2,3,4,7,8-HxCDD	ND	-----	0.036		2,3,7,8-TCDD-37Cl4	0.20	84
1,2,3,6,7,8-HxCDD	ND	-----	0.035				
1,2,3,7,8,9-HxCDD	ND	-----	0.037				
Total HxCDD	ND	-----	0.036				
1,2,3,4,6,7,8-HpCDF	ND	-----	0.036		Total 2,3,7,8-TCDD		
1,2,3,4,7,8,9-HpCDF	ND	-----	0.038		Equivalence: 0.00051 ng/Kg		
Total HpCDF	ND	-----	0.037		(Using 2005 WHO Factors)		
1,2,3,4,6,7,8-HpCDD	-----	0.046	0.028	IJ			
Total HpCDD	0.076	-----	0.028	J			
OCDF	ND	-----	0.055				
OCDD	-----	0.170	0.061	IJ			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
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J = Estimated value
I = Interference present

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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Minneapolis, MN 55414

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Method 8290 Laboratory Control Spike Results

Lab Sample ID	LCS-52559		
Filename	U161101B_18	Matrix	Solid
Total Amount Extracted	20.1 g	Dilution	NA
ICAL ID	U161025	Extracted	10/27/2016 16:25
CCal Filename(s)	U161101B_03 & U161101B_19	Analyzed	11/02/2016 04:01
Method Blank ID	BLANK-52558	Injected By	SMT

Native Isomers	**Qs** (ng)	**Qm** (ng)	% Rec.	**Internal Standards**	**ng's Added**	**Percent Recovery**
2,3,7,8-TCDF	0.20	0.19	96	2,3,7,8-TCDF-13C	2.0	67
Total TCDF				2,3,7,8-TCDD-13C	2.0	83
				1,2,3,7,8-PeCDF-13C	2.0	77
2,3,7,8-TCDD	0.20	0.17	85	2,3,4,7,8-PeCDF-13C	2.0	73
Total TCDD				1,2,3,7,8-PeCDD-13C	2.0	90
				1,2,3,4,7,8-HxCDF-13C	2.0	70
1,2,3,7,8-PeCDF	1.0	0.97	97	1,2,3,6,7,8-HxCDF-13C	2.0	67
2,3,4,7,8-PeCDF	1.0	1.0	104	2,3,4,6,7,8-HxCDF-13C	2.0	75
Total PeCDF				1,2,3,7,8,9-HxCDF-13C	2.0	76
				1,2,3,4,7,8-HxCDD-13C	2.0	80
1,2,3,7,8-PeCDD	1.0	0.95	95	1,2,3,6,7,8-HxCDD-13C	2.0	63
Total PeCDD				1,2,3,4,6,7,8-HpCDF-13C	2.0	75
				1,2,3,4,7,8,9-HpCDF-13C	2.0	81
1,2,3,4,7,8-HxCDF	1.0	1.1	107	1,2,3,4,6,7,8-HpCDD-13C	2.0	91
1,2,3,6,7,8-HxCDF	1.0	1.0	103	OCDD-13C	4.0	78
2,3,4,6,7,8-HxCDF	1.0	0.97	97			
1,2,3,7,8,9-HxCDF	1.0	1.0	101	1,2,3,4-TCDD-13C	2.0	NA
Total HxCDF				1,2,3,7,8,9-HxCDD-13C	2.0	NA
1,2,3,4,7,8-HxCDD	1.0	1.1	109	2,3,7,8-TCDD-37Cl4	0.20	81
1,2,3,6,7,8-HxCDD	1.0	1.1	114			
1,2,3,7,8,9-HxCDD	1.0	1.1	112			
Total HxCDD						
1,2,3,4,6,7,8-HpCDF	1.0	1.1	107			
1,2,3,4,7,8,9-HpCDF	1.0	1.00	100			
Total HpCDF						
1,2,3,4,6,7,8-HpCDD	1.0	0.97	97			
Total HpCDD						
OCDF	2.0	1.9	95			
OCDD	2.0	2.1	106			

Qs = Quantity Spiked
Qm = Quantity Measured
Rec. = Recovery (Expressed as Percent)
R = Recovery outside of target range
Y = RF averaging used in calculations
Nn = Value obtained from additional analysis
NA = Not Applicable
* = See Discussion

REPORT OF LABORATORY ANALYSIS

Appendix E

Classical Oneway ANOVA Statistics Tests

1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.14/28/2017 8:16:25 AM								
3			From File	Copy of USE Scanlon Fish Stats v3_0.xls								
4			Full Precision	OFF								
5			Tropic Level 4 Species									
6												
7			Total Mercury (mg/kg)									
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	2	0.12	0	0					
11			boulder	3	0.113	0.0162	2.6133E-4					
12			Grand Statistics (All data)	5	0.116	0.0121	1.4680E-4					
13												
14			Some groups have < 3 observations ANOVA Results based on such data sets may not be reliable.									
15			You may want to perform ANOVA without groups with too few observations.									
16												
17	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
18	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
19	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
20												

	A	B	C	D	E	F	G	H	I	J	K	L
1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.14/28/2017 8:30:33 AM								
3			From File	Copy of USE Scanlon Fish Stats v3_0.xls								
4			Full Precision	OFF								
5												
6	Tropic Level 4 Species											
7	Methyl Mercury (ug/kg)											
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	2	105	7.071	50					
11			boulder	3	130	10	100					
12			Grand Statistics (All data)	5	120	15.81	250					
13												
14	Some groups have < 3 observations ANOVA Results based on such data sets may not be reliable.											
15	You may want to perform ANOVA without groups with too few observations.											
16												
17	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
18	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
19	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
20												

1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.16/20/2017 3:21:24 PM								
3			From File	Scanlon fish data.xls								
4			Full Precision	OFF								
5	Trophic Level 4 Species											
6												
7			TEQ Fish1 (ng TEQ/kg)									
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	2	0.329	0.013	1.6942E-4					
11			boulder	3	0.0917	0.0632	0.00399					
12			Grand Statistics (All data)	5	0.187	0.138	0.0189					
13												
14	Some groups have < 3 observations ANOVA Results based on such data sets may not be reliable.											
15	You may want to perform ANOVA without groups with too few observations.											
16												
17	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
18	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
19	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
20												

1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.16/20/2017 3:22:22 PM								
3			From File	Scanlon fish data.xls								
4			Full Precision	OFF								
5												
6	Trophic Level 4 Species											
7			TEQ HH2 (ng TEQ/kg)									
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	2	0.315	0.0165	2.7165E-4					
11			boulder	3	0.0916	0.073	0.00533					
12			Grand Statistics (All data)	5	0.181	0.133	0.0177					
13												
14	Some groups have < 3 observations ANOVA Results based on such data sets may not be reliable.											
15	You may want to perform ANOVA without groups with too few observations.											
16												
17	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
18	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
19	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
20												

1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.14/28/2017 8:33:09 AM								
3			From File	Copy of USE Scanlon Fish Stats v3_0_a.xls								
4			Full Precision	OFF								
5												
6	Tropic Level 3 Species											
7			Total Mercury (mg/kg)									
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	6	0.125	0.0668	0.00446					
11			boulder	5	0.0726	0.00451	2.0300E-5					
12			Grand Statistics (All data)	11	0.101	0.0546	0.00298					
13												
14			Classical One-Way Analysis of Variance Table									
15			Source	SS	DOF	MS	V.R.(F Stat)	P-Value				
16			Between Groups	0.00739	1	0.00739	2.974	0.119				
17			Within Groups	0.0224	9	0.00249						
18			Total	0.0298	10							
19												
20			Pooled Standard Deviation	0.0499								
21			R-Sq	0.248								
22												
23	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
24	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
25	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
26												

	Classical Oneway ANOVA	
Date/Time of Computation	ProUCL 5.14/28/2017 8:33:37 AM	
From File	Copy of USE Scanlon Fish Stats v3_0_a.xls	
Full Precision	OFF	

Tropic Level 3 Species

Methyl Mercury (ug/kg)

Group	Obs	Mean	SD	Variance
scanlon	6	128.5	59.71	3566
boulder	5	60.8	9.731	94.7
Grand Statistics (All data)	11	97.73	55.41	3071

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	12500	1	12500	6.179	0.0347
Within Groups	18206	9	2023		
Total	30706	10			

Pooled Standard Deviation	44.98
R-Sq	0.407

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

	Classical Oneway ANOVA	
Date/Time of Computation	ProUCL 5.16/20/2017 3:22:51 PM	
From File	Scanlon fish data_a.xls	
Full Precision	OFF	

Trophic Level 3 Species

TEQ Fish1 (ng TEQ/kg)

Group	Obs	Mean	SD	Variance
scanlon	6	0.674	0.197	0.0386
boulder	5	0.0578	0.00993	9.8694E-5
Grand Statistics (All data)	11	0.394	0.351	0.123

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	1.036	1	1.036	48.19	6.7437E-5
Within Groups	0.194	9	0.0215		
Total	1.23	10			

Pooled Standard Deviation	0.147
R-Sq	0.843

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

Classical Oneway ANOVA		
Date/Time of Computation	ProUCL 5.16/20/2017 3:23:15 PM	
From File	Scanlon fish data_a.xls	
Full Precision	OFF	

Trophic Level 3 Species

TEQ HH2 (ng TEQ/kg)

Group	Obs	Mean	SD	Variance
scanlon	6	0.558	0.198	0.0392
boulder	5	0.0556	0.0111	1.2359E-4
Grand Statistics (All data)	11	0.329	0.297	0.0884

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	0.688	1	0.688	31.47	3.3002E-4
Within Groups	0.197	9	0.0218		
Total	0.884	10			

Pooled Standard Deviation	0.148
R-Sq	0.778

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

	Classical Oneway ANOVA	
Date/Time of Computation	ProUCL 5.14/28/2017 8:35:32 AM	
From File	Copy of USE Scanlon Fish Stats v3_0_b.xls	
Full Precision	OFF	

Tropic Level 2 Species

Total Mercury (mg/kg)

Group	Obs	Mean	SD	Variance
scanlon	4	0.0698	0.0124	1.5292E-4
boulder	6	0.0635	0.00831	6.9100E-5
Grand Statistics (All data)	10	0.066	0.00999	9.9778E-5

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	9.3750E-5	1	9.3750E-5	0.933	0.362
Within Groups	8.0425E-4	8	1.0053E-4		
Total	8.9800E-4	9			

Pooled Standard Deviation	0.01
R-Sq	0.104

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

1				Classical Oneway ANOVA								
2			Date/Time of Computation	ProUCL 5.14/28/2017 8:37:48 AM								
3			From File	Copy of USE Scanlon Fish Stats v3_0_b.xls								
4			Full Precision	OFF								
5												
6												
7			Methyl Mercury (ug/kg)									
8												
9			Group	Obs	Mean	SD	Variance					
10			scanlon	4	72.5	27.45	753.7					
11			boulder	6	72.83	19.97	399					
12			Grand Statistics (All data)	10	72.7	21.75	472.9					
13												
14			Classical One-Way Analysis of Variance Table									
15			Source	SS	DOF	MS	V.R.(F Stat)	P-Value				
16			Between Groups	0.267	1	0.267	5.0127E-4	0.983				
17			Within Groups	4256	8	532						
18			Total	4256	9							
19												
20			Pooled Standard Deviation	23.06								
21			R-Sq	6.2655E-5								
22												
23	Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in											
24	mean/median characteristics of the various groups at 0.05 or other selected level of significance											
25	A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.											
26												

Tropic Level 2 Species

	Classical Oneway ANOVA	
Date/Time of Computation	ProUCL 5.16/20/2017 3:23:46 PM	
From File	Scanlon fish data_b.xls	
Full Precision	OFF	

Trophic Level 2 Species

TEQ Fish1 (ng TEQ/kg)

Group	Obs	Mean	SD	Variance
scanlon	4	0.705	0.502	0.252
boulder	6	0.152	0.171	0.0291
Grand Statistics (All data)	10	0.373	0.426	0.182

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	0.733	1	0.733	6.512	0.0341
Within Groups	0.901	8	0.113		
Total	1.634	9			

Pooled Standard Deviation	0.336
R-Sq	0.449

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

Classical Oneway ANOVA		
Date/Time of Computation	ProUCL 5.16/20/2017 3:24:12 PM	
From File	Scanlon fish data_b.xls	
Full Precision	OFF	

Trophic Level 2 Species

TEQ HH2 (ng TEQ/kg)

Group	Obs	Mean	SD	Variance
scanlon	4	0.573	0.383	0.147
boulder	6	0.155	0.178	0.0316
Grand Statistics (All data)	10	0.322	0.336	0.113

Classical One-Way Analysis of Variance Table

Source	SS	DOF	MS	V.R.(F Stat)	P-Value
Between Groups	0.42	1	0.42	5.614	0.0453
Within Groups	0.599	8	0.0748		
Total	1.019	9			

Pooled Standard Deviation	0.274
R-Sq	0.412

Note: A p-value <= 0.05 (or some other selected level) suggests that there are significant differences in mean/median characteristics of the various groups at 0.05 or other selected level of significance

A p-value > 0.05 (or other selected level) suggests that mean/median characteristics of the various groups are comparable.

Appendix B

Technical Analysis

Appendix B – Technical Analysis
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Three remedial alternatives involving construction activities and one alternative involving a no action approach were developed and evaluated as part of the Scanlon Reservoir Focused Feasibility Study (FFS) and include the following:

Alternative 2 – Enhanced Monitored Natural Recovery with Thin-Layer Amended Cover

Alternative 3 – Potential Bioactive Zone Cap

Alternative 4 – Sediment Dredging and Excavation

Alternative 5 – Enhanced Monitored Natural Recovery with Broadcast Amendment

Class 4 rough order of magnitude cost analyses (+50/-30) were developed for each of these alternatives and are summarized within **Section 3** of the FFS document. This Technical Analysis serves to provide the calculations and outline the assumptions used to compile each of the alternative cost analyses.

Cost estimates were compiled using a variety of sources. These sources include construction cost data from RSMeans estimating software for open shop pricing in Duluth, Minnesota; current Bay West LLC (Bay West) and state contract rates for labor, equipment, and sample analysis; personal communication with vendors; historic cost data from projects similar in size and scope; other FFS documents, presentations, or technical papers that provided estimated or real construction cost data; and available online vendor pricing of materials.

The selection of construction equipment, production rates, remedial volumes, remedial action areas, and other "design-type" elements used as a starting point to develop alternative costs are based on a current understanding of Site conditions at this early feasibility study-level stage.

This document is divided into the following sections:

Section 1: Remedial Areas and Volumes
Section 2: Construction Implementation Assumptions and Production Rates
Section 3: Sediment Dewatering/Staging Areas
Section 4: Environmental Controls and Construction Monitoring
Section 5: Sediment Stabilization
Section 6: Water Treatment
Section 7: Transportation and Disposal
Section 8: Cover/Cap Materials
Section 9: References

The following tables were used to calculate values incorporated into each alternative cost analysis and are included within this Technical Analysis:

Appendix B Table 1: Volume, Rate, and Timeframe Calculations
Appendix B Table 2: Unit Rate Calculations
Appendix B Table 3: Lump Sum Costs
Appendix B Table 4: Monitoring and Evaluation Costs
Appendix B Table 5: Present Value Calculations

Many of the assumptions used to compile the cost analyses for the alternatives are included within the tables. Those aspects of alternative development not readily apparent within the tables and the FFS text are described in the following sections

Section 1: Remedial Areas and Volumes

Areas targeted for remedial action (referred to as the "remedial footprint" within the FFS document) were selected based on a limited set of data which included sample results from events conducted in 2011 and 2014. Areas were selected for inclusion into the remedial footprint if concentrations of dioxins and/or mercury (the Site contaminants of concern [COC]) in sediments exceeded their respective Midpoint SQT

(the Site cleanup levels [CULs]) or were believed to be depositional in nature and therefore potentially contain COCs above the CULs. The remedial footprint is presented in Figure 5 of the FFS text and totals approximately 17 acres. It is anticipated that the remedial footprint would be further refined during the design phase.

The total volume of contaminated sediment at the Site was calculated by splitting the remedial footprint into two areas. These areas include the "eastern arm", which is the large area east of and outside the main river channel and is known to contain contaminated sediments, and the smaller inlets immediately adjacent to the main river channel. Limited data exists for these small inlets and aerial imagery and Site bathymetry indicate these areas are depositional in nature. These areas were included within the remedial footprint as a conservative approach to estimating contamination at the Site as Site data is limited.

Several sediment cores were collected from within the eastern arm and were collected to refusal. The greatest penetration achieved during sampling was 0.61 meter. It was therefore assumed that a hard substrate is present immediately below this depth and contaminated sediments extend to 0.65 meter below the sediment surface (bss). Multiplying the area of the eastern arm (approximately 13.5 acres) by an assumed average contaminant depth of 0.65 meter results in a contaminated sediment volume of approximately 47,000 cubic yards.

The depth of contamination within the smaller inlet areas adjacent the river channel was estimated at 0.50 meter, as data is limited in these areas. The 0.50 meter estimate was chosen as a "middle ground" between two scenarios – no contamination within these areas and contamination up to 1.0 meter bss. As stated previously, additional data collection is necessary to refine the remedial footprint and remedial volumes. Multiplying the total combined area of these inlets (approximately 3.5 acres) by an assumed average contaminant depth of 0.50 meter results in a contaminated sediment volume of approximately 9,000 cubic yards.

Alternative 4 (Sediment Dredging and Excavation) incorporates removal of contaminates sediments but also incorporates an over dredge thickness of 0.15 meter (0.5 foot) across the entire remedial footprint. This increases the total volume of sediment requiring removal to approximately 69,000 cubic yards. An over dredge was assumed as a means of increasing sediment removal efficiency and mitigating the presence of dredge residuals.

Section 2: Construction Implementation Assumptions and Production Rates

Unit rate costs were developed for all cover construction, cap construction, sediment dredging, and sediment excavation elements by summing labor and equipment costs and dividing by an assumed production rate; therefore, the production rate has a substantial impact on the unit rate cost of these activities and the overall project cost. The following sections detail the construction methods developed for remedy implementation and their associated production rates. It is important to note that these methods were developed solely to assist in developing cost estimates for each alternative, and final construction methods would be determined during the design and/or construction bidding phase.

Sand Cover/Cap Construction ("In the Wet")

A general order of operations was assumed in order to facilitate costing of alternatives involving placement of sand and/or amendment materials over open water, referred to as "in the wet" (Alternatives 2, 3, and 4). This order of operations was used to assist in selecting construction equipment, labor, production rates, time frames, etc.

The general order of cover/cap placement activities used to compile the cost analyses are described as follows:

- Clean washed sand meeting project specifications would be purchased from a local upland borrow source and imported to the staging area located along the Site's southeastern shoreline via on-road dump trucks. Amendment materials would be purchased from a supplier, shipped to

the staging area, and stockpiled. Mixing of amendment materials would be conducted mechanically using an end loader, excavator, or similar equipment.

- Cover/Cap materials would be stockpiled at a near-shore material loading area, where an excavator or Derrick crane would load small, 25-cubic yard hopper barges with the stockpiled cover/cap materials on a regular basis. An end loader would be used to manage materials imported to the staging area and maintain the material loading area stockpile.
- A single work boat or small tug would be used to manage the two hopper barges. The work boat would transfer the hopper barges between the material loading area and the cover/cap placement barge on a regular basis. A full hopper barge would be delivered to the placement barge by the time the second hopper barge had been emptied. The emptied hopper barge would then be returned to the loading area and filled again with cover/cap material.
- These activities would be conducted until cover/cap construction is complete.

Placement of cover/cap materials in this manner assumes that sufficient draft is available across the Site to float the placement barge. Areas with insufficient draft may require placement via other methods. Based on available bathymetry, it is assumed that sufficient water depth is available for cover/cap construction via the method outlined above.

Production of sand cover/cap construction was estimated at 50 cubic yards per hour. This estimate assumes that two, 25-cubic yard hopper barges could be emptied within 1 hour. This production rate is roughly equivalent to a 2-cubic yard bucket, filled 75 percent (%), with a cycle time of 2 minutes. It was assumed that construction of the sand cover/cap would take place for 10.5 hours each day, for a total daily production of 525 cubic yards.

Sand Cover/Cap Construction ("In the Dry")

Alternative 4 incorporates construction of a cofferdam, dewatering of the area inside the cofferdam, and sediment removal within the cofferdam via conventional earth moving equipment. This is referred to as sediment removal/excavation "in the dry" (see "Sediment Removal in the Dry" section presented below). Any cover constructed following sediment removal in the dry would also be constructed in the dry. Although complete removal of contaminated sediments could be accomplished more readily in the dry as compared to removal in the wet, construction of a sand cover was still included in the cost analysis in the event contamination remained in inaccessible areas (i.e., bedrock crevices, shoreline embankments, etc.) following sediment removal.

The general order of cover placement activities used to compile the cost analysis is described as follows:

- Clean washed sand meeting project specifications would be purchased from a local upland borrow source and imported directly into the dewatered area inside the cofferdam. Sand would be dumped directly adjacent to the area being graded at that time or dumped into a stockpile and later transported as needed via end loader. Crane mats and/or gravel haul roads would be constructed as needed to facilitate material import by on-road dump trucks.
- A bulldozer and survey equipment would be used to construct the 0.15-meter (0.5-foot) sand cover.

Production of the sand cover in the dry was estimated at 72 cubic yards per hour. This estimate assumes that a 12-cubic yard truckload of sand would be delivered to the Site every 10 minutes, and that the 12 cubic yards of sand could be graded within a 10 minute timeframe. It was assumed that sand would be imported for 10.5 hours per day, for a total daily production of 756 cubic yards.

Sediment Removal "In the Wet"

Alternative 4 incorporates removal of sediments in the wet from several inlets located adjacent to the river channel. These inlets appear to be depositional in nature and therefore may contain contaminated sediments. The total combined area of the inlets is 3.5 acres. These areas are located outside the cofferdam area and would therefore require sediment removal in the wet. A traditional approach of

sediment removal using a barge-mounted excavator was assumed for the purposes of the cost analysis due to the small volume of sediments estimated to require removal.

The general order of sediment dredging activities used to compile the cost analysis is described as follows:

- Sediments would be removed from the river bottom using a barge-mounted excavator and environmental clamshell bucket. The dredged sediments would be placed into a hopper barge located adjacent the dredge barge.
- The hopper barge, once filled, would be pulled away from the dredge barge using a work boat or small tug. An empty hopper barge would then be repositioned adjacent the dredge barge to replace the filled hopper barge. Dredging would then continue as the work boat transported the filled hopper barge to the sediment offload area, located at the staging area adjacent to the Site's southeastern shoreline.
- Surficial water would be pumped from the hopper barge and retained for treatment. Sediments would then be offloaded from the hopper barge using a derrick crane located onshore. The emptied hopper barge would be returned to the dredge barge and the cycle would be repeated until dredging is complete.
- Offloaded sediments would be placed directly on a lined, paved, and bermed sediment stabilization pad. Sediments would be mixed with an amendment material such as Portland cement to remove free water and render sediments stackable. The amendment material would be mixed into the dredged sediments using an end loader and stockpiled until free liquid is no longer present (i.e., until amended sediments pass the paint filter test).
- Sediments meeting disposal criteria would be loaded into on-road dump trucks using an end loader and transported to an off-site landfill for disposal.

Production of sediment removal in the wet was estimated at 50 cubic yards per hour. This estimate assumes that two, 25-cubic yard hopper barges could be filled within 1 hour. This production rate is roughly equivalent to a 2-cubic yard bucket, filled 75%, with a cycle time of 2 minutes. It was assumed that sediment removal would take place for 10.5 hours each day, for a total daily production of 525 cubic yards.

Sediment Removal "In the Dry"

Alternative 4 incorporates installation of a cofferdam to allow the majority of Site sediments to be excavated in the dry, as stated previously. The cofferdam would be used in conjunction with Site geography to isolate the eastern arm from the main river channel. The general order of cofferdam installation, dewatering, and sediment removal activities used to compile the cost analysis are described as follows:

- Cofferdam installation would be conducted concurrently with construction of the staging area. Dewatering of the area inside the cofferdam would commence following closing of the cofferdam. It was assumed for the purposes of the cost analysis that the first 80% of water removed from the cofferdam would not require treatment. The remaining 20% was assumed to contain suspended solids and associated Site COCs and would therefore require treatment (See Section 6: Water Treatment).
- Sediment removal in the wet, as described previously, would be conducted during dewatering of the cofferdam area. This assumes that a portion of shoreline is maintained outside the cofferdam and along the staging area for the sediment offload area.
- Following dewatering, any visual debris such as submerged trees would be removed and the river bottom would be trenched as necessary to control water weeping from in-situ sediments, infiltrating groundwater, infiltrating river water, and precipitation. Trenches would be routed to one or more excavated sumps and collected water would be pumped to the water treatment system.

A grade/ramp suitable for routing on-road dump trucks into and out of the Site would be constructed between the staging and cofferdam areas.

- Sediment removal would begin immediately adjacent to the staging area. Sediments would be removed to designed grades using an excavator and survey equipment. Excavated sediments would be placed into off road dump trucks and then dumped at a sediment stabilization area, where an amendment such as Portland cement would be mixed into the sediments. Crane mats and/or gravel would be used to create haul roads suitable for access to the stabilization/sediment loadout area by on road dump trucks.
- Stabilized sediments would be loaded into on-road trucks via end loader and hauled to an offsite landfill for disposal. Excavation, stabilization, and loadout of sediments would continue until sediment removal is complete.
- Sand cover construction would be completed next, followed by cofferdam removal (see "Sand Cover/Cap Construction in the Dry" section).

Production of sediment removal in the dry was estimated at 72 cubic yards per hour. This estimate assumes that one 12-cubic yard truck can be filled every 10 minutes. It was assumed that sediment removal would take place for 10.5 hours each day, for a total daily production of 756 cubic yards.

Amendment/Sand Cover Construction Equipment

Alternatives involving distribution of sand and/or amendment materials assume that different methods of placement would be utilized in wetland areas as compared to open water areas. Open water areas were assumed to utilize a conventional barge-mounted excavator with environmental clamshell bucket for placing materials as there are no draft limitations in the open water areas.

Wetland areas would not be capable of floating a material placement barge and thus would require a different method of placement. The use of crane mats or equivalent technology was not considered because wetland areas were observed to have water depths exceeding 3 feet during the 2015 RI field sampling event, and bog-type wetland areas were also observed at the Site's southern end. The proposed method used for cost analysis is an amphibious vehicle such as a Marsh Buggy or equivalent outfitted with a 12-cubic-yard bucket and stone slinger attachment. Such a vehicle is capable of navigating open waters and traversing upland areas. Production rates for this equipment was estimated based on round-trip travel times, capacity of each vehicle, and use of two vehicles at a time. Each vehicle was assumed to have an application time of 1.2 hours per load, a travel time to and from the vehicle-loading location (i.e., material transport barge mooring location along the railroad embankment) of 10 minutes and a load time of 5 minutes. A placement time frame of 11 hours per day equates to a total daily production for two vehicles of 168 cubic yards.

Photo showing MBI Marsh Buggy with dump box; photo from http://marshbuggies.com

Photo showing stone slinger equipment; photo from http://bcginvestments.net/Stone_Slinger.html

Cover materials would be placed in open water areas using a conventional barge-mounted excavator. Materials would be delivered to the excavator by two material transport hopper barges, each with a 25-cubic-yard capacity. The production rate for open water material placement was estimated using a bucket size of 2 cubic yards, a 70 percent (%) fill rate, and 2 minutes per cycle. The bucket size and fill percentage was reduced (as compared to the dredging production rate estimate) to allow for ease of placement within the small 25-cubic-yard hopper barges. A placement time frame of 10 hours per day equates to a total daily production for a single excavator of 420 cubic yards.

Dredging Equipment

Alternatives involving dredging of sediments assume that sediments would be slurried with water and pumped as low solids content slurry (e.g., less than 5% solids) to a nearby dewatering area. This assumption was made to avoid passing of contaminated sediments over the railroad embankment into a transport barge and subsequent barging of sediments to Hallett Dock #7 for dewatering. Equipment was assumed to consist of a barge-mounted mechanical excavator with environmental clamshell bucket and slurry tank (i.e., hopper) or hydraulic dredge; costs for this equipment were assumed to be similar enough for FFS-level cost analyses.

The dredging production rate was estimated partially based on U.S. Environmental Protection Agency (USEPA) sediment remediation guidance (USEPA, 2005), which provides production rates for various sizes of mechanical buckets based on an 80% fill and cycle time of 2 minutes. These rates range from 63 cubic yards per hour for smaller buckets to 252 cubic yards per hour for larger buckets. Another source used to determine the dredge production rate was the St. Louis River/Interlake/Duluth Tar (SLRIDT) Data Gap Report (Service, 2002), in which a review of previous projects and discussions with interested parties resulted in a recommended dredge production rate of 50 cubic yards per hour. Based on these two sources, the dredge production rate for the Site was conservatively estimated at 72 cubic yards per hour. This rate assumes a 3-cubic-yard bucket filled 80%, a 2-minute cycle time, and an active dredging time frame of 10 hours per day. Dredging downtime is estimated at 2 hours per day to account for morning meetings/safety briefings, startup times, shutdown times, and periods of downtime throughout the day. These factors equate to a daily production rate of 720 cubic yards per day.

Section 3: Sediment Dewatering/Staging Areas

Implementation of Alternatives 2, 3, and 4 would require construction of an upland staging area in which to stage and conduct construction activities. The staging area would consist of: an office trailer and parking area for Site workers; laydown areas for equipment; and access roadways to move materials into and out of the Site. Alternatives involving cover or capping elements would also require a large area for stockpiling cover/cap materials and a waterside location in which to transfer material into hopper barges. Alternative 4, which incorporates sediment removal, would also require a lined, paved, and bermed pad for contained mixing of removed sediments and an amendment material, as well as an amendment storage area and equipment decontamination area (e.g., truck tire wash). The ideal location for a staging area would be directly adjacent to the remedial footprint, the majority of which is present at the far eastern end of the Site. The staging area would also be located in an easily accessible area that is both acceptable to the land owner (Minnesota Power) and is not disruptive to nearby residences. A review of aerial Site imagery indicates a small clearing at the Reservoir's southeastern shoreline and an access roadway off of County Road 61. This location is a logical choice for a staging area and would present minimal disturbances to the public as few residences are located nearby. It was assumed for the cost analyses that 2 acres of land at the southeastern end of the Site would require clearing and grubbing for construction of the staging area, and that another 1 acre would require clearing and grubbing for widening of the access roadway. Crushed concrete would be placed to construct the haul roads and staging areas and was assumed to be left in place as a Site improvement following construction activities. An additional cost was assumed for Alternative 4 for construction of a 150 feet by 150 feet lined, paved, and bermed sediment stabilization pad. The stabilization pad would be used during sediment removal in the wet and potentially during early phases of sediment removal in the dry.

Section 4: Environmental Controls and Construction Monitoring

Environmental controls and construction monitoring are important elements in mitigating environmental impacts occurring as a direct result from construction activities and also in ensuring remedial/construction goals are achieved. Environmental controls can include surface water control structures (e.g., silt curtains, sheet piling, and absorbent boom), lined sediment dewatering pads, tire washes, stormwater controls, and site fencing (for protection of human health). Construction monitoring can include turbidity monitoring during dredging activities, air monitoring during intrusive site activities, treated dredge contact water sampling, post-dredge verification sampling, cap thickness verification coring, bathymetric surveys, imported materials sampling, dewatered sediment sampling, and collection of pre- and post-construction upland soil samples within the staging area footprint. Alternatives involving amendment application or thin-layer cover construction as a remedy would likely require less controls and monitoring than alternatives incorporating dredging.

For the purposes of this FFS, it was assumed that alternatives consisting of cover or cap construction would incorporate the following control and monitoring elements:

- Fencing around the staging area;
- Chemical and physical sampling of imported cover materials to ensure that they are suitable for use; and
- Cover thickness verification coring to ensure that project specifications are achieved.

Alternative 4, which incorporates sediment removal, would require controls and monitoring as listed above for cover/cap placement and in addition:

- Lined and bermed dewatering pad for sediment stabilization, to include an equipment decontamination area;
- Surface water controls during sediment removal in the wet, to include silt curtains surrounding and downstream of the dredge;
- Real-time turbidity monitoring during sediment removal in the wet and cofferdam dewatering;

- Post-dredge/excavation verification sampling;
- Dewatered sediment sampling;
- Pre- and post-construction soil sampling; and
- Treated cofferdam and dredge contact water sampling.

Section 5: Sediment Stabilization

Sediments removed from the Site in the wet, and potentially some sediments removed in the dry, are expected to have entrained and interstitial water (i.e., dredge contact water) making them unsuitable for direct and/or immediate transportation to an off-site landfill. Therefore, removed sediments would require dewatering/stabilization in order for them to pass the paint filter test (i.e., essentially no free water) and make them suitable for transportation and disposal. The dewatering/stabilization process would rely upon the addition of amendments to the dredged sediments, along with gravity draining of entrained and interstitial water onto the sediment stabilization pad ("pad"; sediments removed in the wet) or the in-cofferdam sediment stabilization area (sediments removed in the dry). It was assumed for the purposes of the Alternative 4 cost analysis that all sediments removed in the wet and in the dry would require stabilization with an amendment such as Portland cement.

The pad would be constructed prior to commencement of dredging activities. The pad would be a primary feature of the sediment staging area and must be large enough so that four operations could be conducted on the pad at once. These operations include: offloading dredged sediments from the hopper barges and placing them on the pad; end loader transport of offloaded dredged sediments to a mixing area and subsequent mixing of sediments with an amendment such as Portland Cement; stockpiling of amended sediments for several days if necessary to attain adequate cohesiveness; and loadout of dewatered sediments into on-road dump trucks. The constructed pad would be lined, paved, and bermed to contain contaminated sediments and to facilitate gravity draining of interstitial water and precipitation falling onto the pad into a sump area. Dredge contact water collected in the sump would be pumped into frac tanks (i.e., equalization tanks) and treated. Construction of a 150-foot by 150-foot pad was incorporated into the cost analysis for Alternative 4.

The dewatering/stabilization process would incorporate the use of binders (i.e., amendments) that generate a cementitious reaction with the available water and solid matrix of the dredged sediments. Common amendments for sediment dewatering/stabilization include Portland cement, fly ash, lime cement, and lime kiln dusts. These amendments are powdered materials that require enclosed transport and storage systems to reduce dust migration and premature hydration. Some materials, such as fly ash, may be available locally at a substantially reduced cost relative to Portland cement. For the purposes of this FFS it was assumed that solidification with 15% Portland cement by volume would be conducted. Pilot scale or treatability studies should be conducted during the design phase to identify desirable amendment materials and amendment rates.

Section 6: Water Treatment

Three operations associated with implementation of Alternative 4 would require treatment of water, analysis of the treated water, and subsequent discharge of treated water back into the river:

1) Following cofferdam installation, water would be pumped from inside the cofferdam and discharged to the river side of the cofferdam as part of dewatering operations. This operation would likely require treatment of water anticipated to contain suspended sediments and therefore sediment contaminants, or COCs, to avoid discharge of contamination into the river. It was assumed that the initial 80% of water pumped from within the cofferdam area would not contain dissolved and/or suspended contaminants and would therefore not require treatment. It was assumed that the final 20% of water pumped from within the cofferdam area would contain dissolved and/or suspended sediments and associated COCs and would therefore require treatment prior to discharge.

2) Sediments dredged in the wet (Alternative 4) would be placed within hopper barges and later transported to the sediment offloading area. Some entrained and interstitial water contained within the dredge bucket and the dredged sediments, respectively, would rise to the surface within the hopper barges during dredging and transport. This surficial water would be highly turbid and contain dissolved and suspended sediment contaminants and would therefore require treatment. Surficial water would be pumped from the hopper barges and to the water treatment plant upon the barge reaching the sediment offloading area.
3) Water would likely accumulate within the cofferdam area following dewatering operations. Potential sources of water to accumulate within the cofferdam include: interstitial water draining from in-situ sediments, water seeping from between joints in the cofferdam sheet piling, infiltrating groundwater, and precipitation falling within the cofferdam area. Trenching would be conducted to encourage drainage of these sources into centrally located sump pits. Water would be pumped from the sump pits to the water treatment plant.

Water generated from the three sources above would be routed to frac/equalization tanks located at the staging area and combined. Treatment of water would be conducted on an as needed, batch basis. The cost analysis for Alternative 4 includes rental rates for equipment, costs for procuring media and filters, disposal costs of media and filters, and labor. Costs were estimated on a lump sum basis and incorporate equipment rental over the entire implementation period, full-time labor over the entire project duration, and a single media change-out event. Costs were obtained from a contractor quote provided for a different project but relevant to the scale of dredging activities evaluated for the Site.

Section 7: Transportation and Disposal

Transportation costs for sediment disposal were estimated on a per ton basis using truck rental and operator rate data obtained from RSMeans cost estimating software. It was assumed that each truck would carry 12 tons or 16 cubic yards (1.4 tons per cubic yard) and would complete 2 round trips per hour.

Disposal costs were obtained for the Vonco V Waste Management Campus (obtained during compilation of the Minnesota Slip Feasibility Study) located at 1100 West Gary Street in Duluth, Minnesota (approximately 12 miles west of the Site) and Shamrock Environmental Landfill located at 761 Highway 45 in Cloquet, Minnesota (approximately 20 miles west of the Site). Costs for these two disposal facilities were comparable for the purposes of this FFS, at $12 per ton and $16 per ton (not including environmental fees and taxes) respectively. The Vonco V landfill was used for the cost analysis due to its closer proximity to the Site.

The final volume of sediments requiring disposal will be a result of in situ volume, bulking of sediments as they are dredged and handled, and the addition of stabilizing agents during the dewatering process. For the purposes of this FFS, sediment bulking was assumed at 10% and amendment addition was assumed at 15% by volume. An average density of 1.4 tons per cubic yard was assumed for dredged and stabilized sediment.

Section 8: Cover/Cap Materials

Potential sources of cover/cap materials include materials from an upland borrow location (e.g., sand and gravel pit), sediments previously dredged for navigational purposes, and common earth upland soil. Natural materials such as dredged sediments and common earth upland soils often contain fine-grained components which make placement more difficult (ITRC, 2014). It was assumed for the purposes of the cost analyses that upland borrow materials would be used as no apparent source of dredged materials is readily available near the Site. Upland borrow material consisting of clean, washed sand was assumed for alternatives incorporating sand cover or cap elements. The exact grain size specifications would be developed during the design phase but would likely consist of medium to coarse grain sands that would withstand mild erosive forces in the slip. Cobble obtained from an upland borrow location was also assumed for alternatives requiring cap armor.

For the sand cover and sand cap alternatives, it was assumed that sand will be purchased from an upland borrow location and loaded into trucks at a rate of $6.90 per CY based on pricing procured from a local supplier. Rip rap or cobbles used as armor over cap materials was estimated at $14.35 per cubic yard based on available online pricing. Assumptions used for importing materials to the site were the same as those for transportation and disposal of dewatered sediments, as described in **Section 8**.

Section 9: References

U.S. Environmental Protection Agency (USEPA), 2005. "Contaminated Sediment Remediation Guidance for Hazardous Waste Sites."

Interstate Technology and Regulatory Council (ITRC) Contaminated Sediments Team, 2014. "Contaminated Sediments Remediation – Remedy Selection for Contaminated Sediments," August.

Appendix B: Table 1
Volume, Rate, and Timeframe Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Contaminated Sediment Volumes		
Total Volume of Contaminated Sediment		
Areas adjacent to river channel (acres)	2.45	
Assumed depth of contamination (feet)	1.64	0.50 (meter)
Volume of contaminated sediment adjacent to river channel (cubic yards)	6482	
Eastern arm of Site (acres)	14.02	
Assumed depth of contamination (feet)	2.13	0.65 (meter)
Volume of contaminated sediment in eastern arm (cubic yards)	48224	
Total combined volume of contaminated sediment at the Site (cubic yards)	54706	
Dredge Volumes (Alternative 4)		
Dredge Volumes for Alternative 4		
Areas to be dredged "in the wet" (acres)	2.5	
Target dredge depth (feet)	1.6	0.50 (meter)
Over-dredge depth (feet)	0.5	0.15 (meter)
Total dredge volume (cubic yards)	8427	
Areas to be excavated "in the dry" (acres)	14.02	
Target excavation depth (feet)	2.13	0.65 (meter)
Over-excavation depth (feet)	0.5	0.15 (meter)
Total excavation volume (cubic yards)	59352	
Total volume of sediment to be removed (cubic yards)	67779	
10% by volume bulking factor (cubic yards)	6778	
15% by volume solidification agent (cubic yards)	10167	
Transport/Disposal volume (cubic yards)	84724	
Transport/Disposal weight (tons)	118614	1.4 tons per cubic yard

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Volume, Rate, and Timeframe Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Amendment/Cover Volumes			
Alternative 2: Thin-Layer Amended Cover (Amendment Requirement)			
Amendment ratio (percent carbon by weight in upper 0.15 meter)	4		Changed to 4% on 3/31/20 per Bench Scale Study
Remedial area (acres)	16.5		
Volume of sediment in upper 0.30 meter (cubic yards)	26153		
Assumed density of in-situ sediment (tons per cubic yard)	1.4		
Assumed weight of sediment in upper 0.30 meter (tons)	36614		
Amount of activated carbon to be added (tons)	1465		
Assumed density of activated carbon (tons per cubic yard)	1.72		
Volume of activated carbon to be added (cubic yards)	851		
Amount of activated carbon to be placed (cubic yards per square yard)	0.071756		
Thickness of amendment (centimeter)	6.561345		
Conservative factor	1		
Assumed amount of activated carbon to be purchased (tons)	1465		Used to determine shipping costs
Alternative 2: Thin-Layer Amended Cover (Sand Requirement)			
Cover area (acres)	16.5		
Cover thickness (inches)	12.0	0.30 (meter)	
Sand and amendment required (cubic yards)	26571.6		
Subtract out amendment (cubic yards)	851		
Sand required, less amendment (cubic yards)	25721		
Alternative 2: Thin-Layer Amended Cover (Armor Requirement)			
Armor area (acres)	2.5		
Armor thickness (feet)	0.5	0.15 (meter)	
Armor required (cubic yards)	1976		
Alternative 3: PBAZ Cap (Sand Requirement)			
Backshore/Foreshore habitat area (acres)	2.15		
Cap thickness (feet)	3.94	1.2 (meter)	
Mixing layer thickness (feet)	0.50		
Total thickness (feet)	4.44		
Total volume (cubic yards)	15387		
Emergent aquatic vegetation habitat area (acres)	8.64		
Cap thickness (feet)	3.28	1.0 (meter)	
Mixing layer thickness (feet)	0.50		
Total thickness (feet)	3.78		
Total volume (cubic yards)	52690		
Submerged aquatic vegetation and deep water habitat area (acres)	5.68		
Cap thickness (feet)	1.64	0.5 (meter)	
Mixing layer thickness (feet)	0.50		
Total thickness (feet)	2.14		
Total volume (cubic yards)	19610		
Total volume of sand required for Alternative 3 (cubic yards)	87688		
Alternative 3: PBAZ Cap (Armor Requirement)			
Armor area (acres)	2.5		
Armor thickness (feet)	0.5	0.15 (meter)	
Armor required (cubic yards)	1945		
Alternative 4: Sediment Dredging and Excavation (Sand Requirement)			
Areas adjacent to river channel (acres)	2.5		
Cover thickness (feet)	0.5	0.15 (meter)	
Cover volume for areas adjacent to river channel (cubic yards)	1945		Assume shallow bedrock present and no deep-rooting plants
Eastern arm of Site (acres)	14.0		
Cover thickness (feet)	0.5	0.15 (meter)	
Cover volume for eastern arm (cubic yards)	11129		
Total combined cover volume at the Site (cubic yards)	13073		
Alternative 5: EMNR with Broadcasted Amendment			
Application areas (acres)	16.5		
Amendment thickness required per acre (inches)	0.384298	0.010 (meter)	
Amendment required (cubic yards)	851		
Contaminated areas (acres)	16.5		
Amendment tons per acre	31		(metric tons)
Amendment required (tons)	510.57		

Appendix B: Table 1
Volume, Rate, and Timeframe Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Production Rates			
Construct Thin-Layer Amended Cover or PBAZ Cap (Alternatives 2 and 3)			
Production Rate (cubic yards per hour)	50		Assume 2, 25-cubic yard transport barges are emptied per hour
Hours worked per day	10.5		
Daily production (cubic yards)	525		
Dredge Sediment "In the Wet" (Alternative 4)			
Dredge production (cubic yards per hour)	50		Assume 2, 25-cubic yard transport barges are filled per hour
Active dredging duration per day (hours)	10.5		
Daily production (cubic yards)	525		
Excavate Sediment "In the Dry" (Alternative 4)			
Time to fill a single off-road dump truck (minutes)	10		
Number of trucks filled per hour	6		
Truck capacity (cubic yards)	12		Assume smaller trucks due to space constraints on crane mats
Material excavated and brought to stabilization area per hour	72		
Daily excavation timeframe (hours)	10.5		
Total daily production (cubic yards)	756		
Construct Sand Cover Following Dredging (Alternative 4)			
Material import rate (minutes per truckload)	10.0		
Number of trucks per hour	6		
Truck capacity (cubic yards)	12		
Material imported per hour (cubic yards)	72.0		
Import duration per day (hours)	10.5		
Total daily production (cubic yards)	756		
Stone Slinger Barge Production Rate (Broadcasted Amendment in Open Water Areas) - Alternative #5			
Cycle Time			
Hopper capacity (cubic yards)	12		
Application time per cubic yard placed (minutes)	6		
Application time per load (minutes)	72	1.2	hours
Load time (minutes)	5	0.083	hours
Add in time for travel (minutes)	10	0.17	hours
Total cycle time (hours)	1.45		
Production Rate			
Active placement time per day (hours)	11		
Number of cycles per day per barge	7		
Number of barges	2		
Total volume of amendment applied per day (cubic yards)	168		

Appendix B: Table 1
Volume, Rate, and Timeframe Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Water Treatment Volume		
Area contained within cofferdam (acres)	14.02	
Average depth (feet)	4	
Estimated volume (cubic feet)	2442845	
Estimated volume (gallons)	18272479	
Estimated dewatering rate (gallons per minute)	2000	Two 1,500 GPM pumps operating at 1,000 GPM; 24 hours per day
Estimated volume dewatered per day (gallons)	2880000	11 hours per day
Volume of untreated water discharge (percent of total)	80	
Volume of untreated water discharged (gallons)	14617983	
Timeframe required for pumping untreated water (days)	6	
Estimated treatment rate (gallons per minute)	150	
Estimated volume treated per day (gallons)	216000	24-hour treatment
Volume of treated water discharge (percent of total)	20	
Volume of water requiring treatment (gallons)	3654496	
Days required for treatment	17	
Total days of dewatering and treatment prior to excavation	23	5
Construction Timeframe		
Alternative 2: Enhanced MNR with Thin-Layer Amended Cover		
Construct staging area and mobilize/setup equipment (days)	10	
Construct cover (days)	51	
Place armor (days)	6	
Breakdown equipment/demobilize and site restoration (days)	5	
Total time on-site (days)	72	14 weeks (5 day work week)
Alternative 3: PBAZ Cap		
Construct staging area and mobilize/setup equipment (days)	10	
Construct cover (days)	167	
Place armor (days)	5	
Breakdown equipment/demobilize and site restoration (days)	5	
Total time on-site (days)	187	31 weeks (6 day work week)
Alternative 4: Sediment Dredging and Excavation		
Construct staging area and mobilize/setup equipment (days)	15	
Construct cofferdam (days)	15	Completed concurrent to staging area construction
Dewater cofferdam and treat water (days)	23	
Dredge sediments "in the wet" (days)	17	Completed concurrent to cofferdam dewatering
Excavate sediments "in the dry" (days)	79	
Construct sand cover "in the wet" (days)	4	
Construct sand cover "in the dry" (days)	15	Completed concurrent to construct sand cover "in the wet"
Remove cofferdam (days)	15	
Site restoration, breakdown equipment/demobilize and site restoration (days)	10	Completed concurrent to cofferdam removal
Total time on-site (days)	141	28 weeks (5 day work week)
Alternative 5: Enhanced MNR with Broadcasted Amendment		
Construct staging area and mobilize/setup equipment (days)	5	
Place amendment (days)	6	
Breakdown equipment/demobilize and site restoration (days)	5	
Total time on-site (days)	16	3 weeks

Appendix B: Table 2
Unit Rate Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Construct Thin-Layer Amended Cover and PBAZ Cap (Alternatives 2 and 3)					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Equipment					
Upland crane	Day	$1,720.08	1	$1,720.08	Load transport hopper barges
Transport hopper barges	Day	$129.00	2	$258.00	25 cubic yard capacity hopper barges
Transport tug	Day	$551.00	1	$551.00	Small tug to transport hopper barges
Barge-mounted excavator	Day	$1,265.00	1	$1,265.00	Place cover and cap material
Clamshell bucket	Day	$70.00	2	$140.00	
RTK DGPS for excavator	Day	$350.00	1	$350.00	
Excavator barge	Day	$355.00	1	$355.00	With spuds and winches
End loader	Day	$580.00	1	$580.00	Handle materials
Survey vessel with equipment	Day	$1,500.00	1	$1,500.00	Track progress and QA/QC data
Pickup trucks	Day	$97.00	3	$291.00	Site supervisor, foreman, mechanic
Telehandler	Day	$567.00	1	$567.00	Move equipment around Site
Site generator	Day	$562.29	1	$562.29	Power office trailers
			SUBTOTAL	$8,139.37	
Labor					
On-site project management	Day	$1,200.00	1	$1,200.00	
Foreman	Day	$854.00	1	$854.00	
Mechanic	Day	$980.00	1	$980.00	
Crane operator	Day	$1,106.00	1	$1,106.00	
Tug operator	Day	$1,036.00	1	$1,036.00	
Excavator operator	Day	$1,106.00	1	$1,106.00	
Bargehand	Day	$812.00	1	$812.00	
End loader operator	Day	$1,106.00	1	$1,106.00	
Laborer	Day	$812.00	1	$812.00	
Lodging and Per-Diem	Day	$146.00	9	$1,314.00	
			SUBTOTAL	$10,326.00	
			TOTAL	$18,465.37	
			DAILY PRODUCTION (CY)	525	Assume 50 cubic yards per hour for 10.5 hours
			UNIT RATE (CY)	$35.17	

Place Armor (Alternatives 2 and 3)					
Assume same costs as cover/cap construction	Day	$18,465.37	1	$18,465.37	
			DAILY PRODUCTION (CY)	368	Assume 70 percent of sand cover/cap construction rate
			UNIT RATE (CY)	$50.25	

Dredge Sediments "In the Wet" and Dewater (Alternative 4)					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Equipment					
Telehandler	Day	$567.00	1	$567.00	Move equipment around Site
Dredge excavator	Day	$1,265.00	1	$1,265.00	Dredge sediments
Clamshell bucket	Day	$70.00	2	$140.00	
RTK GPS equipment for dredge	Day	$350.00	1	$350.00	
Dredge barge	Day	$355.00	1	$355.00	With spuds and winches
Dredge tug	Day	$551.00	1	$551.00	To transport hopper barges
Material transport barges (2)	Day	$129.00	2	$258.00	25 cubic yard capacity hopper barges
Work boat	Day	$373.00	1	$373.00	
Survey vessel with equipment	Day	$1,500.00	1	$1,500.00	Track progress and QA/QC data
Upland crane	Day	$1,545.24	1	$1,545.24	Unload dredged sediments
End loader	Day	$580.00	1	$580.00	Stabilize sediments and load into trucks
Work trucks	Day	$97.00	3	$291.00	Site supervisor, foreman, mechanic
Storage silo	Day	$100.00	1	$100.00	Storage of Portland cement
Portland cement	Day	$120.00	44.1	$5,292.00	6 percent by weight; sediment 1.4 tons per cubic yard
			SUBTOTAL	$13,167.24	
Labor					
On-site project management	Day	1200.00	1	$1,200.00	
Foreman	Day	854	1	$854.00	
Mechanic	Day	980	1	$980.00	
Dredge operator	Day	$1,106.00	1	$1,106.00	
Boat operator	Day	$1,036.00	1	$1,036.00	
Derrick crane operator	Day	$1,106.00	1	$1,106.00	
End loader operator	Day	$1,106.00	1	$1,106.00	
Laborers (2)	Day	$812.00	2	$1,624.00	
Lodging and Per-Diem	Day	$146.00	9	$1,314.00	
			SUBTOTAL	$10,326.00	
			TOTAL	$23,493.24	
			DAILY PRODUCTION (CY)	525	2 25-cubic yard hopper barges filled per hour
			UNIT RATE (CY)	$44.75	

Appendix B: Table 2
Unit Rate Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Excavate Sediments "In the Dry" (Alternative 4)					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Equipment					
Excavator	Day	$1,265.00	2	$2,530.00	Excavate sediments; load trucks
Off-road dump trucks (2)	Day	$1,445.00	2	$2,890.00	Transport excavated sediments to stabilization area
End loader	Day	$580.00	1	$580.00	Mix in amendments, move crane mats, support
Crane mats	Day	$1,000.00	1	$1,000.00	River bottom haul road for dump trucks
Telehandler	Day	$567.00	1	$567.00	Unload and move super sacks and other materials/equipment
Work trucks	Day	$97.00	3	$291.00	Project manager, foreman, mechanic
Site generator	Day	$562.29	1	$562.29	Power office trailers
			SUBTOTAL	$8,420.29	
Labor					
On-site project management	Day	$1,200.00	1	$1,200.00	
Foreman	Day	$854.00	1	$854.00	
Mechanic	Day	$980.00	1	$980.00	
Excavator operators (2)	Day	$1,106.00	2	$2,212.00	
Off-road dump truck operators (2)	Day	$1,106.00	2	$2,212.00	
End loader operator	Day	$1,106.00	1	$1,106.00	
Laborers (3)	Day	$812.00	3	$2,436.00	Shoot grade, back up trucks, support
Lodging and per-diem	Day	$146.00	11	$1,606.00	
			SUBTOTAL	$12,606.00	
			TOTAL	$21,026.29	
			DAILY PRODUCTION (CY)	756	
			UNIT RATE (CY)	$27.81	

Construct Sand Cover Following Dredging (Alternative 4)					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Equipment					
Bulldozer	Day	$1,265.00	1	$1,265	Grade sand
End loader	Day	$580.00	1	$580	Manage any stockpiles that develop
Site generator	Day	$562.29	1	$562	Power office trailers
Pickup Trucks	Day	$97.00	3	$291	Project manager, foreman, mechanic
			SUBTOTAL	$2,698	
Labor					
On-site project management	Day	$1,200.00	1	$1,200	
Foreman	Day	$854.00	1	$854	
Mechanic	Day	$980.00	1	$980	
Bulldozer operator	Day	$1,106.00	1	$1,106	
End loader operator	Day	$1,106.00	1	$1,106	
Laborers (2)	Day	$812.00	2	$1,624	Direct trucks and shoot grade
Lodging and Per-Diem	Day	$146.00	7	$1,022	
			SUBTOTAL	$7,892	
			TOTAL	$10,590	
			DAILY PRODUCTION (CY)	756	
			UNIT RATE (CY)	$14.01	

Sediment Hauling and Landfill Disposal					
Transport sediments to landfill	Ton	$4.93	1	$4.93	
Dispose of sediments at landfill					Vonco V Landfill in Duluth
Disposal	Ton	$12.00	1	$12.00	
Environmental Fee	Ton	$0.27	1	$0.27	
Industrial Solid Waste Tax	Ton	$0.46	1	$0.46	
			UNIT RATE (TON)	$17.66	

Purchase and Import Amendment - Activated Carbon					
Purchase amendment material	Ton	$3,000.00	1	$3,000.00	Activated Carbon - Cost include transport/delivery
Import amendment material to staging area	Ton	$0.00	1	$0.00	
			UNIT RATE (TON)	$3,000.00	

Purchase and Import Amendment - Sediment					
Purchase amendment material	Ton	$4,000.00	1	$4,000.00	Sediment - Cost include transport/delivery
Import amendment material to staging area	Ton	$0.00	1	$0.00	
			UNIT RATE (TON)	$4,000.00	

Purchase and Import Sand					
Purchase sand from upland borrow source	CY	$6.90	1	$6.90	
Import sand to staging area	CY	$13.90	1	$13.90	40 mile cycle; 15 minute wait
			UNIT RATE (CY)	$20.80	

Purchase and Import Armoring					
Purchase cobble from upland borrow source	CY	$14.35	1	$14.35	
Import cobble to staging area	CY	$13.90	1	$13.90	40 mile cycle; 15 minute wait
			UNIT RATE (CY)	$28.25	

Appendix B: Table 2
Unit Rate Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Construction Quality Assurance and Oversight					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
QA/QC and federal oversight personnel	Week	$10,200.00	1	$10,200	Two staff
Lodging and per-diem	Week	$1,460.00	1	$1,460	Two staff
Truck and mileage	Week	$1,142.00	1	$1,142	Includes mileage
			UNIT RATE (WEEK)	$12,802	

Monthly Operating Expenses and Site Security					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Field Offices					
Office trailers and storage boxes (3)	Month	$942.00	1	$942.00	Includes utilities, equipment, and supplies for three units
Security Guard	Month	$17,280.00	1	$17,280.00	$40 per hour; 108 hours per week
			UNIT RATE (MONTH)	$18,000	Rounded

Surface Broadcast Amendment Material in Open Water Areas (Alt. #5)					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Equipment					
Skid steer	Day	366.00	1	$366.00	Consolidate materials on material supply barge
Barge-mounted Derrick crane	Day	466.00	1	$466.00	Load transport hopper barges
Derrick crane barge platform	Day	684.00	1	$684.00	Moored to dolphin pilings
Stone slinger and hopper	Day	508.00	2	$1,016.00	12 cubic yard capacity hopper
Placement barge	Day	129.00	2	$258.00	Carries hopper and stone slinger
Push boat	Day	373.00	2	$746.00	
Pickup trucks	Day	97.00	3	$291.00	Site supervisor, foreman, mechanic
			SUBTOTAL	$3,827.00	
Labor					
On-site project management	Day	1200.00	1	$1,200.00	
Foreman	Day	854.00	1	$854.00	
Mechanic	Day	980.00	1	$980.00	
Derrick crane/skid steer operator	Day	1106.00	1	$1,106.00	
Stone slinger operators	Day	1036.00	2	$2,072.00	
Push boat operators	Day	1036.00	2	$2,072.00	
Lodging and Per-Diem	Day	146.00	8	$1,168.00	
			SUBTOTAL	$9,452.00	
			TOTAL	$13,279.00	
			DAILY PRODUCTION (CY)	168.00	
			UNIT RATE (CY)	$79.04	

Appendix B: Table 3
Lump Sum Costs
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Lump Sum Costs - Alternative 1: No Action

No lump sum costs associated with Alternative 1.

Lump Sum Costs - Alternative 2: Enhanced MNR with Thin-Layer Amended Cover

Description	Unit	Unit Cost	Quantity	Extended	Comments
Mobilization/Demobilization					
Office trailers (3) and connex boxes to staging area	Mile	12.26	240	$2,942.40	Within 20 miles of site
Telehandler	Each	$1,914.00	1	$1,914.00	Within 20 miles of site
Excavator or Derrick crane (2)	Each	$1,914.00	2	$3,828.00	Within 20 miles of site
Barge platform	Each	$1,914.00	1	$1,914.00	250 miles away
Tug	Each	$1,914.00	1	$1,914.00	250 miles away
Material transport barges (2)	Each	$1,914.00	2	$3,828.00	250 miles away
Pickup trucks (3)	Mile	$0.56	1500	$840.00	250 miles each way
Additional mileage for non-local equipment	Mile	$2.52	2000	$5,040.00	Assume 4 loads non-local; 250 miles away
Receive, assemble, and launch equipment	Day	$18,465.37	3	$55,396.11	Includes travel time, assume 2.5 days
Demobilization activities	Day	$18,465.37	3	$55,396.11	Includes travel time, assume 2.5 days
				$133,000.00	Rounded
Construct Staging Area					
Clear and grub staging area	Acre	$10,488.85	3	$31,466.55	Assume 2 acres for laydown and 1 acre total along roadways
Construct haul roads	SY	$13.38	4000	$53,520.00	8-inch crushed concrete; assume 1,800 feet of road at 20 feet wide
Construct laydown areas	SY	$8.52	9680	$82,473.60	4-inch crushed concrete; assume 2 acres
Construct site fencing	LF	$5.39	1000	$5,390.00	Assume 1000 feet
Site supervision during site work	Day	$2,540.00	10	$25,400.00	Assume 10 days for staging area construction
			TOTAL	$198,000.00	Rounded

Lump Sum Costs - Alternative 3: PBAZ Cap

Description	Unit	Unit Cost	Quantity	Extended	Comments
Mobilization/Demobilization					
Same as Alternative 2 above	Lump Sum	$133,000.00	1	$133,000.00	
Construct Staging Area					
Same as Alternative 2 above	Lump Sum	$198,000.00	1	$198,000.00	

Lump Sum Costs - Alternative 4: Sediment Dredging and Excavation

Description	Unit	Unit Cost	Quantity	Extended	Comments
Mobilization/Demobilization					
Office trailers (3) and connex boxes to staging area	Mile	$12.26	240	$2,942	
Telehandler	Each	$1,914.00	1	$1,914	
Dredge excavator	Each	$1,914.00	1	$1,914	
Dredge barge	Each	$1,914.00	1	$1,914	
Clamshell buckets	Each	$1,578.00	1	$1,578	Two per load
Work boats (2)	Each	$1,914.00	1	$1,914	Two per load
Material transport barges (2)	Each	$1,914.00	2	$3,828	One per load
Derrick crane	Each	$5,592.00	1	$5,592	
End loader	Each	$1,914.00	1	$1,914	
Storage silo	Each	$1,578.00	1	$1,578	
Off-road dump trucks	Each	$1,914.00	2	$3,828	One per load
Crane mats	Truckload	$1,914.00	8	$15,312	Assume 8 loads
Pickup trucks (3)	Mile	$0.56	1500	$840	500 miles round trip per truck
Additional mileage for non-local equipment	Mile	$2.52	2000	$5,040	Assume 4 items; 250 miles away
Receive, assemble, and launch equipment	Day	$23,493.24	3	$70,480	Includes travel time, assume 2.5 days
Demobilization activities	Day	$23,493.24	3	$70,480	Includes travel time, assume 2.5 days
			TOTAL	$191,068	
Site Work					
Clear and grub staging area	Acre	$10,489.00	4	$41,956.00	Assume 4 acres for laydown and 1 acre total along roadways
Construct haul roads	SY	$13.38	4000	$53,520.00	8-inch crushed concrete; assume 1,800 feet of road at 20 feet wide
Construct laydown areas	SY	$8.52	14520	$123,710.40	4-inch crushed concrete; assume 3 acres
Construct site fencing	LF	$5.39	1000	$5,390.00	Assume 1000 feet
Construct dewatering pad	Lump Sum	$100,000.00	1	$100,000.00	150' by 150'
Site supervision during site work	Day	$2,540.00	10	$25,400.00	Assume 10 days for staging area construction
			TOTAL	$350,000.00	Rounded

Appendix B: Table 3
Lump Sum Costs
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Construct and Remove Cofferdams (1,500 feet)					
Equipment and Labor					
Work barge	Day	$684.00	1	$684	
Spuds	Day	$204.00	1	$204	
Tug	Day	$1,127.00	1	$1,127	
Crane	Day	$1,720.08	1	$1,720	
Hammer	Day	$591.40	1	$591	
Tug captain	Day	$1,036.00	1	$1,036	12-hour workday with overtime
Crane operator	Day	$1,106.00	1	$1,106	12-hour workday with overtime
Laborer	Day	$812.00	2	$1,624	12-hour workday with overtime
				$8,092	
Installation Work Activities					
Prep/"de-prep" equipment	Day	$8,092.48	2	$16,184.96	At home location
Travel to/from Duluth; launch/pull equipment	Day	$8,092.48	3	$24,277.44	
Purchase sheet piling	LF	$150.00	1500	$225,000.00	
Haul sheet piling to Site	Truckload	$1,260.00	15	$18,900.00	Sourced 250 miles from Site; $2.52 per mile;
Unload sheet piling from trucks	Day	$8,092.48	1	$8,092.48	
Drive pilings	Day	$8,092.48	15	$121,387.20	Assume 100 feet per day; 8 hours drive pilings; 4 hours load and other; 6 2' sheets/hour
				$413,842.08	
Removal Work Activities					
Remove pilings	Day	$8,092.48	15	$121,387.20	Assume 100 feet per day
Load trucks	Day	$8,092.48	1	$8,092.48	
Salvage sheet piling	Lump Sum	$0.00	1	$0.00	
Haul sheet piling for recycling	Truckload	$1,914.00	15	$28,710.00	Assume local salvage
				$158,190	
			TOTAL PROJECT COST	$572,031.76	Rounded
Dewater Area Within Cofferdam					
Pumps	Day	$815.00	2	$1,630.00	1500 GPM pump operating at 1000 GPM; 24-hour operation
Controls	Day	$200.71	1	$200.71	
Generator	Day	$2,095.80	1	$2,095.80	Large 250kW diesel generator
1000' 6" Hose	Day	$134.00	2	$268.00	
Operator	Day	$1,036.00	2	$2,072.00	2 shifts per day; 12 hours per shift
Laborer	Day	$812.00	2	$1,624.00	
			DAILY TOTAL	$7,890.51	3 million gallons over 24 hours; 7 days required
			PUMPING DAYS REQUIRED	6	
			SETUP/BREAKDOWN DAYS	2	
			TOTAL COST	$63,124.11	

Treat Water from Within Cofferdam

Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Mobilization/Demobilization	Lump Sum	$71,375.00	1	$71,375.00	Includes procurement, travel, assembly, and disassembly time
Equipment	Month	$23,940.00	8	$191,520.00	Assume 8 months
Materials	Lump Sum	$26,330.00	2	$52,660.00	Assume 1 changeout
Disposal	Lump Sum	$12,860.00	2	$25,720.00	Assume 1 changeout
Plant operator	Month	$21,756.00	8	$174,048.00	Assume 6 months
Plant laborer	Month	$17,052.00	8	$136,416.00	Assume 6 months
Labor, per-diem, and mileage	Month	$6,132.00	8	$49,056.00	Assume 6 months
			TOTAL	$700,795.00	

Debris Removal

Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Cost of labor and equipment for sediment excavation	Day	21026	3	$63,078.00	Assume three days for debris removal

Turbidity Controls					
Turbidity controls during sediment dredging	SF	7.6	10280	$78,128.00	50' radius around dredge; 200' section downstream, 20' deep
Construction Monitoring and Sample Analysis					
Air Monitoring	Week	$800.00	16	$12,800.00	Sediment excavation and stabilization area; active excavation timeframe only
Turbidity Monitoring	Week	$500.00	4	$2,000.00	Two buoys and software; dredging duration
Pre- and Post-Construction Soil Sampling					
Dioxins/Furans (EPA 8290A)	Per Sample	$595.00	14	$8,330.00	One composite sample per 1/4 acre, 4 grabs/composite
Mercury* (EPA 7471B)	Per Sample	$28.00	14	$392.00	One composite sample per 1/4 acre, 4 grabs/composite
Treated Discharge Water Sampling					
TSS (SM 2540 D)	Per Sample	$14.00	28	$392.00	1 sample per week
Dioxins/Furans (EPA 8290A)	Per Sample	$595.00	28	$16,660.00	1 sample per week
Mercury* (EPA 7471B)	Per Sample	$32.00	28	$896.00	1 sample per week
Low-level Mercury	Per Sample	$85.00	28	$2,380.00	1 sample per week
Post-Dredge Verification Sampling					
Dioxins/Furans (EPA 8290A)	Per Sample	$595.00	64	$38,080.00	One sample per 1/4 acre
Mercury* (EPA 7471B)	Per Sample	$32.00	64	$2,048.00	One sample per 1/4 acre
Dewatered Sediment Sampling					
TCLP Metals* (EPA 6020A/7471B)	Per Sample	$110.00	17	$1,870.00	One sample per 5,000 CY
Flash Point	Per Sample	$10.00	17	$170.00	One sample per 5,000 CY
pH (EPA 9045)	Per Sample	$10.00	17	$170.00	One sample per 5,000 CY
Paint Filter	Per Sample	$0.00	17	$0.00	One sample per 5,000 CY
			TOTAL	$86,000.00	Rounded

Appendix B: Table 3
Lump Sum Costs
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Lump Sum Costs - Alternative 5: Enhanced MNR with Broadcasted Amendment					
Description	**Unit**	**Unit Cost**	**Quantity**	**Extended**	**Comments**
Mobilization/Demobilization					
Office trailers (3) and connex boxes to staging area	Mile	12.26	240	$2,942.40	To staging area; within 20 miles of site
Skid steer	Each	$1,578.00	1	$1,578.00	To staging area
Telehandler	Each	$1,914.00	1	$1,914.00	To staging area
Hopper/conveyor	Each	$1,914.00	1	$1,914.00	To staging area
Pickup trucks (3)	Mile	$0.56	1500	$840.00	To staging area; 250 miles each way
Push boats (2)	Each	$1,914.00	1	$1,914.00	To staging area; 1 load
Derrick crane	Each	$2,796.00	1	$2,796.00	To staging area
Derrick crane barge platform	Hour	$1,634.00	4	$6,536.00	To staging area; sourced from Duluth Harbor
Stone slinger and hoppers (2)	Each	$1,914.00	1	$1,914.00	To staging area; 1 load
Placement barges (2)	Each	$1,914.00	2	$3,828.00	To staging area; 2 loads
Amphibious dump trucks (2)	Each	$11,184.00	2	$22,368.00	To staging area; assumed double cost for wide load and chase vehicles
Material supply barge	Hour	$1,634.00	4	$6,536.00	To staging area; sourced from Duluth Harbor
Large tug	Hour	$1,634.00	4	$6,536.00	To staging area; sourced from Duluth Harbor
Additional mileage for non-local equipment	Mile	$2.52	2500	$6,300.00	Assume 5 loads non-local; 250 miles away
Additional mileage for amphibious dump trucks	Mile	$5.04	2000	$10,080.00	Assume double cost; sourced from 1,000 miles away
Install staging area fencing	LF	$5.39	1500	$8,085.00	Install fencing around staging area perimeter
Assemble and launch equipment	Day	$10,000.00	1	$10,000.00	Half day each mob/demob
Mobilize equipment from Hallett Dock #7 to Site	Day	$10,000.00	1	$10,000.00	Half day each mob/demob
Staging area setup/breakdown	Day	$10,000.00	4	$40,000.00	Setup/breakdown staging area; 2 days each
Equipment setup and breakdown	Day	$10,000.00	6	$60,000.00	Setup/breakdown equipment; 3 days each
				$206,000.00	Rounded
Install and Remove Dolphin Pilings					
Equipment and Labor					
Work barge	Day	$855.00	1	$855.00	Monthly rate times 1.25
Tug	Day	$2,985.30	1	$2,985.30	Monthly rate times 1.25
Crane	Day	$2,150.10	1	$2,150.10	Monthly rate times 1.25
Hammer	Day	$143.48	1	$143.48	Monthly rate times 1.25
Tug captain/crane operator	Day	$1,106.00	1	$1,106.00	12-hour workday with overtime
Laborers	Day	$812.00	2	$1,624.00	12-hour workday with overtime
			TOTAL DAILY COST	$8,863.88	
Installation Work Activities					
Prep/"de-prep" equipment	Day	$8,863.88	1	$8,863.88	
Travel to/from Duluth; launch/pull equipment	Day	$8,863.88	3	$26,591.63	
Travel to/from Site; drive pilings	Day	$8,863.88	1	$8,863.88	
Removal Work Activities	Lump Sum	$44,319.38	1	$44,319.38	Same costs as installation
Materials	Lump Sum	$6,000.00	1	$6,000.00	
			TOTAL PROJECT COST	$95,000.00	Rounded
Construct Staging Area					
Clear and grub staging area	Acre	$10,488.85	3	$31,466.55	Assume 2 acres for laydown and 1 acre total along roadways
Construct haul roads	SY	$13.38	4000	$53,520.00	8-inch crushed concrete; assume 1,800 feet of road at 20 feet wide
Construct laydown areas	SY	$8.52	9680	$82,473.60	4-inch crushed concrete; assume 2 acres
Construct site fencing	LF	$5.39	1000	$5,390.00	Assume 1000 feet
Site supervision during site work	Day	$2,540.00	10	$25,400.00	Assume 10 days for staging area construction
			TOTAL	$198,000.00	Rounded

Appendix B: Table 4
Monitoring Elements
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Monitoring and Evaluation Costs - Alternative 1: No Action

No monitoring and evaluation costs associated with Alternative 1.

Monitoring and Evaluation Costs - Alternative 2: Enhanced MNR with Thin-Layer Amended Cover

Monitoring Elements	Unit	Cost	Extended	Total	Comment
Monitoring and Evaluation Report	Each	$4,000.00	6	$24,000	Every 5 years for 30 years
Field Sampling	Event	$34,000.00	6	$204,000	Every 5 years for 30 years
Sample Analysis	Event	$49,427.00	6	$296,562	Every 5 years for 30 years
Dioxins/Furans (EPA 8290A)	*Sample*	*$595.00*	*12*	*$7,140.00*	*8 locations plus 4 cover samples*
Mercury (EPA 7471B)	*Sample*	*$28.00*	*12*	*$336.00*	*8 locations plus 4 cover samples*
Grain Size (ASTM D422 w/ Hydrometer)	*Sample*	*$375.00*	*4*	*$1,500.00*	*Needed for tox/bio; 4 locations*
TOC Quad Burn (EPA 9060A)	*Sample*	*$105.00*	*4*	*$420.00*	*Needed for tox/bio; 4 locations*
10-d toxicity C. tentans	*Sample*	*$1,638.00*	*4*	*$6,552.00*	*4 locations*
28-d toxicity H. azteca	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
28-d bioaccumulation	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
Dioxins (Benthic Tissue)	*Sample*	*$595.00*	*20*	*$11,900.00*	*Individual replicate analysis*
Mercury (Benthic Tissue)	*Sample*	*$100.00*	*20*	*$2,000.00*	
Lipids content (Pace SOP)	*Sample*	*$0.00*	*20*	*$0.00*	
Dioxins (Fish Tissue)	*Sample*	*$595.00*	*5*	*$2,975.00*	*Five composite samples from five species*
Mercury (Fish Tissue)	*Sample*	*$100.00*	*5*	*$500.00*	*Five composite samples from five species*
				$49,427.00	*Rounded*
				$525,000	Rounded

Monitoring and Evaluation Costs - Alternative 3: PBAZ Cap

Monitoring Elements	Unit	Cost	Extended	Total	Comment
Monitoring and Evaluation Report	Each	$4,000.00	6	$24,000	Every 5 years for 30 years
Field Sampling	Event	$34,000.00	6	$204,000	Every 5 years for 30 years
Sample Analysis	Event	$49,427.00	6	$296,562	Every 5 years for 30 years
Dioxins/Furans (EPA 8290A)	*Sample*	*$595.00*	*12*	*$7,140.00*	*8 locations plus 4 cover samples*
Mercury (EPA 7471B)	*Sample*	*$28.00*	*12*	*$336.00*	*8 locations plus 4 cover samples*
Grain Size (ASTM D422 w/ Hydrometer)	*Sample*	*$375.00*	*4*	*$1,500.00*	*Needed for tox/bio; 4 locations*
TOC Quad Burn (EPA 9060A)	*Sample*	*$105.00*	*4*	*$420.00*	*Needed for tox/bio; 4 locations*
10-d toxicity C. tentans	*Sample*	*$1,638.00*	*4*	*$6,552.00*	*4 locations*
28-d toxicity H. azteca	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
28-d bioaccumulation	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
Dioxins (Benthic Tissue)	*Sample*	*$595.00*	*20*	*$11,900.00*	*Individual replicate analysis*
Mercury (Benthic Tissue)	*Sample*	*$100.00*	*20*	*$2,000.00*	
Lipids content (Pace SOP)	*Sample*	*$0.00*	*20*	*$0.00*	
Dioxins (Fish Tissue)	*Sample*	*$595.00*	*5*	*$2,975.00*	*Five composite samples from five species*
Mercury (Fish Tissue)	*Sample*	*$100.00*	*5*	*$500.00*	*Five composite samples from five species*
				$49,427.00	*Rounded*
				$525,000	Rounded

Monitoring and Evaluation Costs - Alternative 4:Dredging with Wetland Restoration

No monitoring and evaluation costs associated with Alternative 4.

Monitoring and Evaluation Costs - Alternative 5: Enhanced MNR with Broadcast Amendment

Monitoring Elements	Unit	Cost	Extended	Total	Comment
Monitoring and Evaluation Report	Each	$4,000.00	6	$24,000	Every 5 years for 30 years
Field Sampling	Event	$34,000.00	6	$204,000	Every 5 years for 30 years
Sample Analysis	Event	$46,935.00	6	$281,610	Every 5 years for 30 years
Dioxins/Furans (EPA 8290A)	*Sample*	*$595.00*	*8*	*$4,760.00*	*8 locations*
Mercury (EPA 7471B)	*Sample*	*$28.00*	*8*	*$224.00*	*8 locations*
Grain Size (ASTM D422 w/ Hydrometer)	*Sample*	*$375.00*	*4*	*$1,500.00*	*Needed for tox/bio; 4 locations*
TOC Quad Burn (EPA 9060A)	*Sample*	*$105.00*	*4*	*$420.00*	*Needed for tox/bio; 4 locations*
10-d toxicity C. tentans	*Sample*	*$1,638.00*	*4*	*$6,552.00*	*4 locations*
28-d toxicity H. azteca	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
28-d bioaccumulation	*Sample*	*$2,013.00*	*4*	*$8,052.00*	*4 locations*
Dioxins (Benthic Tissue)	*Sample*	*$595.00*	*20*	*$11,900.00*	*Individual replicate analysis*
Mercury (Benthic Tissue)	*Sample*	*$100.00*	*20*	*$2,000.00*	
Lipids content (Pace SOP)	*Sample*	*$0.00*	*20*	*$0.00*	
Dioxins (Fish Tissue)	*Sample*	*$595.00*	*5*	*$2,975.00*	*Five composite samples from five species*
Mercury (Fish Tissue)	*Sample*	*$100.00*	*5*	*$500.00*	*Five composite samples from five species*
				$46,935.00	*Rounded*
				$510,000	Rounded

Appendix B: Table 4
Monitoring Elements
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Field Sampling Event					
Description	**Unit**	**Cost**	**Extended**	**Total**	**Comment**
Project Management	Hour	$115.00	30	$3,450.00	Project coordination
Scientist II	Hour	$84.00	10	$840.00	Field event planning and coordination
QA/QC	Hour	$94.00	20	$1,880.00	Chemical, tox/bio, tissue results
Field Sampling					
Field Labor	Person	$4,452.00	4	$17,808.00	5 hours meetings; 40 sampling; 8 mob/demob
Truck	Day	$75.00	10	$750.00	2 trucks; boat and office trailer
Mileage	Mile	$0.57	750	$423.75	
Pontoon	Day	$200.00	5	$1,000.00	
Vibracore rental	Lump Sum	$2,500.00	1	$2,500.00	Includes freight
Disposables	Lump Sum	$1,500.00	1	$1,500.00	Vibracore tubing
Office trailer	Day	$75.00	5	$375.00	
GPS	Day	$75.00	5	$375.00	
Generator	Day	$45.00	5	$225.00	
Drum	Each	$105.00	2	$210.00	
Sediment bundle	Day	$65.00	5	$325.00	
Fuel	Lump Sum	$50.00	1	$50.00	
IDW Disposal	Lump Sum	$250.00	1	$250.00	
Lodging	Night	$100.00	16	$1,600.00	
Per-Diem	Day	$35.00	20	$700.00	
			TOTAL	$34,000.00	Rounded

Bathymetric Survey Break-Down					
Parameter	**Unit**	**Cost**	**Extended**	**Total Cost**	
Daily labor cost					
Scientist III	Hour	$109	16	$1,744	Prep equipment; mob/demob; perform survey
Field Tech II	Hour	$64	16	$1,024	Prep equipment; mob/demob; perform survey
Lodging	Night	$100	2	$200	1 night each
Per-diem	Day	$36	4	$144	2 days each
Daily equipment cost					
Boat	Day	$200	2	$400	
Fuel	Day	$25	1	$25	
Multi-beam survey equipment	Day	$1,500	2	$3,000	
GPS	Day	$75	2	$150	
Truck	Day	$75	2	$150	
Mileage	Mile	$0.56	350	$196	
Data reduction/mapping	Hour	$109	20	$2,180	
GIS	Hour	$64	10	$640	
			TOTAL	$10,000	Rounded

Appendix B: Table 5
Present Value Calculations
Focused Feasibility Study
Scanlon Reservoir
Minnesota Pollution Control Agency

Discount rate used for present worth calculations:	7.00%
Present worth calculation is: [(2016 Cost)/(1.07^Event Year 1)]+[(2016 Cost)/(1.07^Event Year 2)]+…	
Year 0 is 2016.	

Alternative 1: No Action	2016 Costs	Years	Total Present Worth
No Costs Associated with this Alternative			

Alternative 2: Enhanced MNR with Thin-Layer Amended Cover	2016 Costs	Years						Total Present Worth
Construction Costs								
Mobilization/Demobilization	$133,000	1						$124,299
Construct Staging Area	$198,000	1						$185,047
Purchase Amendment Materials and Stockpile at Staging Area	$4,393,729	1						$4,106,289
Purchase Sand and Stockpile at Staging Area	$534,991	1						$499,992
Purchase Armor and Stockpile at Staging Area	$55,831	1						$52,179
Construct Cover	$934,580	1						$873,439
Place Armor	$99,303	1						$92,806
Construction Monitoring/CQA and Oversight	$179,228	1						$167,503
Monthly Operating Expenses and Site Security	$72,000	1						$67,290
Implement Institutional Controls	$20,000	1						$18,692
Long-Term Monitoring								
Monitoring and Evaluation Report	$4,000	5	10	15	20	25	30	$8,631
Field Sampling	$34,000	5	10	15	20	25	30	$73,366
Sample Analysis	$49,427	5	10	15	20	25	30	$106,654
Professional and Technical Services								
Remedial Design (6%)	$470,000	0						$470,000
Project Management and Permitting (5%)	$392,000	1						$366,355
Construction Management (6%)	$470,000	1						$439,252

Alternative 3: Potential BAZ Cap	2016 Costs	Years						Total Present Worth
Construction Costs								
Mobilization/Demobilization	$133,000	1						$124,299
Construct Staging Area	$198,000	1						$185,047
Purchase Sand and Stockpile at Staging Area	$1,823,901	1						$1,704,581
Purchase Armor and Stockpile at Staging Area	$54,938	1						$51,344
Construct Cap	$3,084,159	1						$2,882,392
Place Armor	$97,714	1						$91,321
Construction Monitoring/CQA and Oversight	$396,862	1						$370,899
Monthly Operating Expenses and Site Security	$144,000	1						$134,579
Implement Institutional Controls	$20,000	1						$18,692
Long-Term Monitoring								
Monitoring and Evaluation Report	$4,000	5	10	15	20	25	30	$8,631
Field Sampling	$34,000	5	10	15	20	25	30	$73,366
Sample Analysis	$49,427	5	10	15	20	25	30	$106,654
Professional and Technical Services								
Remedial Design (6%)	$486,000	0						$486,000
Project Management and Permitting (5%)	$405,000	1						$378,505
Construction Management (6%)	$486,000	1						$454,206

Alternative 4: Sediment Dredging and Excavation	2016 Costs	Years						Total Present Worth
Construction Costs								
Mobilization/Demobilization	$191,068	1						$178,568
Site Work	$350,000	1						$327,103
Install and Remove Cofferdam	$572,032	1						$534,609
Dewater Excavation Area	$63,124	1						$58,994
Treat Excavation Area Water and Infiltration/Precipitation	$700,795	1						$654,949
Debris Removal	$63,078	1						$58,951
Dredge Sediments "In the Wet"	$377,104	1						$352,434
Excavate Sediments "In the Dry"	$1,650,733	1						$1,542,741
Sediment Hauling and Landfill Disposal	$2,094,150	1						$1,957,150
Purchase Sand and Stockpile at Staging Area	$271,923	1						$254,134
Construct Sand Cover "in the Wet"	$68,400	1						$63,925
Construct Sand Cover "in the Dry"	$155,888	1						$145,689
Turbidity Controls	$78,128	1						$73,017
Restore Wetland Areas	$16,880	1						$15,776
Construction Monitoring/CQA and Oversight	$358,456	1						$335,006
Monthly Operating Expenses and Site Security	$144,000	1						$134,579
Construction Monitoring and Sample Analysis	$86,000	1						$80,374
Monthly Operating Expenses and Site Security	$126,000	1						$117,757
Professional and Technical Services								
Remedial Design (6%)	$550,000	0						$550,000
Project Management and Permitting (5%)	$460,000	1						$429,907
Construction Management (6%)	$550,000	1						$514,019

Alternative 5: Enhanced MNR with Thin-Layer Amended Cover	2016 Costs	Years						Total Present Worth
Construction Costs								
Mobilization/Demobilization	$133,000	1						$124,299
Construct Staging Area	$198,000	1						$185,047
Install and Remove Dolphin Pilings	$95,000	1						$88,785
Purchase Amendment Materials and Stockpile at Staging Area	$2,042,280	1						$1,908,673
Broadcast Amendment	$67,264	1						$62,864
Construction Monitoring/CQA and Oversight	$38,406	1						$35,893
Monthly Operating Expenses and Site Security	$18,000	1						$16,822
Implement Institutional Controls	$20,000	1						$18,692
Long-Term Monitoring								
Monitoring and Evaluation Report	$4,000	5	10	15	20	25	30	$8,631
Field Sampling	$34,000	5	10	15	20	25	30	$73,366
Sample Analysis	$46,935	5	10	15	20	25	30	$101,277
Professional and Technical Services								
Remedial Design (6%)	$203,000	0						$203,000
Project Management and Permitting (5%)	$170,000	1						$158,879
Construction Management (6%)	$203,000	1						$189,720

Appendix C

Benchscale Treatability Testing Report, February 2020

February 2020
Research and Development Pilot Project Design for Remediation of Contaminated Sediments at the Scanlon Reservoir, Scanlon, Minnesota

ANCHOR QEA Baird Joint Venture

Contract Number: W912P4-16-D-0001

Benchscale Treatability Testing Report

Prepared for U.S. Army Corps of Engineers – Detroit District

DRAFT

February 2020
Research and Development Pilot Project Design for Remediation
of Contaminated Sediments at the Scanlon Reservoir,
Scanlon, Minnesota

Contract Number: W912P4-16-D-0001

Benchscale Treatability Testing Report

Prepared for
U.S. Army Corps of Engineers – Detroit District
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Project Number: 192041-01.01

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TABLE OF CONTENTS

TABLES

FIGURES

APPENDICES

ABBREVIATIONS

^{13}C	carbon-13
µm	micrometer
AC	activated carbon
ASTM	ASTM International
cm	centimeter
dioxin/furan	polychlorinated dibenzo dioxin and furan
DSR	*Pre-Remedial Design Data Summary Report*
EGL	Environmental Geochemistry Laboratory
EPA	U.S. Environmental Protection Agency
f_e	fraction of equilibrium
$f_{e,PRC}$	fraction of PRC equilibrium
GAC	granular activated carbon
HOC	hydrophobic organic contaminant
HPLC	high-performance liquid chromatography
JV	Anchor QEA-Baird Joint Venture
KM	Kaplan-Meier
K_{ow}	octanol-water partitioning coefficient
K_{PE-W}	LDPE-water partitioning coefficient
LDPE	low-density polyethylene
mg	milligram
NA	not available
NaN_3	sodium azide
NC	not calculated
ng/g	nanograms per gram
OCDD	octachlorodibenzo-p-dioxin
OCDF	octachlorodibenzofuran
PAC	powdered activated carbon
PDI	pre-remedial design investigation
PDI Workplan	*Pre-Remedial Design Investigation Workplan*
PE	polyethylene
pg/L	picograms per liter
PRC	performance reference compound
QA/QC	quality assurance/quality control
QAPP	*Quality Assurance Project Plan*
RPD	relative percent difference
SC	soot carbon

SGS	SGS North America Inc.
Site	Scanlon Reservoir Site
TEF	toxic equivalency factor
TEQ	toxic equivalent
TOC	total organic carbon
Treatability Report	*Benchscale Treatability Testing Report*
Treatability Testing Workplan	*Benchscale Treatability Testing Workplan*
USACE	U.S. Army Corps of Engineers

1 Introduction

This *Benchscale Treatability Testing Report* (Treatability Report) has been prepared by the Anchor QEA-Baird Joint Venture (JV), on behalf of the U.S. Army Corps of Engineers (USACE), Detroit District, as required under the USACE Contract Number W912P4-16-D-0001. This Treatability Report describes the methodology and results from a treatability study for amendment application to contaminated sediment at the Scanlon Reservoir (Site) of the St. Louis River located in Scanlon, Minnesota. The treatability study was conducted to evaluate the effectiveness of activated carbon (AC) to reduce the bioavailability of polychlorinated dibenzo dioxins and furans (dioxins/furans) in surface sediments at the Site. Sediment sampling procedures and minor deviations from the *Pre-Remedial Design Investigation Workplan* (PDI Workplan; JV 2019a) are presented in the *Pre-Remedial Design Data Summary Report* (DSR; JV 2019b). Treatability testing was performed in Anchor QEA, LLC's Environmental Geochemistry Laboratory (EGL) in Portland, Oregon, in accordance with the *Benchscale Treatability Testing Workplan* (Treatability Testing Workplan; JV 2019c). Sample analyses were performed by SGS North America Inc. (SGS) in Wilmington, North Carolina.

1.1 Report Organization

This Treatability Report is organized into six sections as follows:

- Section 1 (this section) presents the report organization and treatability testing purpose and objectives.
- Section 2 presents materials and methods utilized for treatability testing, including laboratory set-up, sample collection, sample preparation, sampling procedures, sampling frequency, and initial characterization analyses.
- Section 3 discusses the laboratory data quality assessment, including quality assurance/quality control (QA/QC), data validation, and data completeness.
- Section 4 briefly summarizes the results of this study.
- Section 5 provides a summary and recommendations for future evaluations.
- Section 6 is a list of references cited in this document.

1.2 Treatability Testing Purpose and Objectives

As described in the Treatability Testing Workplan (JV 2019c), the *Final Focused Feasibility Study* (Bay West 2017) identified dioxins/furans as the primary contaminants of concern for the Scanlon Reservoir, with concentrations elevated above Minnesota Pollution Control Agency Level II Sediment Quality Targets. Additional investigations by Bay West (2017) indicated that dioxins/furans in Site sediments may bioaccumulate in fish tissue above reference levels. Dioxins/furans are hydrophobic organic contaminants (HOCs) that adsorb strongly to carbonaceous material such as natural organic matter or AC. It has been shown for polychlorinated biphenyls (another group of HOCs) that adsorption to AC is 2 to 3 orders of magnitude stronger compared to natural organic matter

(Gomez-Eyles et al. 2013). Adsorption of dioxins/furans to AC has been shown to significantly reduce sediment porewater concentrations in laboratory-scale studies (Fagervold et al. 2010; Chai et al. 2012) and in a large-scale field study (Cornelissen et al. 2012).

The objective of the treatability testing described in this report was to evaluate the effectiveness of different AC amendments and doses to reduce the bioavailability of dioxins/furans in Site sediments in order to identify optimal amendments for potential application to Scanlon Reservoir. Bioavailability was assessed based on measurements of freely dissolved concentrations of dioxins/furans in sediment porewater using polyethylene (PE) passive samplers in unamended (control) sediments compared with AC-amended sediments. The rationale and methods for carrying out the benchscale treatability testing to assess amendment effectiveness are described in the Treatability Testing Workplan (JV 2019c).

2 Materials and Methods

2.1 Sample Collection

All sediment samples used in the benchscale treatability testing were collected as part of the pre-remedial design investigation (PDI) sampling effort. Sample acquisition and processing methods are reported in the DSR (JV 2019b) and briefly summarized in Section 2.1.1. Section 2.1.2 describes the selection of the AC amendments.

2.1.1 Sediment

Ten surface sediment (0 to 15 centimeters [cm]) samples were collected from the Site between September 23 and 24, 2019. Six of these samples were collected from areas that previously contained locally elevated surface sediment dioxin/furan concentrations. After collecting the required sample mass at each of the six locations, the samples were homogenized, and aliquots were sent out for the following analyses:

- Dioxins/furans (U.S. Environmental Protection Agency [EPA] Method 1613B)
- Total mercury (EPA Method 7471B)
- Total organic carbon (TOC) (EPA Method 9060A)
- Soot carbon (SC) (EPA Method 9060A [modified])
- Moisture content (ASTM International [ASTM] D2216)
- Atterberg limits (ASTM D4318)
- Particle size (ASTM D422)
- Specific gravity (ASTM D854)
- Total solids (Standard Method 2540)

In addition to laboratory testing, an aliquot of each sediment sample was set aside for benchscale treatability testing, sealed in large Mylar bags, and sent to the EGL, in accordance with the *Quality Assurance Project Plan* (QAPP; Appendix A to the PDI Workplan; JV 2019a), pending the results of laboratory testing. At EGL, an aliquot of each sediment sample was treated with AC and allowed to mix prior to starting the benchscale treatability tests. Sample preparations are described in more detail in Section 2.5.

Following receipt of laboratory testing results, two representative sediment samples were selected for benchscale treatability testing. The sediment sample selection process is described further in Section 2.2.

2.1.2 Activated Carbon

As discussed previously, the objective of the treatability testing was to assess the effectiveness of AC amendments in reducing the bioavailability of dioxins/furans in Site sediments as measured in

porewater. Two AC particle size ranges were evaluated—a silt-sized powdered activated carbon (PAC) and a fine sand-sized granular activated carbon (GAC). Both of the AC amendments tested were bituminous coal-based.

The AC products were sourced from Calgon Carbon and are as follows:

- GAC: Calgon Carbon – TOG LF 80x325; 60 U.S. mesh/0.25 millimeter (0.5% weight), 80 U.S. mesh/0.18 millimeter (4% weight), less than 325 U.S. mesh/0.045 millimeter (10% weight)
- PAC: Calgon Carbon – VPAC-I; less than 325 U.S. mesh/0.045 millimeter (90% volume)

The basis for selection of these two products is described in more detail in the Treatability Testing Workplan.

2.2 Sample Selection

This section describes the data review process that was used to select the samples for treatability testing following receipt of the data from the PDI sampling effort. In accordance with the Treatability Testing Workplan, two of the six sediment samples submitted for treatability testing were selected following a review of chemical and physical data to select representative samples that span a range of sediment types to assess the performance of AC treatment at the Site. The data review focused primarily on bulk sediment concentrations of dioxins/furans, as well as sediment TOC and SC content.

2.2.1 Sediment Data Treatment

Prior to review of the bulk sediment data, total dioxin/furan toxic equivalents (TEQs) were calculated for the 17 2,3,7,8-substituted dioxins/furans presented in the 2005 World Health Organization toxic equivalency factors (TEFs) for protection of fish, per EPA recommendations (EPA 2010). Total TEQ levels represent the sum of each individual congener concentration multiplied by its respective TEF. Calculated total TEQs are presented in Table 1.

The total dioxin/furan TEQ results presented in this report have been transformed using a variety of data treatment techniques to address the data reported at the analytical laboratory detection limit. Kaplan-Meier (KM) statistics with Efron's adjustments were applied to all samples with reportable detected and non-detected values (Helsel 2009). In instances where a sample had all non-detect values or all non-detect and not reportable values, KM statistics do not apply, and a KM value could not be calculated. In addition to KM statistics, non-detected dioxin/furan congeners are also reported at the analytical laboratory detection limit, as well as substituting a value of zero. These two techniques provide the higher and lower range of possible values for the non-detected value, and therefore, the corresponding total dioxin/furan TEQ results represent the higher and lower range of possible values.

2.2.2 Sample Screening

Representative sediment samples SR-2019-04 and SR-2019-06 were selected to carry forward into benchscale treatability testing. Compared to other samples collected during the PDI, these samples contained relatively higher total TEQ levels, and spanned the general range of TOC and SC content measured at the Site (Table 1). TOC and SC are common, naturally occurring sorbent phases that can affect the bioavailability of dioxins/furans in sediments. The TOC and SC contents of sample SR-2019-04 was generally typical of PDI samples collected from the Site, while sample SR-2019-06 contained the highest TOC and SC contents (8.1% and 2.25%, respectively). These data suggest a greater untreated sediment dioxin/furan sorption potential in sample SR-2019-06 compared to the others, with a corresponding lower potential for sequestration by AC. Thus, sample SR-2019-06 was selected as one of the two treatability testing samples to provide a conservative representation of AC amendment effectiveness.

Figure 1 presents the total dioxin/furan TEQ concentration of each sediment sample collected during the PDI. Individual dioxin/furan congeners plotted against their corresponding fraction of total TEQ are presented in Appendix A. Because congener distributions and "fingerprints" were similar amongst the PDI samples (suggesting a likely common legacy source), fingerprint characteristics did not influence sample screening.

Table 1
Total Dioxin/Furan TEQ and Carbon Concentrations in PDI Samples

Sample	Total TEQ (ng/kg)[1]	TOC (%)	SC (%)
SR-2019-01	0.343 – 1.66	2.2	0.03
SR-2019-02	13.7 – 13.8	4.0	0.14
SR-2019-03	20.1 – 20.2	3.5	0.15
SR-2019-04	**62.0 – 62.1**	**4.6**	**0.65**
SR-2019-05	27.9 – 27.9	4.0	0.53
SR-2019-06	**29.4 – 29.4** (19.3 – 19.3)	**8.1**	**2.25**

Notes:
1. Per Section 2.2.1, total dioxin/furan TEQ results are reported with non-detect results set to zero and the analytical laboratory detection limit.

Values in **bold** were selected for the benchscale treatability test.
Values in parentheses represent field duplicate sample results.
ng/kg: nanograms per kilogram

2.3 Treatability Study Design

To assess the effectiveness of AC in reducing bioavailable dioxin/furan concentrations in sediment porewater, the selected sediment samples (SR-2019-04 and SR-2019-06) were amended with two

types of AC (PAC and GAC). The PAC amendment was applied at target AC doses of 2% and 4% by dry weight, while the GAC was amended at a target AC dose of 4% by dry weight. An unamended control sample was also prepared in parallel for each sample.

Eight unique sediment batches (4% PAC-amended, 2% PAC-amended, 4% GAC-amended, and control) were prepared. In addition, one duplicate sediment batch for SR-2019-04 was also included to assess the reproducibility of the treatability testing. Therefore, a total of nine sediment batches were assembled (as shown in Table 2).

Table 2
Sediment Batch Test Design Scenarios

Sediment Sample	Amendment	Number of Sediment Batches	Number of Deployed LDPE Strips (Set 1 and Set 2)[1]
SR-2019-04	Untreated Control	1	2
	AC 1 – 4% PAC	1	4
	AC 1 – 4% PAC (Duplicate)	1	4
	AC 2 – 4% GAC	1	4
	AC 1 – 2% PAC	1	4
SR-2019-06	Untreated Control	1	2
	AC 1 – 4% PAC	1	4
	AC 2 – 4% GAC	1	4
	AC 1 – 2% PAC	1	4

Note:
1. The number (and dimensions) of low-density polyethylene (LDPE) strips was determined after selection of test sediments. This is discussed further in Section 2.5.2.

2.4 Sediment Batch Preparation

The sediment batches were prepared as described in the Treatability Testing Workplan (JV 2019c), with some minor modifications. As stated in the Treatability Testing Workplan, bulk sediment samples were sealed in large Mylar bags and shipped on ice in coolers to EGL. Upon arrival at EGL, bulk sediment samples were homogenized within their original sample containers, and coarse rocks and debris were removed by hand.

Homogenized bulk sediment samples were then transferred to stainless-steel bowls and homogenized further and added to wide-mouth, EPA-certified, pre-cleaned glass jars (1 liter) with Teflon-lined lids. An aliquot of this sediment was removed to measure moisture content using the methods provided in ASTM D2216. The moisture content of each sediment was then used to calculate the mass of high-performance liquid chromatography (HPLC)-grade water containing 200 milligrams per liter sodium azide (NaN_3) needed to be mixed into the sediment to make a slurry with

a weight ratio of 1:2 (dry sediment to water). As stated in the Treatability Testing Workplan, NaN_3, a biocide, was added to inhibit the biodegradation of dioxins/furans during testing. AC was then added to sediment jars according to the target doses outlined in Table 2. The sediment jars were then sealed and loaded onto an orbital shaker table and gently agitated for 30 days before the deployment of low-density polyethylene (LDPE). The sediment jars were also manually shaken approximately once per day to enhance uptake of dioxins/furans onto the AC.

2.5 LDPE Passive Sampling

LDPE passive samplers were used to measure the freely dissolved concentrations of dioxins/furans in sediment porewater. Passive sampling using LDPE is a well-documented approach for measuring the freely dissolved concentrations of HOCs such as dioxins/furans in sediment porewater (EPA et al. 2017). Once deployed, these samplers absorb freely dissolved HOCs from the porewater into the sampler. Passive sampling using LDPE is a continuous sampling process, providing time-averaged concentrations of HOCs in sediment porewater. It should be noted that porewater concentrations reported in this treatability study represent time-averaged concentrations following 31 and 37 days of contact with AC-amended sediments for the Set 1 and Set 2 LDPE samplers, respectively. LDPE sheets are impregnated with isotopically labeled (carbon-13 [^{13}C]) performance reference compounds (PRCs), which are analytically noninterfering, not native to the sediment, and have similar diffusivities and partitioning properties as the target dioxins/furans. Isotropic exchange kinetics are generally assumed in the PRC approach (Ghosh et al. 2014), and the depletion rate of the PRCs during the deployment of the LDPE sheet reflects the uptake rate of a target dioxin/furan. The differences in the uptake rates of dioxin/furan congeners into the LDPE samplers can be estimated by the depletion observed in the PRC concentrations and corrected for differences in the chemical characteristics. The PRCs used in this study were selected to cover a wide range of hydrophobicity of the target dioxins/furans. During deployment, PRCs diffuse out of the LDPE samples as target dioxins/furans diffuse into the LDPE samplers. The fraction of PRC equilibrium ($f_{e,PRC}$) is determined by dividing the final, post-retrieval concentration by the initial, pre-deployment concentration. The calculated $f_{e,PRC}$ is then used to estimate the fraction of equilibrium (f_e) of the target dioxins/furans, as described in detail in Section 2.5.3.2.

2.5.1 Preparation of LDPE Sheets

The LDPE sheets (25.4 micrometers [µm] thick) used for this study were obtained from Poly-America (Grand Prairie, Texas). LDPE sheets were cut and cleaned, as described in the Treatability Testing Workplan, with minor modifications. Each LDPE sheet was cut so that it was at least 60 milligrams (mg; approximately 5 cm by 5 cm). The LDPE sheets were then cleaned by sequentially soaking in HPLC-grade toluene, hexane, methanol, and water in a glass jar on a shaker table to extract any contaminants that may interfere with subsequent analysis.

Clean LDPE sheets then were spiked with the PRC, which was done by soaking in an 80:20 volume to volume methanol/water mixture containing four different PRCs (^{13}C-1,2,7,8-TCDD, ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD) purchased from Wellington Laboratories, Inc. (Guelph, Ontario, Canada) (Booij et al. 2002). The PRCs were selected to cover a wide range of hydrophobicity of the target dioxins/furans and not to interfere with the analysis of target congeners. The LDPE sheets equilibrated with the PRC-spiked solution for 14 days on an orbital shaker table. During the spiking process, methanol in the PRC-spiked solution caused the LDPE sheet to swell (this helps increase the PRC uptake rate). Therefore, after 14 days, all LDPE sheets were removed from the PRC-spiked solution and rinsed with HPLC-grade water for a day to purge methanol from the LDPE strips and reduce the swelling. Following the PRC spiking process, three PRC-spiked LDPE strips were immediately sent to SGS for analysis of the PRCs and dioxin/furan congeners, to assess if any contamination occurred as part of the spiking process (see Section 3 for further discussion on this topic).

The remainder of the LDPE sheets from this batch (Set 1) were deployed in prepared sediment batches. Using the same procedures, a second batch of LDPE sheets (Set 2) were prepared to deploy following the retrieval of Set 1. This process is discussed further in Section 2.5.2.

2.5.2 Deployment and Retrieval

Following receipt of the sediment data, the porewater concentrations were estimated based on general equilibrium partitioning theory (Lohmann 2011) to determine appropriate LDPE masses to detect the target dioxin/furan congeners and not to significantly deplete them in sediment porewater. Four of the 60-mg LDPE strips were deployed in the AC-treated sediment jars (i.e., 2% PAC, 4% PAC, and 4% GAC), while two of the 60-mg LDPE strips were deployed in untreated sediment jars. All Set 1 LDPE strips were deployed on October 25, 2019, and retrieved on November 25, 2019, for a total of 31 days of exposure.

Upon retrieval from the sediment jars, the Set 1 LDPE strips were thoroughly rinsed with HPLC-grade water and blotted dry with Kim wipes to remove water and adhering particles. The LDPE strips were then weighed and stored in EPA-certified, pre-cleaned volatile organic compound vials. Samples were placed in a cooler with ice and shipped for overnight delivery to SGS for analysis. Following retrieval, the Set 2 LDPE strips were deployed into the same sediment batches using the same number of sheets per batch on November 27, 2019 (Table 2). The Set 2 LDPE strips were later retrieved on January 2, 2020, resulting in a total of 37 days of exposure. The procedure described above for rinsing, weighing, and packing the vials was also followed for Set 2.

All results and data analysis are discussed in Section 4.

2.5.3 Data Analysis

This section discusses data analysis associated with the following: calculating freely dissolved porewater concentrations based on measured LDPE concentrations, correcting porewater data for non-equilibrium using the PRCs, and correcting porewater data for sediment depletion.

2.5.3.1 Analysis of Dioxins/Furans in the LDPE Strips

The retrieved LDPE strips were sonicated in toluene three times to extract dioxins/furans at SGS. The extracts were concentrated and analyzed for dioxins/furans by EPA Method 1613B.

2.5.3.2 Calculation of Dioxin/Furan Porewater Concentrations

Freely dissolved porewater dioxin/furan concentrations are calculated using the laboratory-measured LDPE dioxin/furan concentration, the LDPE-water partitioning coefficient (K_{PE-W}), and the f_e achieved within the LDPE strips, as shown in Equation 2-1:

Equation 2-1

$$C_w = \frac{C_{PE}}{K_{PE-W} \times f_e}$$

where:

C_w	=	Concentration in sediment porewater
C_{PE}	=	Concentration in LDPE sampler
K_{PE-W}	=	LDPE-water partitioning coefficient
f_e	=	Fraction of equilibrium of target dioxins/furans

Published K_{PE-W} values are not available for all target dioxin/furan congeners, but K_{PE-W} values can be predicted from the octanol-water partitioning coefficient (K_{ow}) using a linear regression. Adams et al. (2007) published a regression of the logarithm of measured K_{PE-W} (log K_{PE-W}) against published logarithms of the octanol-water partitioning coefficient (log K_{ow}); this regression is presented as Equation 2-2. Using Equation 2-2, the log K_{PE-W} value for each dioxin/furan congener was calculated based on its log K_{ow} value. The log K_{ow} and calculated log K_{PE-W} values for the target dioxins/furans and PRCs used in the calculation of C_w are presented in Table 3.

Equation 2-2

$$\log K_{PE-W} = 1.13 \times \log K_{OW} - 0.86 \qquad (r^2 = 0.89)$$

where:

K_{PE-W} = LDPE-water partitioning coefficient

K_{ow} = Octanol-water partitioning coefficient

Table 3
Log K_{ow} and Calculated Log K_{PE-W} of the Target Dioxin/Furan Congeners and PRCs

Type	Congener	Log K_{ow}[1]	Calculated Log K_{PE-W}[2]
Target Analyte	2,3,7,8-TeCDD	6.96	7.00
	1,2,3,7,8-PeCDD	7.50	7.59
	1,2,3,4,7,8-HxCDD	7.94	8.11
	1,2,3,6,7,8-HxCDD	7.98	8.16
	1,2,3,7,8,9-HxCDD	8.02	8.20
	1,2,3,4,6,7,8-HpCDD	8.40	8.65
	OCDD	8.60	8.89
	2,3,7,8-TCDF	6.96	6.95
	1,2,3,7,8-PeCDF	6.99	6.99
	2,3,4,7,8-PeCDF	7.50	7.59
	1,2,3,4,7,8-HxCDF	7.94	8.11
	1,2,3,6,7,8-HxCDF	7.57	7.67
	1,2,3,7,8,9-HxCDF	7.76	7.90
	2,3,4,6,7,8-HxCDF	8.02	8.20
	1,2,3,4,6,7,8-HpCDF	8.40	8.65
	1,2,3,4,7,8,9-HpCDF	8.25	8.48
	OCDF	8.60	8.89
PRCs	^{13}C-1,2,7,8-TCDD	6.99	7.04
	^{13}C-1,2,4,7,8-PeCDD	7.36	7.46
	^{13}C-1,2,3,4,6,8-HxCDD	7.77	7.92
	^{13}C-1,2,3,4,6,7,9-HpCDD	8.25	8.46

Notes:
1. Cited from Govers and Krop (1998)
2. Calculated using Equation 2-2 cited from Adams et al. (2007)
OCDD: octachlorodibenzo-p-dioxin
OCDF: octachlorodibenzofuran

The $f_{e\text{-}PRC}$ of the four PRCs was calculated using the ratio of the final PRC concentration (i.e., following deployment) to the initial PRC concentration, as shown in Equation 2-3. Subsequently, the calculated $f_{e,PRC}$ and log $K_{PE\text{-}W}$ values (Table 3) were plotted and a linear regression was developed for each sample as described in Apell et al. (2018) for well-mixed systems (Appendix B). The linear regression and the log $K_{PE\text{-}W}$ values of the target compounds were used to calculate the fraction of equilibrium of the target dioxins/furans ($f_{e,}$) (Equation 2-4). Finally, f_e was used to calculate the freely dissolved equilibrium porewater concentration (C_w) for each congener using Equation 2-1.

Equation 2-3

$$f_{e,PRC} = 1 - \frac{C_{PRC,final}}{C_{PRC,init}}$$

where:

$f_{e,PRC}$	=	Fraction of equilibrium of PRCs
$C_{PRC,final}$	=	Final PRC concentration in LDPE sampler
$C_{PRC,init}$	=	Initial PRC concentration in LDPE sampler

Equation 2-4

$$f_e = a \times logK_{PE-W} + b$$

where:

f_e	=	Fraction of equilibrium of target dioxins/furans
$K_{PE\text{-}W,\ PRC}$	=	LDPE-water partitioning coefficient
a	=	Slope of linear regression curve (Appendix B)
b	=	Y-intercept of linear regression curve (Appendix B)

3 Laboratory Data Quality Assessment

3.1 Quality Assurance/Quality Control

3.1.1 LDPE Passive Sampler Data

A method blank, PRC-loaded LDPE passive sampler reproducibility standards, and laboratory duplicates were prepared to assess the data quality of LDPE passive sampling for both Set 1 and Set 2. Details of these QA/QC samples are summarized in the following:

- **Method blank:** A method blank was used to assess background contamination introduced to the LDPE strips during cutting and cleaning. The method blank (a 60-mg LDPE strip) was cut and cleaned with the other LDPE strips, then wrapped with aluminum foil, and stored in an air-tight bag in a refrigerator at 4°C until the other LDPE strips were deployed in the benchscale treatability test. Following the start of the treatability test, the method blank sample was shipped to SGS for analysis. No target or PRC compounds were detected in the method blanks of Set 1 and Set 2.
- **Laboratory duplicates:** One laboratory duplicate was included in each set (i.e., Set 1 and Set 2) of LDPE sampler deployment in the benchscale treatability test. An additional batch of SR-2019-04 amended with 4% PAC was prepared and LDPE strips were deployed to provide a measure of experimental reproducibility. The relative percent difference (RPD) for the Set 1 sampler uptake for the two identical batches was 25%, and the RPD for the freely dissolved concentrations corrected for the fraction of PRC loss was 4%, both within the QAPP data quality objective. The RPD for the Set 2 sampler uptake was 16% and the RPD for the freely dissolved concentrations corrected for the fraction of PRC loss was 36%, both within the QAPP data quality objective.

3.2 Data Validation

Data quality criteria and data validation procedures are provided in the QAPP. Data from each laboratory package were evaluated and documented in a data validation report by the JV. Data validation reports are provided in Appendix C. All data qualifiers applied to the data during final validation have been incorporated into the database for this project. Most data were acceptable as reported, and all other data were acceptable as qualified. The data qualifier "U" was assigned to various results during validation to indicate the associated numerical value was non-detect at or above the specified limit. The data qualifier "J" was assigned to various results during data validation to indicate the associated numerical value is an estimated concentration. All dioxin/furan data that were qualified by the laboratory as estimated maximum possible concentration were assigned "J" qualifiers to indicate a detected and estimated concentration. Other results were assigned a "J"

qualifier based on a method or technical criterion, as stated in the EPA National Functional Guidelines (EPA 2016) or the QAPP.

Overall, reporting limits were deemed acceptable to meet project objectives and reporting limits, because undetected results were met or below those specified for the project.

4 Results

4.1 Bulk Sediment Results

The results of the TEQ concentrations in the bulk sediments are presented in Table 1. Additional physical parameters such as grain size, plasticity, liquid limits, and specific gravity are presented in Table 1 of the DSR (JV 2019b). The sediments collected for the benchscale treatability test predominately comprised fine to coarse sands and silt (89% to 93%). The clay fraction of the samples ranged from 6.9% to 10%, and gravels were measured in two samples SR-2019-02 (0.1%) and SR-2019-04 (0.4%). The TOC and SC content of the samples are discussed in Section 2.2.2.

As shown in Appendix A, the two predominant TEQ congeners in the PDI sediment samples were 1,2,3,7,8-PeCDD and 1,2,3,4,6,7,8-HpCDF, representing approximately half of the total TEQ. As discussed in Section 2.2, representative samples SR-2019-04 and SR-2019-06 were selected for benchscale treatability testing.

4.2 Sediment Porewater Results

4.2.1 Performance Reference Compounds

Low variability in initial PRC concentrations is a key step in accurately characterizing the fraction of equilibrium of a target dioxin/furan congener. To assess variability, the four ^{13}C-labeled dioxin/furan congeners were spiked into QA/QC LDPE samplers, along with the samplers deployed in the treatability test. The initial PRC concentrations in the Set 1 QA/QC samplers (i.e., PRC-loaded LDPE sampler reproducibility standards) had low variability, with a 1.0% to 5.3% coefficient of variation. The initial PRC concentrations in the Set 2 QA/QC samplers had 1.5% to 28.4% coefficient of variation, with only the highest K_{ow} PRC (^{13}C-1,2,3,4,6,7,9-HpCDD) having a coefficient of variation above 6%. The increase in variability from Set 1 to Set 2 is likely the result in a smaller number of initial PRC samples (two initial PRC samples in Set 2 as opposed to three initial PRC samples in Set 1). For the Set 2 initial PRC samples, one of the three samples was rejected because the initial concentrations were much lower than those of the other two samples. The sensitivity analysis indicated that rejecting the PRC sample did not change the estimated porewater concentrations of dioxins/furans (less than 5% differences). The two remaining PRC samples were used to calculate the initial PRC concentrations for Set 2.

$f_{e\text{-}PRC}$ was calculated separately for each sediment batch as described in Section 2.5.3. The lower molecular weight PRCs (^{13}C-1,2,7,8-TCDD and ^{13}C-1,2,4,7,8-PeCDD) were approximately 100% dissipated from LDPE samplers after 31 and 37 days of deployment in Set 1 and Set 2, respectively. The $f_{e\text{-}PRC}$ of the higher molecular weight PRCs (^{13}C-1,2,3,4,6,8-HxCDD and ^{13}C-1,2,3,4,6,7,9-HpCDD)

were on average 50% and 15% dissipated in Set 1 and average 83% and 78% dissipated in Set 2, respectively.

On average, higher $f_{e\text{-}PRC}$ were achieved for larger molecular weight PRCs in Set 2 compared to Set 1. The increase in $f_{e\text{-}PRC}$ is likely attributable to the increased variability in the initial PRC results. However, the significant difference in $f_{e\text{-}PRC}$ values, and therefore, the f_e of the target dioxins/furans, did not result in a significant difference between the Set 1 and Set 2 freely dissolved TEQ concentrations (see Table 7 for freely dissolved TEQ concentrations). The fractions of equilibrium of the PRCs for Set 1 and Set 2 are shown in Table 4.

Table 4
Fraction of Equilibrium of the PRCs ($f_{e\text{-}PRC}$) Achieved in Set 1 LDPE Samplers and Set 2 LDPE Samplers

PRCs	SR-2019-04					SR-2019-06			
	Control	4% PAC	4% PAC (Dup.)	2% PAC	4% GAC	Control	4% PAC	2% PAC	4% GAC
Set 1 (60 Days)									
^{13}C-1,2,7,8-TCDD[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
^{13}C-1,2,4,7,8-PeCDD	0.98	0.98	0.98	0.98	0.98	0.97	0.96	0.96	1.00
^{13}C-1,2,3,4,6,8-HxCDD	0.76	0.50	0.52	0.58	0.58	0.61	0.31	0.33	0.26
^{13}C-1,2,3,4,6,7,9-HpCDD	0.29	0.13	0.21	0.18	0.22	0.21	0.16	0.17	0.00
Set 2 (97 Days)									
^{13}C-1,2,7,8-TCDD[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
^{13}C-1,2,4,7,8-PeCDD	0.98	0.99	0.99	0.99	0.98	0.97	0.97	0.98	0.97
^{13}C-1,2,3,4,6,8-HxCDD	0.93	0.75	0.87	0.90	0.80	0.84	0.77	0.80	0.77
^{13}C-1,2,3,4,6,7,9-HpCDD	0.84	0.63	0.87	0.88	0.67	0.78	0.79	0.81	0.76

1. Indicates a congener with a $K_{PE\text{-}W}$ less than that of ^{13}C-1,2,4,7,8-PeCDD, and therefore, was applied a fraction of equilibrium value of 1.00.

After calculating $f_{e,PRC}$, a linear regression between $f_{e,PRC}$ and log $K_{PE\text{-}W}$ was developed for each sediment batch (Appendix B), and the fraction of equilibrium of target dioxin/furan congeners in the LDPE samples was estimated for each sediment batch using Equation 2-4 (Appendix B). Because ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from the LDPE samplers in all sediment batches, the other three PRCs were used to develop the linear regression. Not including ^{13}C-1,2,7,8-TCDD greatly improved the fit of the linear regression to the dataset, and all target dioxin/furan congeners with $K_{PE\text{-}W}$ values smaller than that of ^{13}C-1,2,4,7,8-PeCDD (7.46) were assigned an $f_{e\text{-}PRC}$ of

1.00. The f_e for the remaining target dioxins/furans were estimated from the linear regression and are presented in Table 5.

Table 5
Linear Regression Between the Fraction of Equilibrium of the PRCs ($f_{e,PRC}$) and the Log of the LDPE-Water Partitioning Coefficient (Log K_{PE-W}) in Set 1 and Set 2 LDPE Samplers

Sediment	Amendment	Linear Regression (Equation 2-4: $f_{e,PRC} = a \times \log K_{PE-W} + b$)					
		Set 1 (60 Days)			Set 2 (97 Days)		
		a	b	R^2	a	b	R^2
SR-2019-04	Control	-0.69	6.14	0.98	-0.135	1.99	0.992
	4% PAC	-0.84	7.21	0.99	-0.346	3.54	0.944
	4% PAC (Duplicate)	-0.76	6.60	0.98	-0.117	1.84	0.749
	2% PAC	-0.80	6.92	1.00	-0.107	1.77	0.859
	4% GAC	-0.75	6.54	0.99	-0.302	3.22	0.975
SR-2019-06	Control	-0.76	6.60	1.00	-0.182	2.31	0.923
	4% PAC	-0.79	6.73	0.86	-0.169	2.19	0.611
	2% PAC	-0.77	6.62	0.87	-0.161	2.14	0.665
	4% GAC	-0.94	7.90	0.91	-0.204	2.46	0.737

The values of f_e for target compounds calculated from the regression equations for Set 1 and Set 2 are listed in Table 6. To avoid over-correction of the freely dissolved porewater concentrations, a freely dissolved porewater concentration was not calculated for any congener with a calculated f_e of less than 10%. Congeners with an f_e of less than 10% are indicated in Table 6 with a value of "NC" (not calculated). As shown in Equation 2-1, an f_e value of 10% or below results in a correction of greater than 1 order of magnitude. Analytical uncertainty in these data has a much larger effect on the calculated freely dissolved porewater concentration. 1,2,3,4,6,7,8-HpCDD, OCDD (octachlorodibenzo-p-dioxin), 1,2,3,4,6,7,8-HpCDF, and OCDF (octachlorodibenzofuran) were not calculated for SR-2019-04; and 1,2,3,4,6,7,8-HpCDD, OCDD, 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, and OCDF were not calculated for SR-2019-06. Freely dissolved equilibrium porewater concentrations for target dioxin/furan congeners with f_e values greater than 0.10 were calculated using Equation 2-1.

Table 6
Estimated Fraction of Equilibrium of the Target Dioxin/Furan Congeners ($f_{e\text{-}PRG}$) in Set 1 LDPE Samplers and Set 2 LDPE Samplers

Congeners	SR-2019-04					SR-2019-06			
	Control	4% PAC	4% PAC (Duplicate)	2% PAC	4% GAC	Control	4% PAC	2% PAC	4% GAC
Set 1 (60 Days)									
2,3,7,8-TeCDD[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
1,2,3,7,8-PeCDD	0.92	0.84	0.84	0.87	0.86	0.87	0.76	0.76	0.74
1,2,3,4,7,8-HxCDD	0.57	0.40	0.45	0.45	0.47	0.47	0.35	0.36	0.25
1,2,3,6,7,8-HxCDD	0.53	0.36	0.41	0.41	0.44	0.44	0.31	0.33	0.21
1,2,3,7,8,9-HxCDD	0.50	0.32	0.38	0.38	0.40	0.40	0.28	0.29	0.17
1,2,3,4,6,7,8-HpCDD	NC	NC	NC	NC	NC	NC	NC	NC	NC
OCDD	NC	NC	NC	NC	NC	NC	NC	NC	NC
2,3,7,8-TCDF[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
1,2,3,7,8-PeCDF[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
2,3,4,7,8-PeCDF	0.92	0.84	0.84	0.87	0.86	0.87	0.76	0.76	0.74
1,2,3,4,7,8-HxCDF	0.57	0.40	0.45	0.45	0.47	0.47	0.35	0.36	0.25
1,2,3,6,7,8-HxCDF	0.87	0.77	0.78	0.80	0.80	0.80	0.69	0.70	0.67
2,3,4,6,7,8-HxCDF	0.50	0.32	0.38	0.38	0.40	0.40	0.27	0.29	0.17
1,2,3,7,8,9-HxCDF	0.71	0.58	0.61	0.62	0.63	0.63	0.52	0.53	0.45
1,2,3,4,6,7,8-HpCDF	NC	NC	NC	NC	NC	NC	NC	NC	NC
1,2,3,4,7,8,9-HpCDF	0.31	0.10	0.17	0.16	0.20	NC	NC	NC	NC
OCDF	NC	NC	NC	NC	NC	NC	NC	NC	NC
Set 2 (97 Days)									
2,3,7,8-TeCDD[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
1,2,3,7,8-PeCDD	0.97	0.91	0.95	0.96	0.93	0.93	0.91	0.92	0.87
1,2,3,4,7,8-HxCDD	0.90	0.73	0.89	0.91	0.77	0.83	0.82	0.84	0.68
1,2,3,6,7,8-HxCDD	0.89	0.72	0.89	0.90	0.76	0.83	0.81	0.83	0.66
1,2,3,7,8,9-HxCDD	0.88	0.70	0.88	0.90	0.74	0.82	0.80	0.82	0.65
1,2,3,4,6,7,8-HpCDD	0.82	0.55	0.83	0.85	0.61	0.74	0.73	0.75	0.48
OCDD	0.79	0.46	0.80	0.82	0.54	0.69	0.69	0.71	0.40
2,3,7,8-TCDF[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
1,2,3,7,8-PeCDF[1]	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
2,3,4,7,8-PeCDF	0.97	0.91	0.95	0.96	0.93	0.93	0.91	0.92	0.87

Congeners	SR-2019-04					SR-2019-06			
	Control	4% PAC	4% PAC (Duplicate)	2% PAC	4% GAC	Control	4% PAC	2% PAC	4% GAC
1,2,3,4,7,8-HxCDF	0.90	0.73	0.89	0.91	0.77	0.83	0.82	0.84	0.68
1,2,3,6,7,8-HxCDF	0.95	0.89	0.94	0.95	0.90	0.91	0.89	0.91	0.84
2,3,4,6,7,8-HxCDF	0.88	0.70	0.88	0.90	0.74	0.82	0.80	0.82	0.65
1,2,3,7,8,9-HxCDF	0.92	0.81	0.92	0.93	0.83	0.87	0.85	0.87	0.76
1,2,3,4,6,7,8-HpCDF	0.82	0.55	0.83	0.85	0.61	0.74	0.73	0.75	0.48
1,2,3,4,7,8,9-HpCDF	0.85	0.61	0.85	0.87	0.66	0.77	0.76	0.78	0.55
OCDF	0.79	0.46	0.80	0.82	0.54	0.69	0.69	0.71	0.40

Notes:
1. Indicates a congener with a K_{PE-W} less than that of ^{13}C-1,2,4,7,8-PeCDD, and therefore, was applied a fraction of equilibrium value of 1.00.

NC: not calculated

4.2.2 Dioxin/Furan Concentrations and AC Performance

KM-transformed sediment porewater total TEQ concentrations in the control and AC-amended sediments were calculated as described in Sections 2.2.1 and 2.5.3 (Table 7). TEQ concentrations in the control and AC-amended sediments for Set 1 and Set 2 samples for SR-2019-04 are presented in Figures 2 and 3, respectively; and for Set 1 and Set 2 samples for SR-2019-06, they are presented in Figures 4 and 5, respectively. As stated in Section 4.2.1, freely dissolved concentrations could not be reliably calculated for 1,2,3,4,6,7,8-HpCDD, OCDD, 1,2,3,4,6,7,8-HpCDF, and OCDF in SR-2019-04 and 1,2,3,4,6,7,8-HpCDD, OCDD, 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, and OCDF in SR-2019-06.

Table 7
Reductions of Bioavailable TEQ Concentrations Measured in Sediment Porewater in Set 1 after 60 Days and in Set 2 after 97 Days of AC Amendment

Sediment	Amendment	Set 1 (60 Days)		Set 2 (97 Days)	
		Freely Dissolved TEQ ($\times 10^{-4}$ pg/L TEQ)[1]	TEQ Reduction (%)	Freely Dissolved TEQ ($\times 10^{-4}$ pg/L TEQ)[1]	TEQ Reduction (%)
SR-2019-04	Control	86.5 – 106	NA	108 – 109	NA
	4% PAC	0.876 – 15.8 (0.982 – 13.5)	99% – 85% (99% – 87%)	0.997 – 12.0 (0.603 – 9.97)	99% – 89% (99% – 91%)
	2% PAC	4.38 – 18.1	95% – 83%	2.18 – 12.6	98% – 88%
	4% GAC	10.2 – 27.6	88% – 74%	7.14 – 20.5	93% – 81%
SR-2019-06	Control	30.2 – 49.3	NA	25.2 – 57.2	NA
	4% PAC	0 – 15.0	100% – 70%	0.0631 – 12.3	100% – 79%
	2% PAC	0.0315 – 14.5	100% – 71%	0.551 – 11.1	98% – 81%
	4% GAC	5.07 – 18.3	83% – 63%	1.86 – 14.8	93% – 74%

Notes:
1. Per Section 2.2.1, total dioxin/furan TEQ results are reported with non-detect results set to zero and the analytical laboratory detection limit.

Values in parentheses represent duplicate sample results.
NA: not available
pg/L: picograms per liter

Significant porewater concentration reductions in the amended sediments compared to the controls were observed across both Sets 1 and 2 (Figures 2 through 5).

In Set 1, the calculated freely dissolved porewater total TEQ concentrations in the control sediments ranged from 86.5 × 10^{-4} to 106 × 10^{-4} picograms per liter (pg/L) TEQ in SR-2019-04 and from 30.2 × 10^{-4} to 49.3 × 10^{-4} pg/L TEQ in SR-2019-06. Reductions in quantified total TEQ porewater concentrations ranged from 74% to 99% in SR-2019-04, and from 63% to 100% in SR-2019-06, with the 4% GAC amendment proving the lowest reductions and the 4% PAC amendment providing the highest reductions for both sediments. However, because freely dissolved concentrations of the few strongly hydrophobic congeners could not be reliably calculated, these reductions are representative of the less hydrophobic congeners.

In Set 2, freely dissolved total TEQ concentrations in the control sediments ranged from 108 × 10^{-4} to 109 × 10^{-4} pg/L TEQ for SR-2019-04 and from 25.2 × 10^{-4} to 57.2 × 10^{-4} pg/L TEQ for SR-2019-06. Reductions in total TEQ porewater concentrations ranged from 81% to 99% in SR-2019-04, and from 74% to 100% in SR-2019-06, with the 4% GAC amendment proving the lowest reductions and the 4% PAC amendment providing the highest reductions for both sediments. Freely dissolved concentrations were calculated for all congeners in Set 2.

Overall, Set 2 achieved higher TEQ reduction rates than Set 1 for all AC amendments in SR-2019-04 and SR-2019-06. Although 4% GAC achieved the lesser reduction of all amendments, more than 90% reduction was achieved in both SR-2019-04 and SR-2019-06 after 97 days of AC amendment.

To understand the effect of the AC amendments on the more hydrophobic congeners for which freely dissolved concentrations could not be reliably calculated, dioxin/furan congener concentrations in the LDPE samplers were directly compared (Table 8; Figures 6 through 9). Because all sample batches were maintained under the same experimental conditions (i.e., mixing rate, temperature), the uptake kinetics of the congeners by LDPE passive sampler during the study should be similar among the different sediment batches. The figures presented in Appendix D show the percent reduction of individual congeners, measured above the detection limit, plotted against their Log K_{ow}, for SR-2019-04 and SR-2019-06, respectively. These data indicate that the percent reduction, based on passive sampler uptake, decreases with increasing K_{ow}, which is the same trend observed in short-term porewater studies (Fagervold et al. 2010). In this case, it is reasonable to assume that reductions measured in passive sampler uptake are similar to the reductions measured in porewater. For Set 1, total TEQ reductions in the LDPE samplers ranged from 70% to 99%. For Set 2, total TEQ reductions ranged from 73% to 100% (Table 8; Figures 10 through 13). The total TEQ in LDPE sampler TEQ concentration, as shown in Table 8, are two to nine times higher (depending on which type of non-detect treatment is selected) in the 4% GAC amendment compared to the 4% PAC amendment. This would result in the need to apply two to nine times the amount of GAC to achieve the same LDPE TEQ concentrations observed in the 4% PAC amendment. Overall, both the PAC and GAC substantially reduced the dioxin/furan concentrations in each treatability testing batch.

Table 8
Reductions of TEQ Concentrations in LDPE Passive Samplers after 60 Days (Set 1) and 97 Days (Set 2) of AC Amendment

Sediment	Amendment	Set 1 (60 Days)		Set 2 (97 Days)	
		Total TEQ in LDPE (ng/g TEQ)[1]	TEQ Reduction (%)	Total TEQ in LDPE (ng/g TEQ)[1]	TEQ Reduction (%)
SR-2019-04	Control	0.402 – 0.432	NA	0.396 – 0.400	NA
	4% PAC	0.0241 – 0.0508 (0.00506 – 0.0284)	94% – 88% (99% – 93%)	0.0146 – 0.0392 (0.00195 – 0.0219)	96% – 90% (100% – 95%)
	2% PAC	0.0521 – 0.0769	87% – 82%	0.0344 – 0.0573	91% – 86%
	4% GAC	0.0964 – 0.129	76% – 70%	0.0818 – 0.107	79% – 73%
SR-2019-06	Control	0.146 – 0.178	NA	0.165 – 0.200	NA
	4% PAC	0.00277 – 0.0286	98% – 84%	0.00207 – 0.0274	99% – 86%
	2% PAC	0.00675 – 0.0330	95% – 82%	0.00766 – 0.0291	95% – 85%
	4% GAC	0.0269 – 0.0473	82% – 74%	0.0191 – 0.0421	89% – 79%

Notes:
1. Per Section 2.2.1, total dioxin/furan TEQ results are reported with non-detect results set to zero and the analytical laboratory detection limit.

Values in parentheses represent duplicate sample results.
NA: not available
ng/g: nanograms per gram

5 Summary and Recommendations

Two different types of AC amendments (PAC at 2% and 4% dose, and GAC at 4% dose) were mixed directly into Site sediments, and continuous agitation was applied to accelerate the uptake kinetics of dioxins/furans by the AC. The continuous agitation enabled the study to produce meaningful results within the project schedule constraints. The goal of the study was to evaluate the effectiveness of the AC amendments at reducing bioavailable concentrations of dioxins/furans. Freely dissolved concentrations of dioxins/furans in the sediment porewater were measured twice by LDPE passive samplers 60 and 97 days after AC amendment. The benchscale testing results are summarized as follows:

- In all the tested conditions, the freely dissolved dioxin/furan concentrations in the sediment porewater were substantially reduced by both the PAC and GAC amendments.
- Among the three amendment conditions, 4% PAC amendment was the most effective, with TEQ reductions in the freely dissolved phase of approximately 79% to 100% after 97 days.
- While the 4% GAC amendment achieved a lower TEQ reduction (TEQ reductions in the freely dissolved phase of approximately 74% to 93% after 97 days—two to nine times less effective than 4% PAC due to larger grain size and lower specific surface area), the GAC amendment performed well enough to be retained for further engineering evaluations.

The results of this benchscale treatability study are promising, indicating that different AC amendments and doses (PAC at 2% and 4% dose, and GAC at 4% dose) are likely to be effective at significantly reducing bioavailable concentration of dioxins/furans in Site sediments. Extrapolating the results of this study to assess the short- and long-term effectiveness of different AC amendment application methods requires additional calculations to be conducted in a follow-on engineering phase. Application methods will be retained for further engineering and cost evaluations.

6 References

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Figures

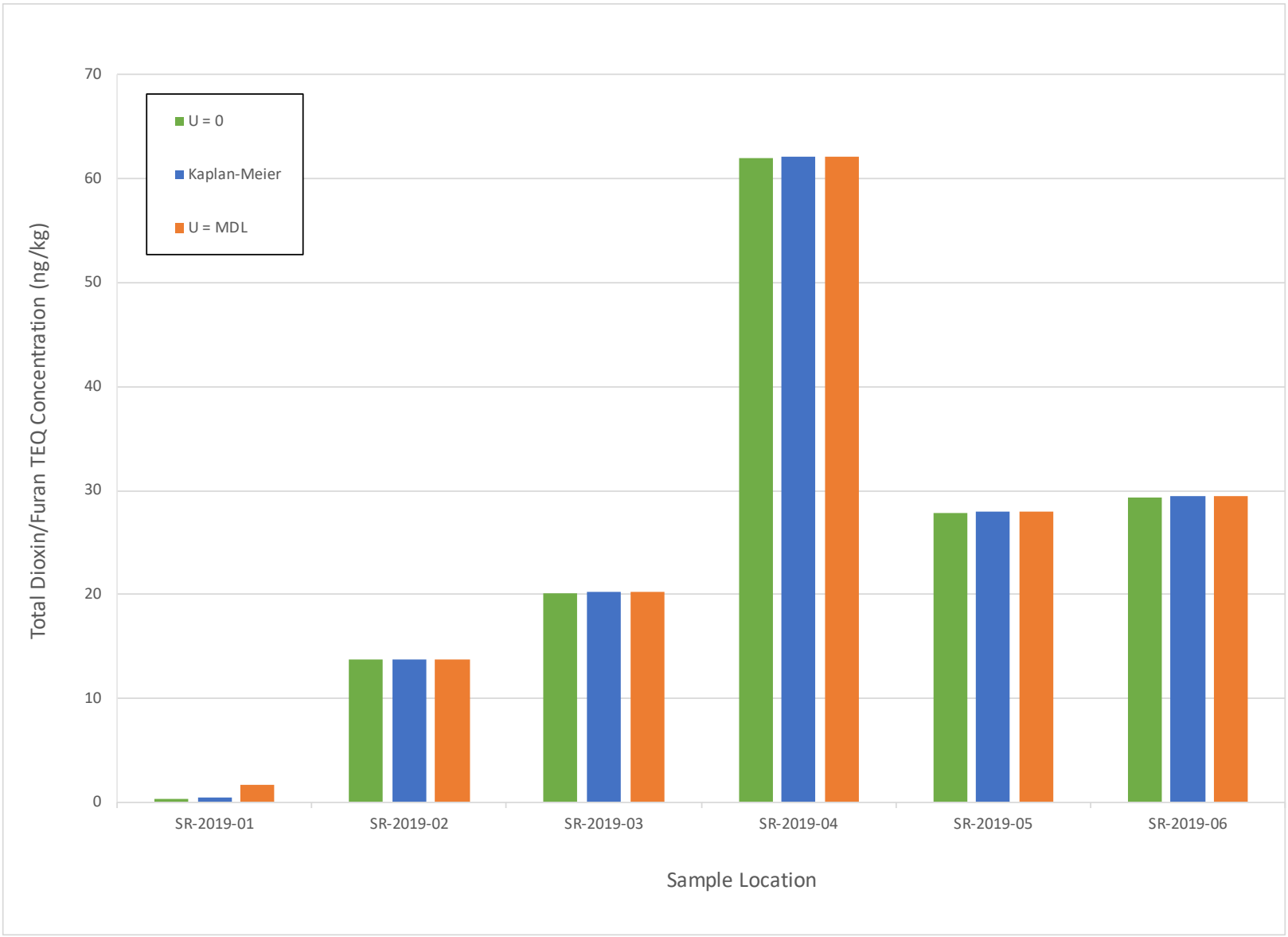

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Figure 1
Dioxin/Furan TEQ Congener Distribution in Site Sediment Samples
Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

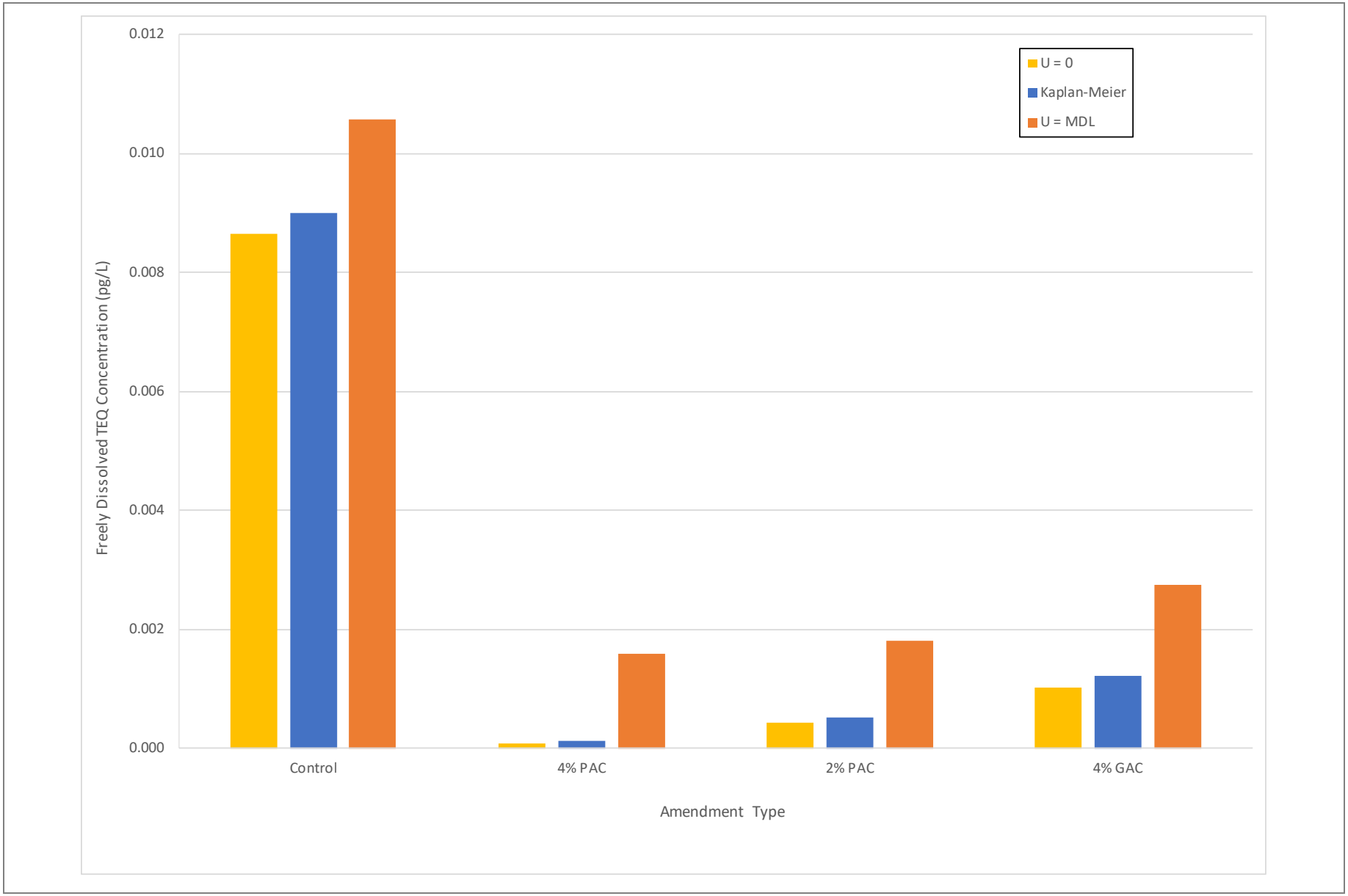

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Figure 2

Set 1 Dioxin/Furan Congener TEQ Profiles in the Sediment Porewater – SR-2019-04

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

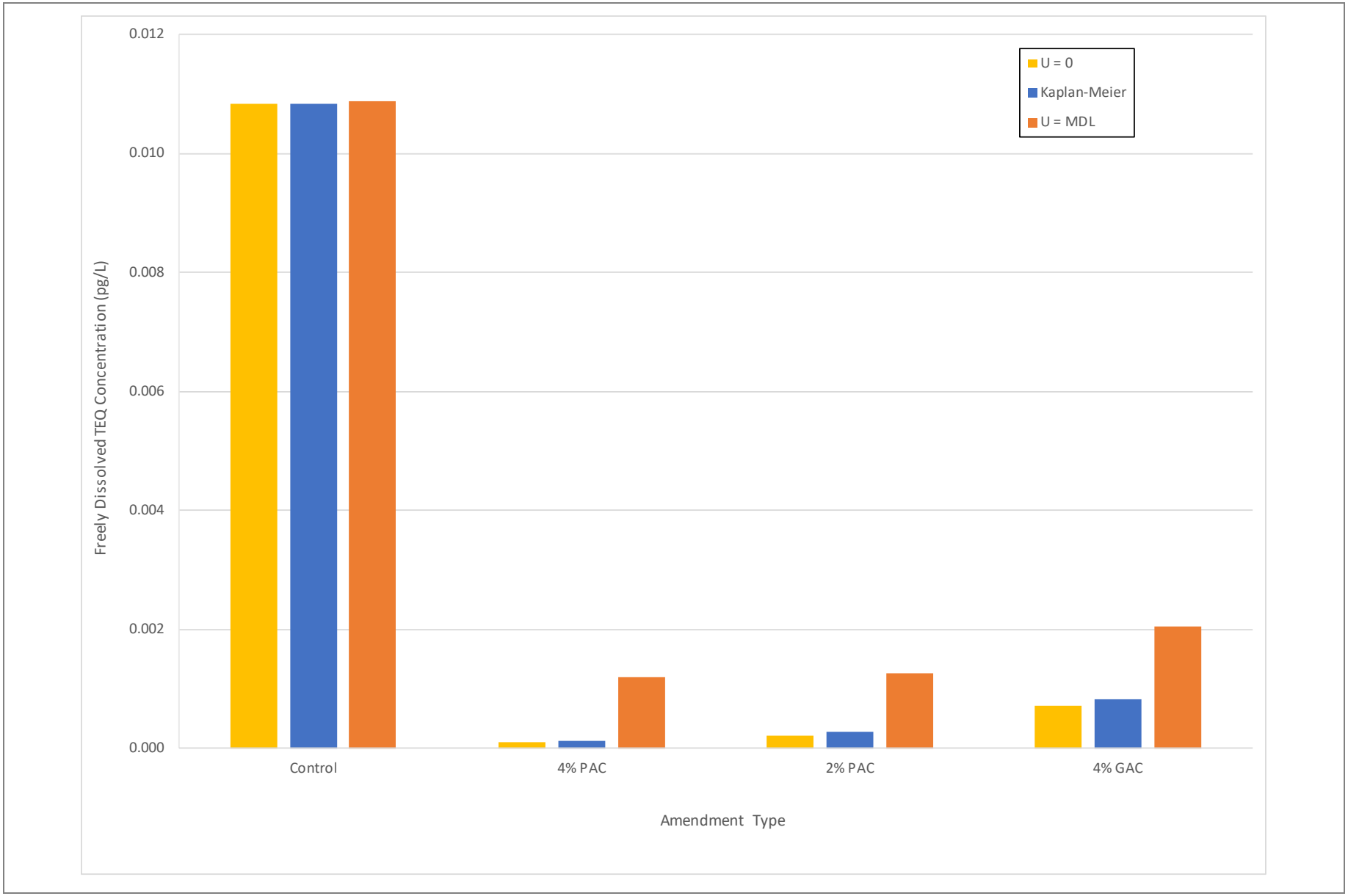

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Figure 3

Set 2 Dioxin/Furan Congener TEQ Profiles in the Sediment Porewater – SR-2019-04

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

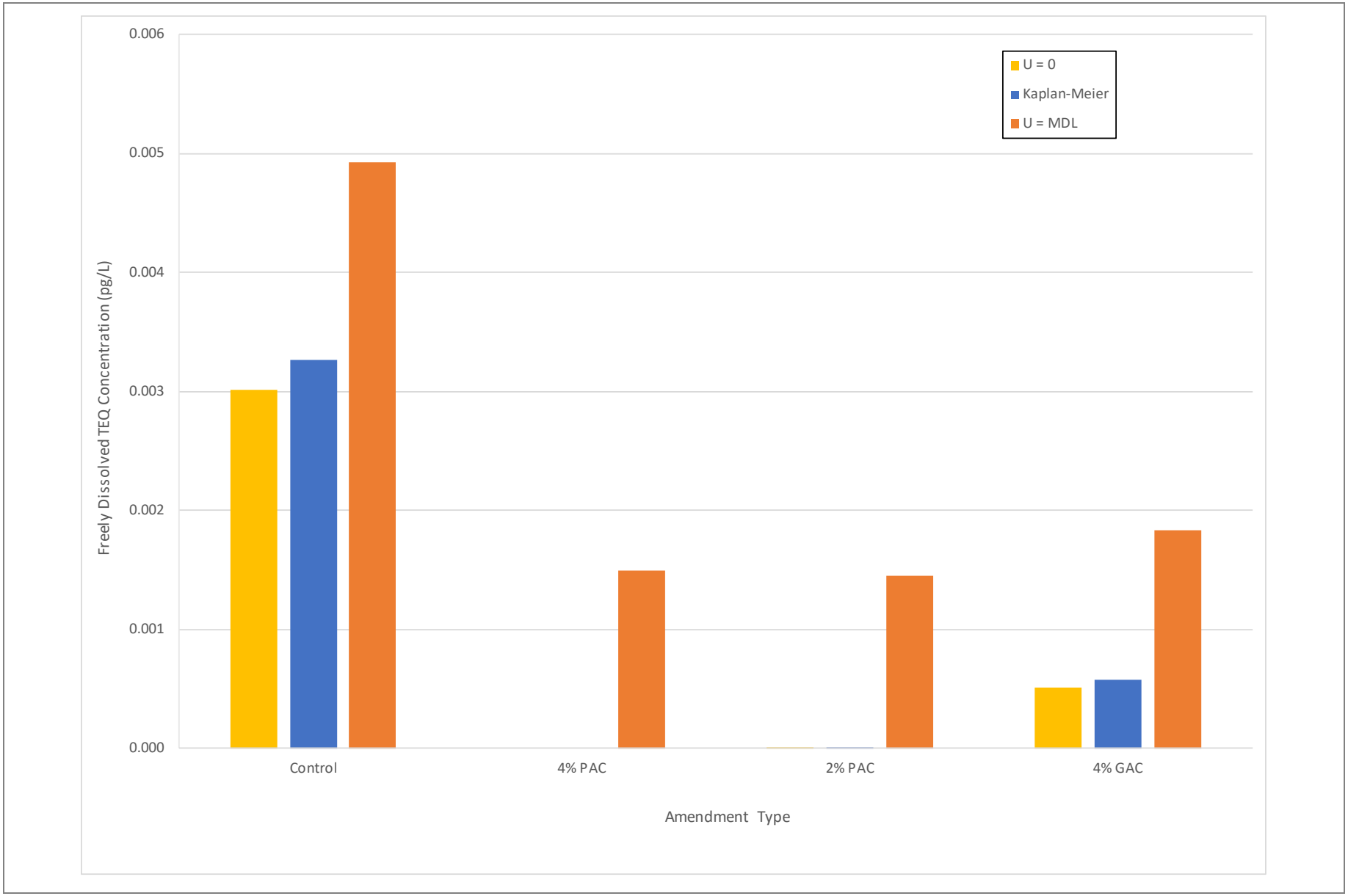

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Figure 4

Set 1 Dioxin/Furan Congener TEQ Profiles in the Sediment Porewater – SR-2019-06

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

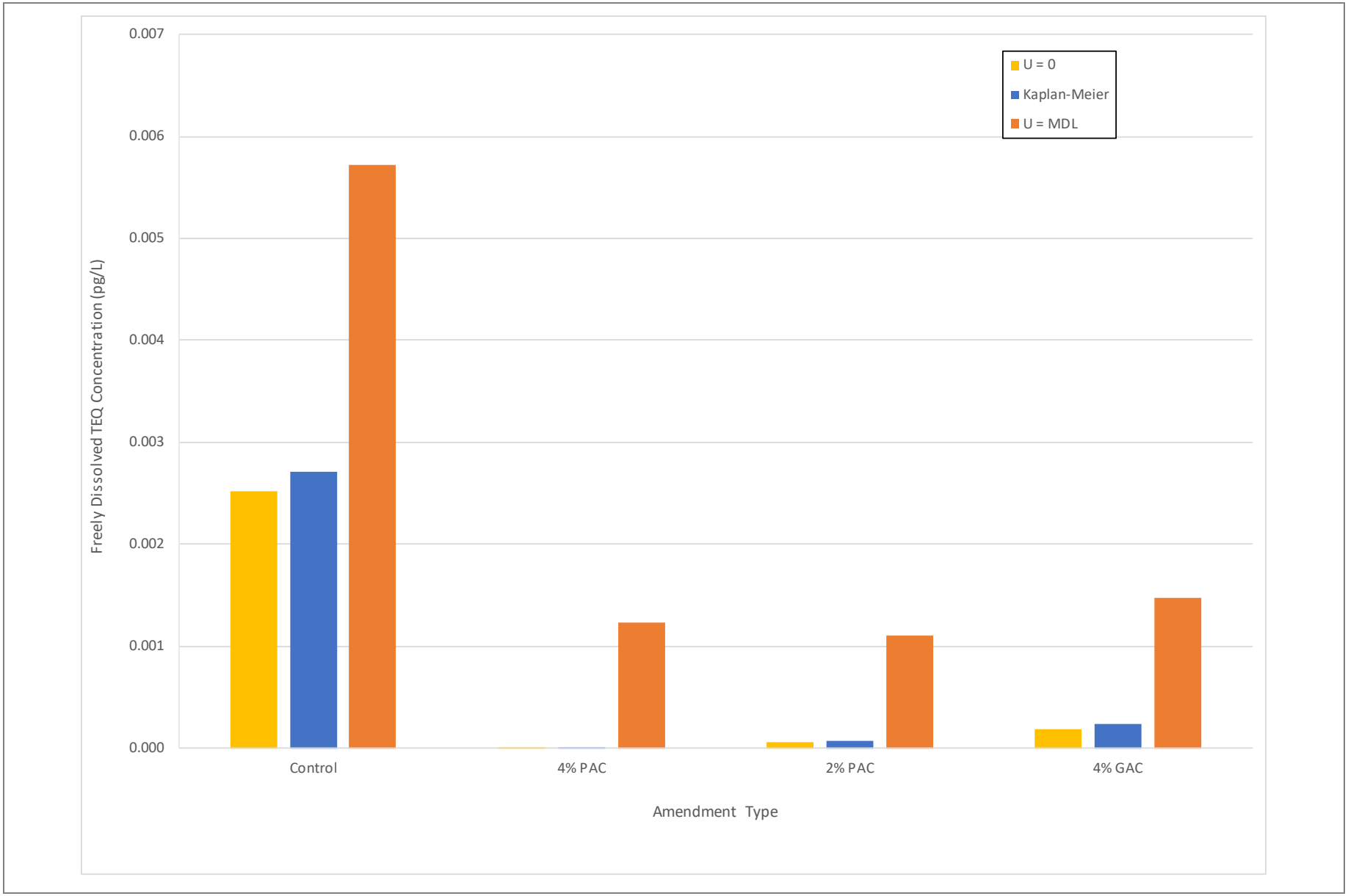

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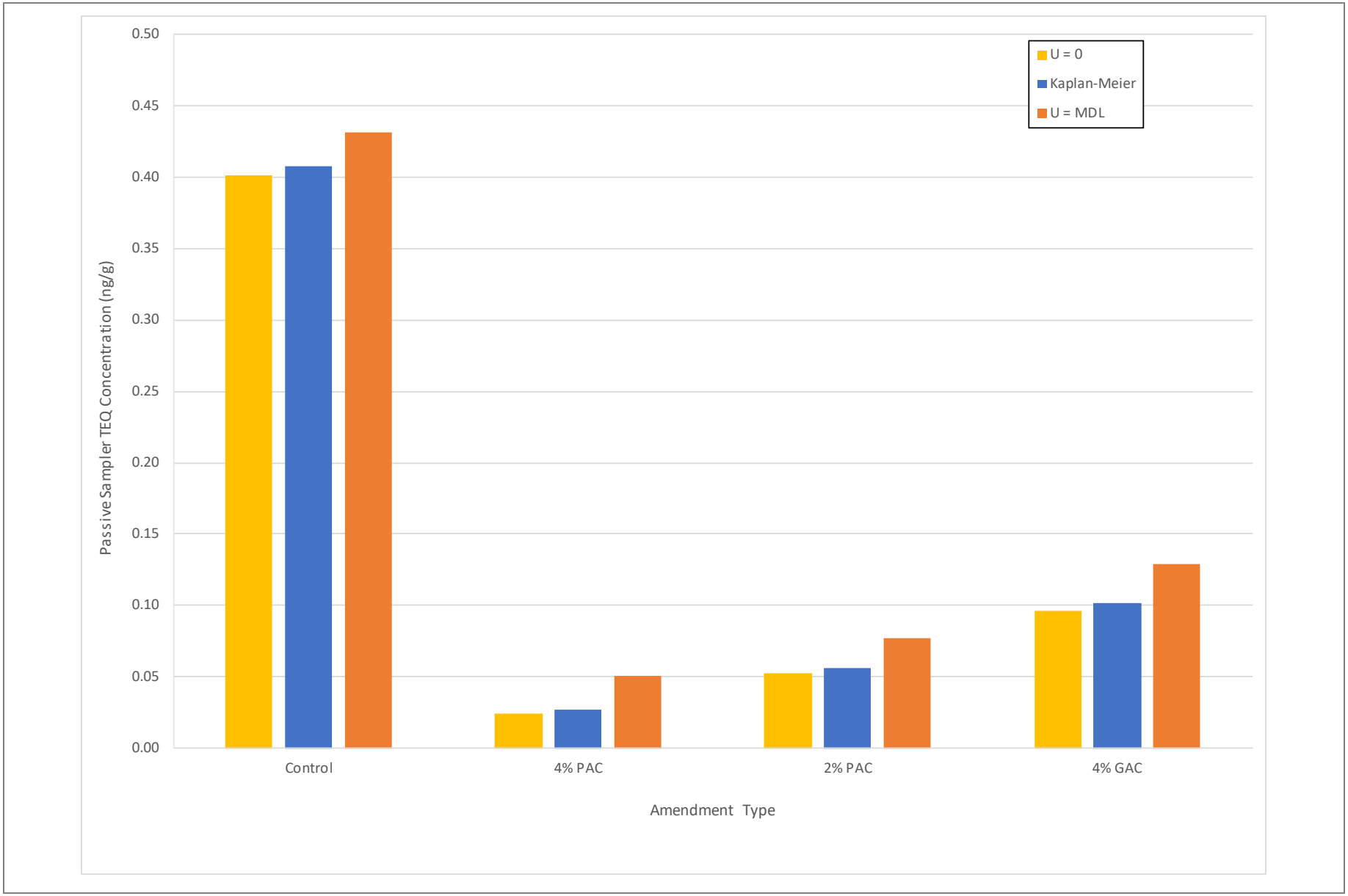
Figure 5

Set 2 Dioxin/Furan Congener TEQ Profiles in the Sediment Porewater – SR-2019-06

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design


0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Passive Sampler TEQ Concentration (ng/g)
U = 0
Kaplan-Meier
U = MDL
Control
4% PAC
2% PAC
4% GAC
Amendment Type

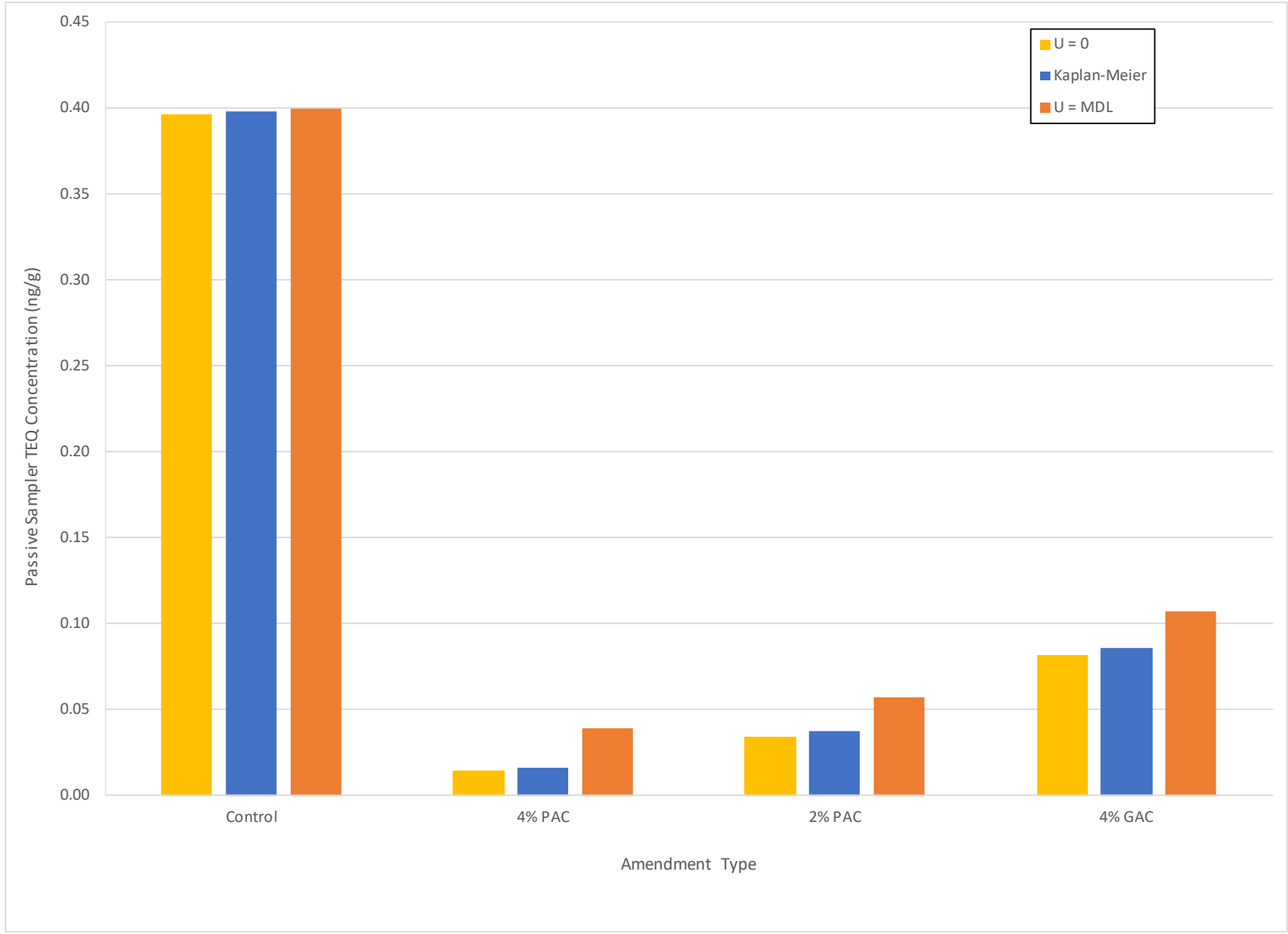


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Figure 7

Set 2 Dioxin/Furan TEQ Concentration Profile in Passive Samplers – SR-2019-04

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

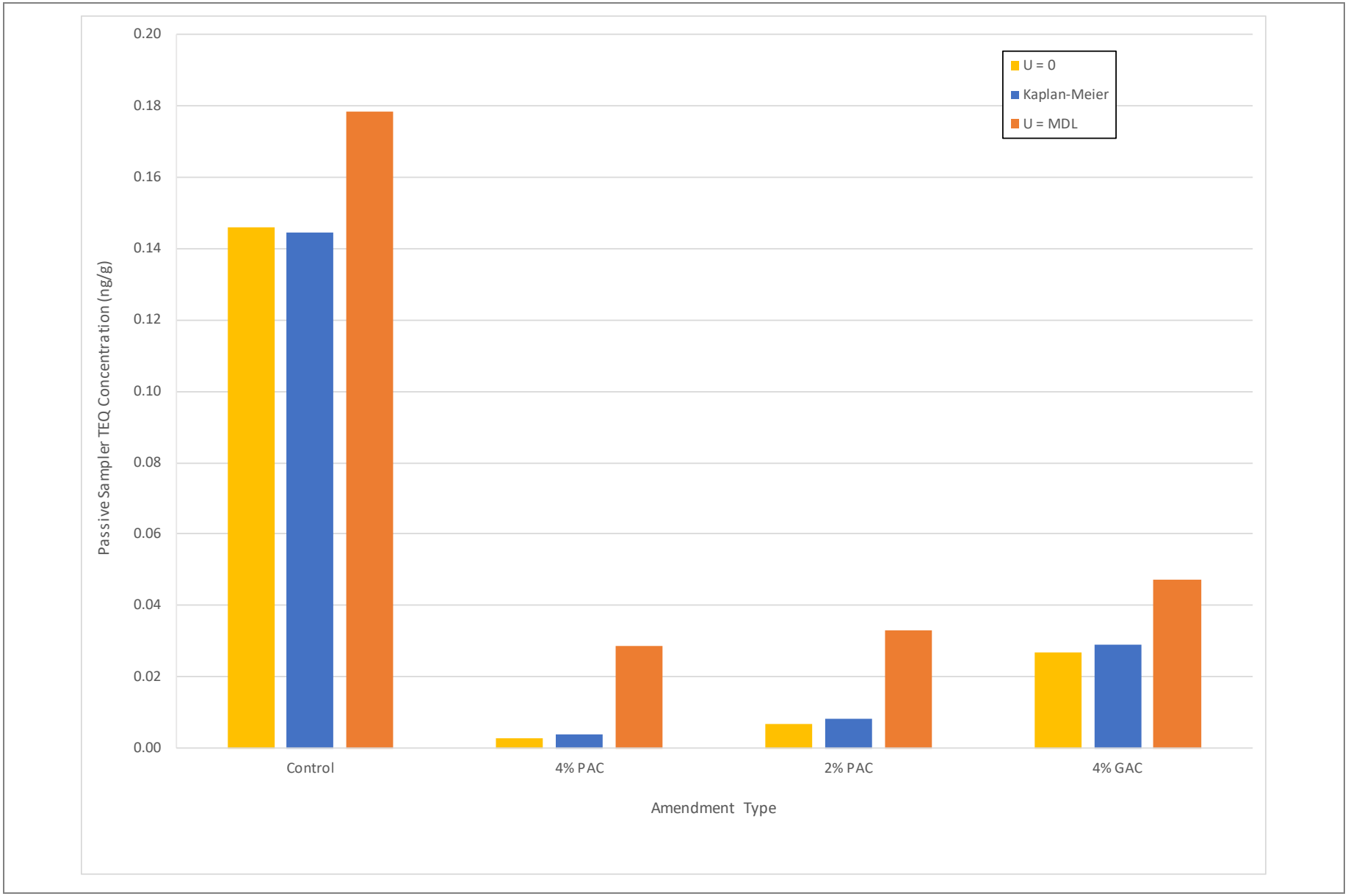

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Figure 8

Set 1 Dioxin/Furan TEQ Concentration Profile in Passive Samplers – SR-2019-06

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

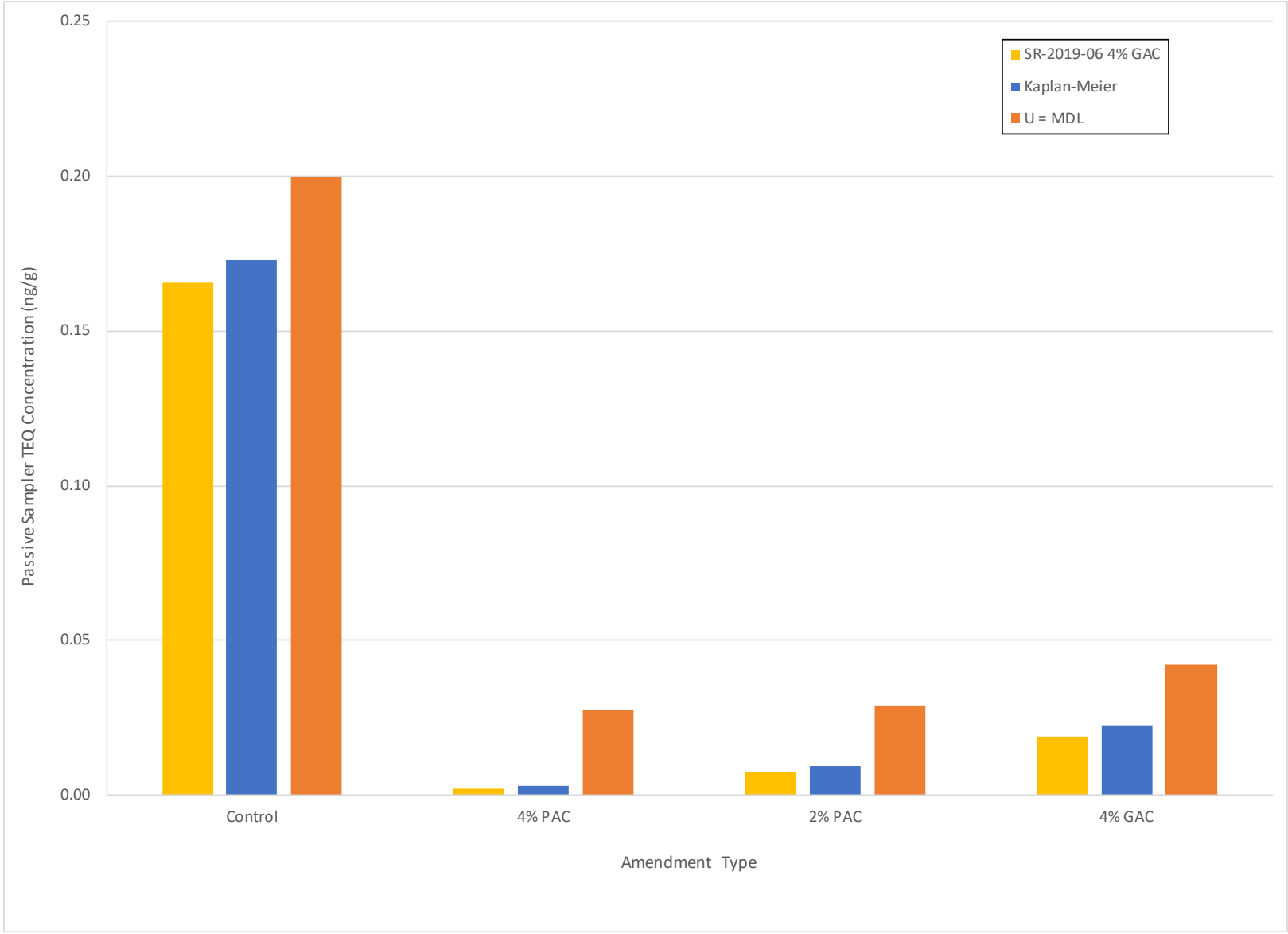

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Figure 9

Set 2 Dioxin/Furan TEQ Concentration Profile in Passive Samplers – SR-2019-06

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

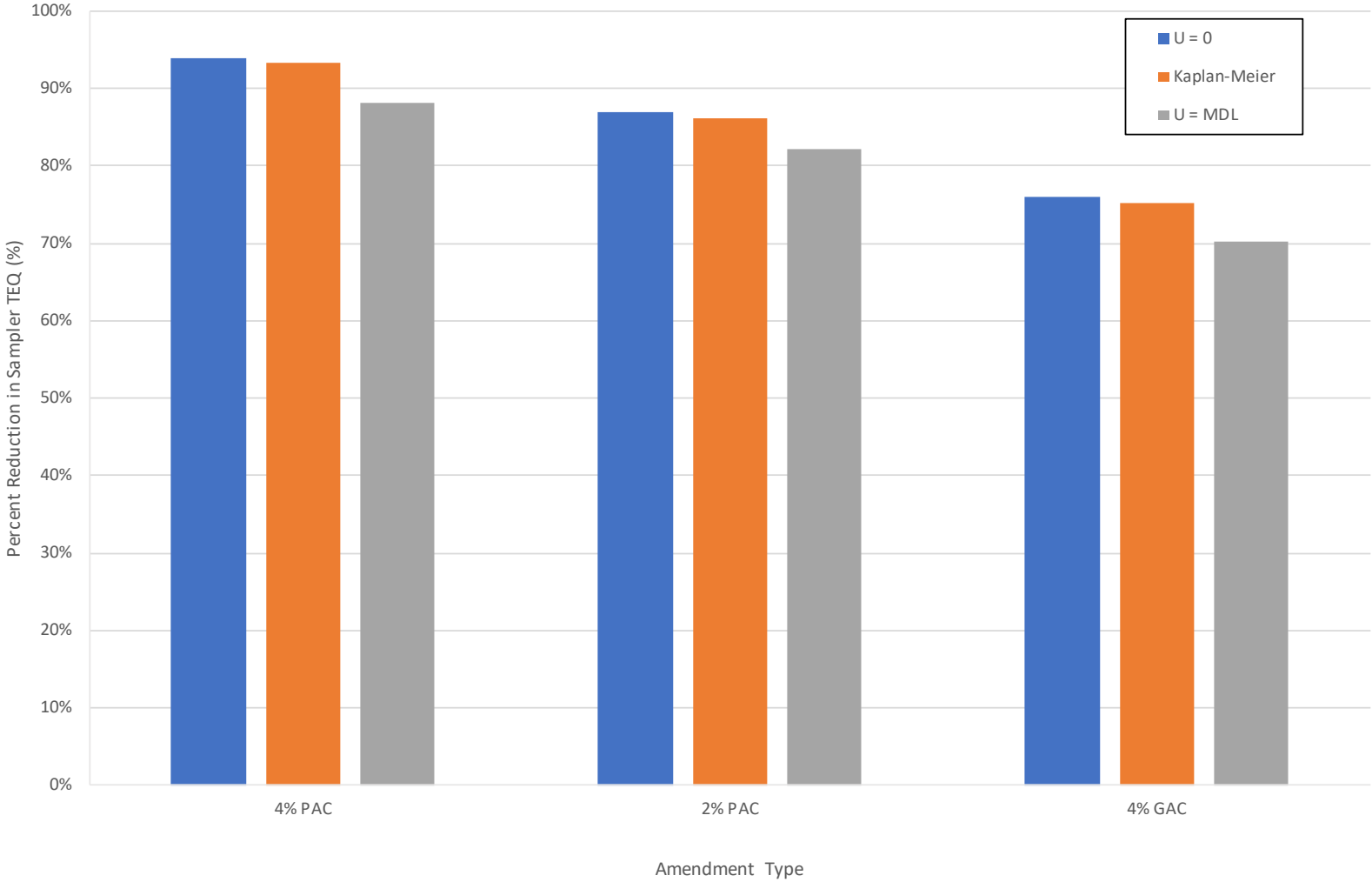

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Figure 10

Set 1 Percent Reduction in Passive Sampler Dioxin/Furan TEQ Concentration by Congener – SR-2019-04

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

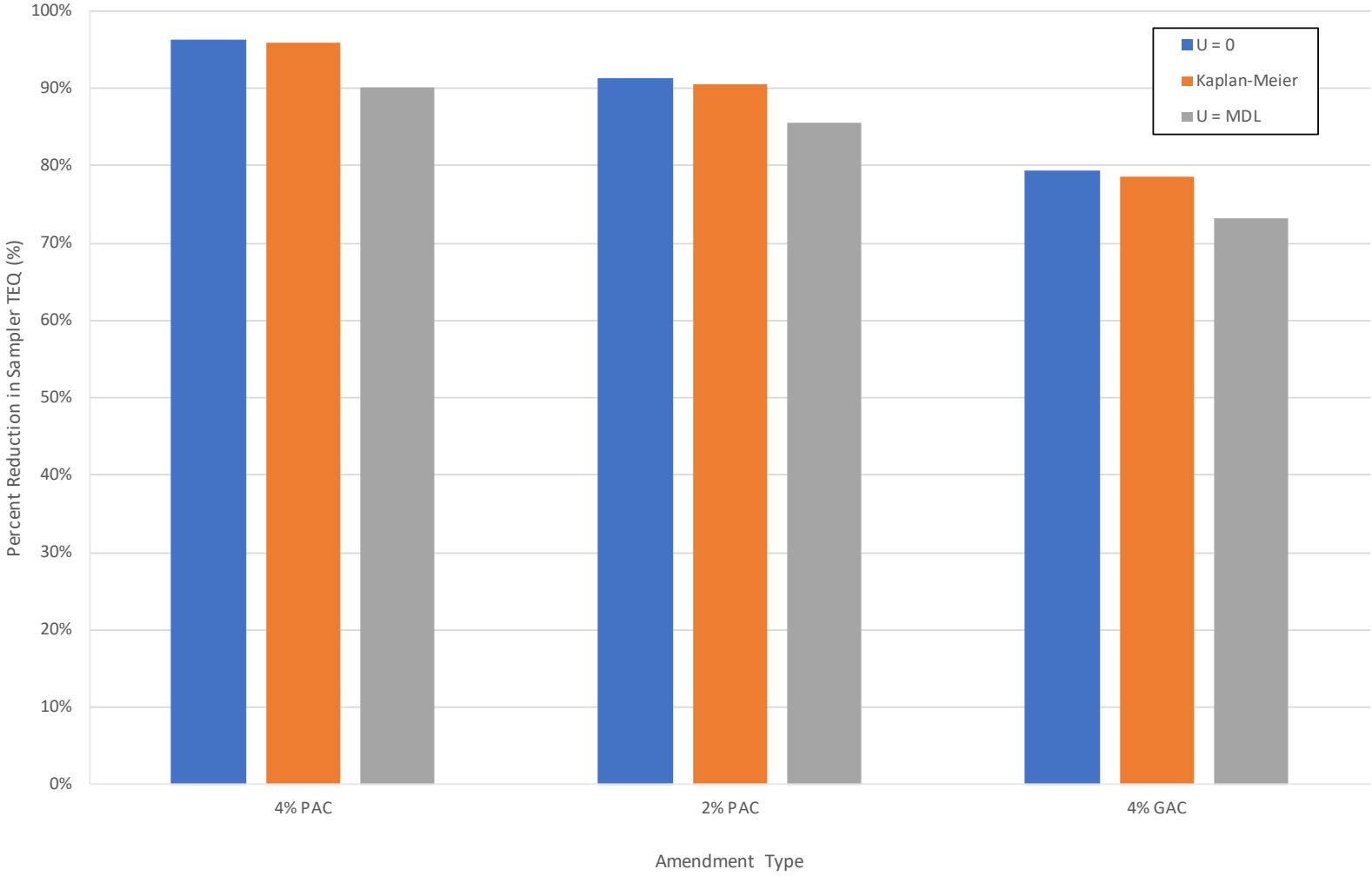

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Figure 11

Set 2 Percent Reduction in Passive Sampler Dioxin/Furan TEQ Concentration by Congener – SR-2019-04

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

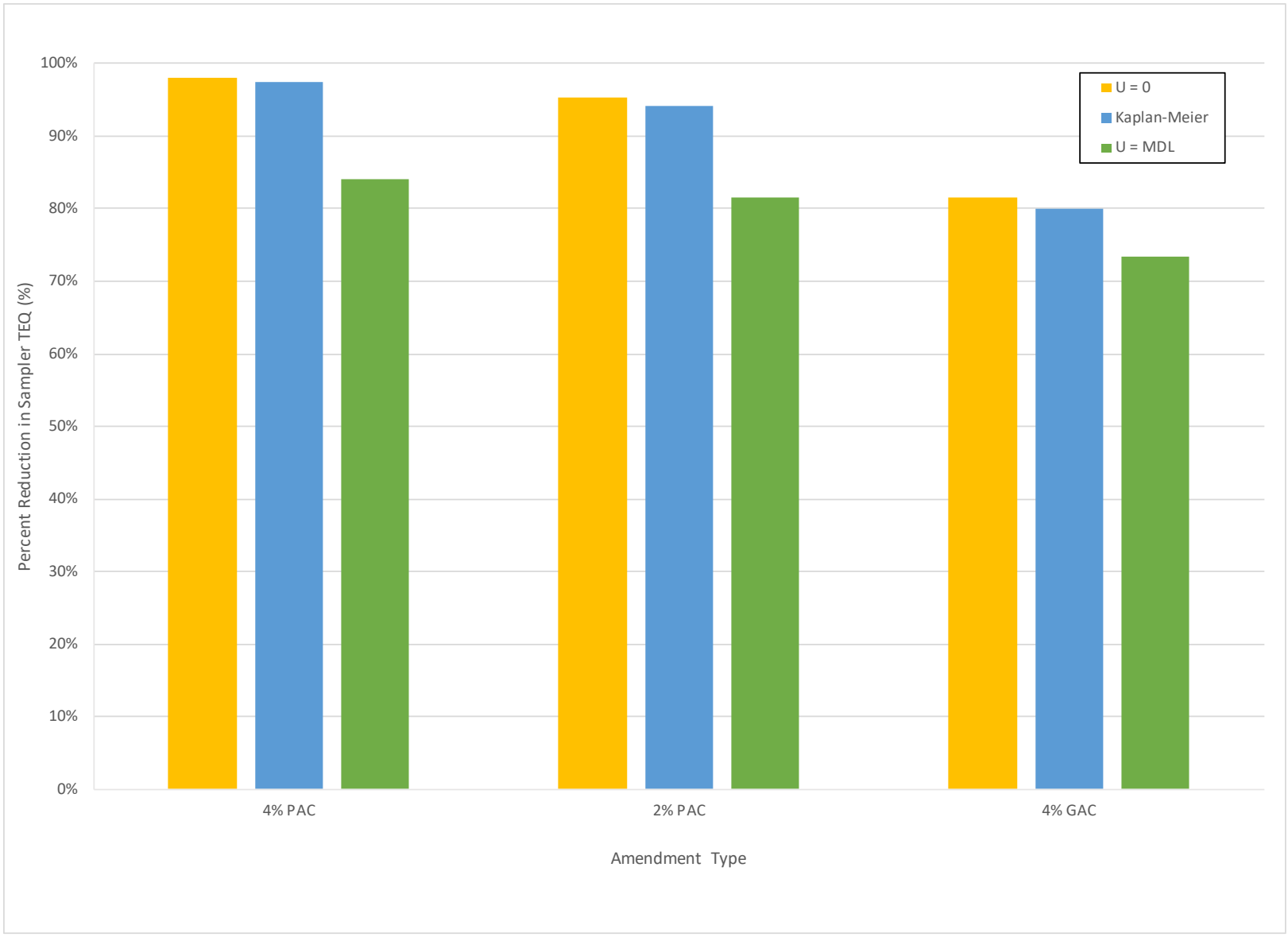


Figure 12

Set 1 Percent Reduction in Passive Sampler Dioxin/Furan TEQ Concentration by Congener – SR-2019-06

Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

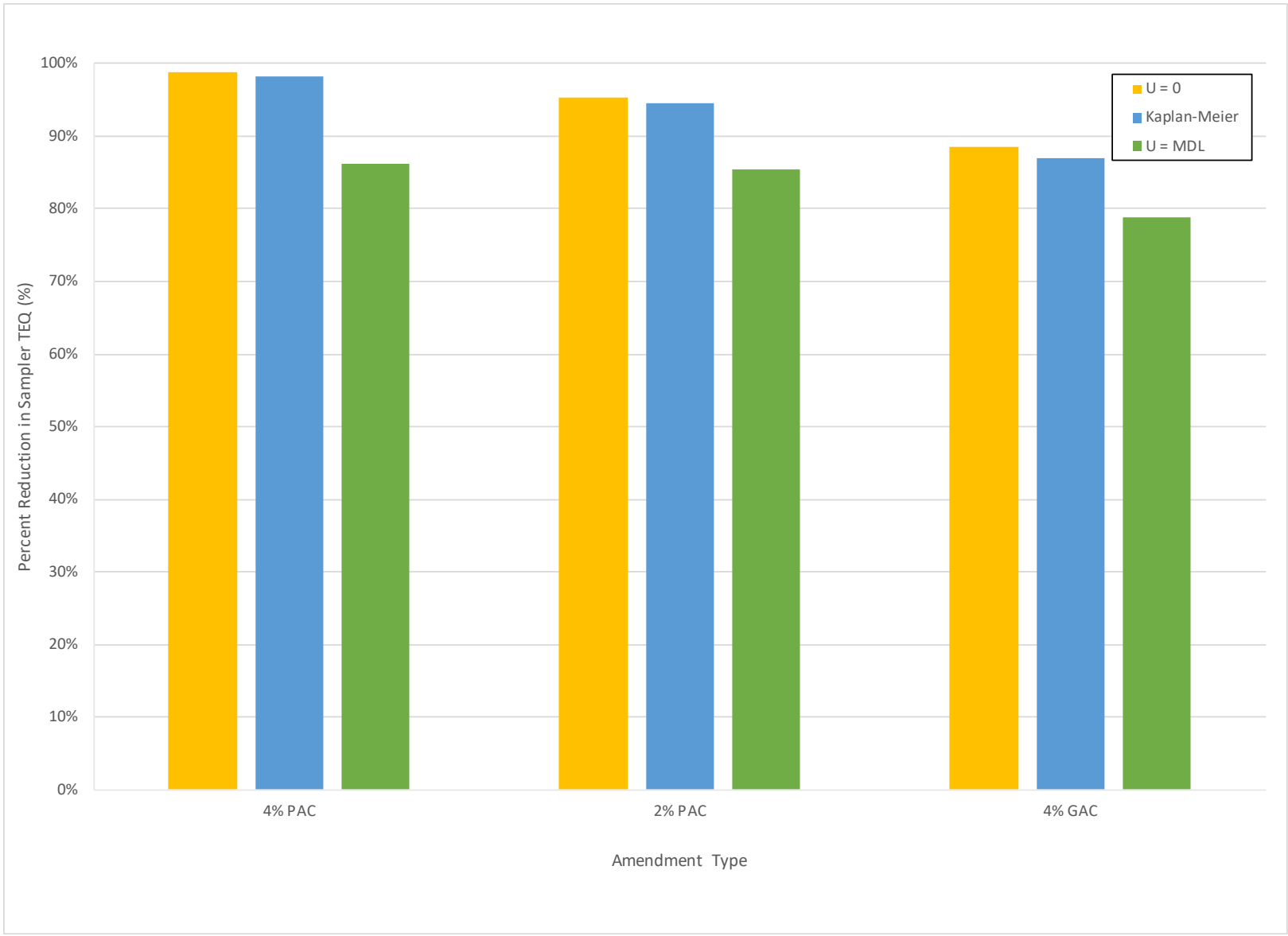
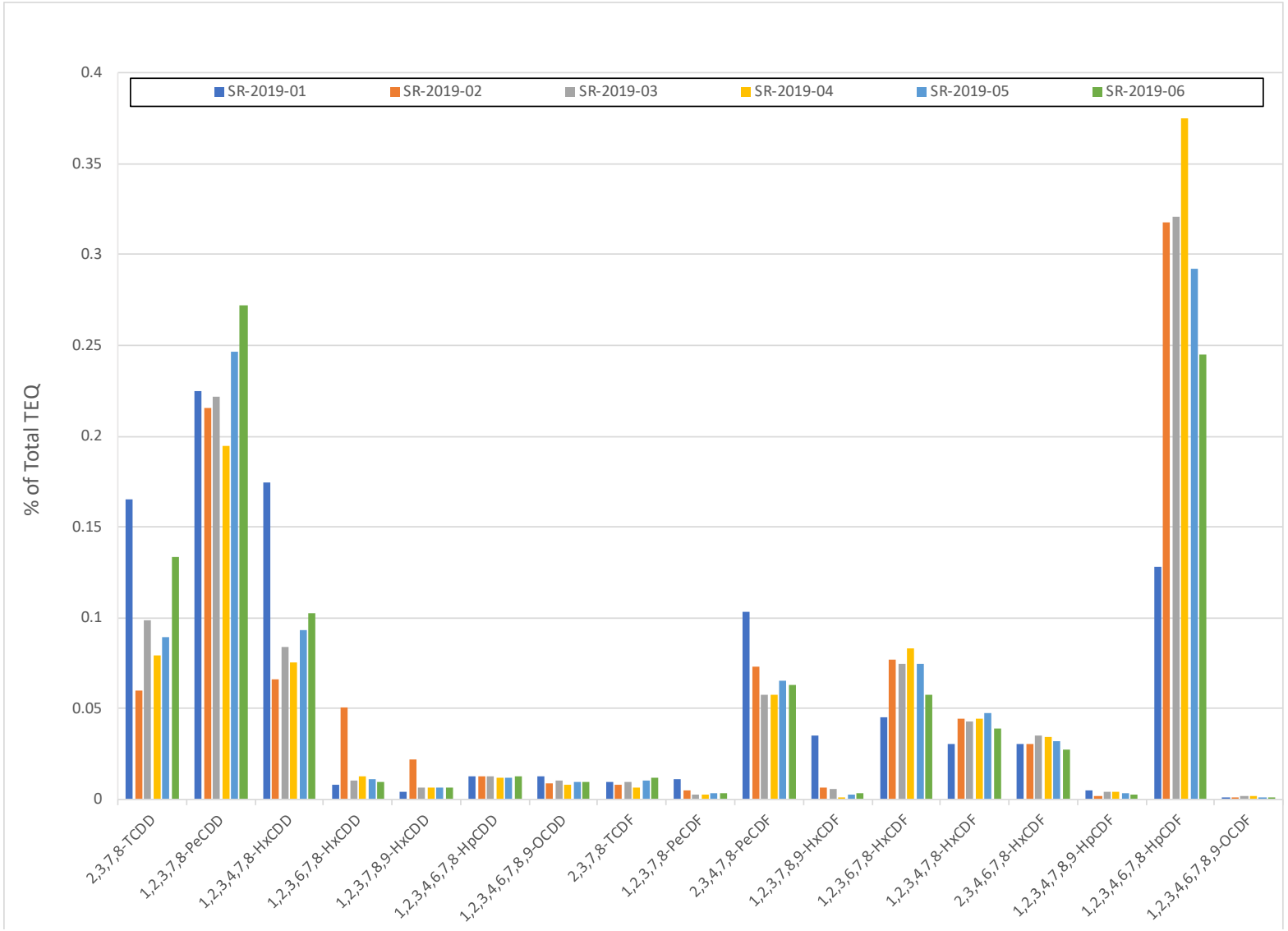


Figure 13

Set 2 Percent Reduction in Passive Sampler Dioxin/Furan TEQ Concentration by Congener – SR-2019-06

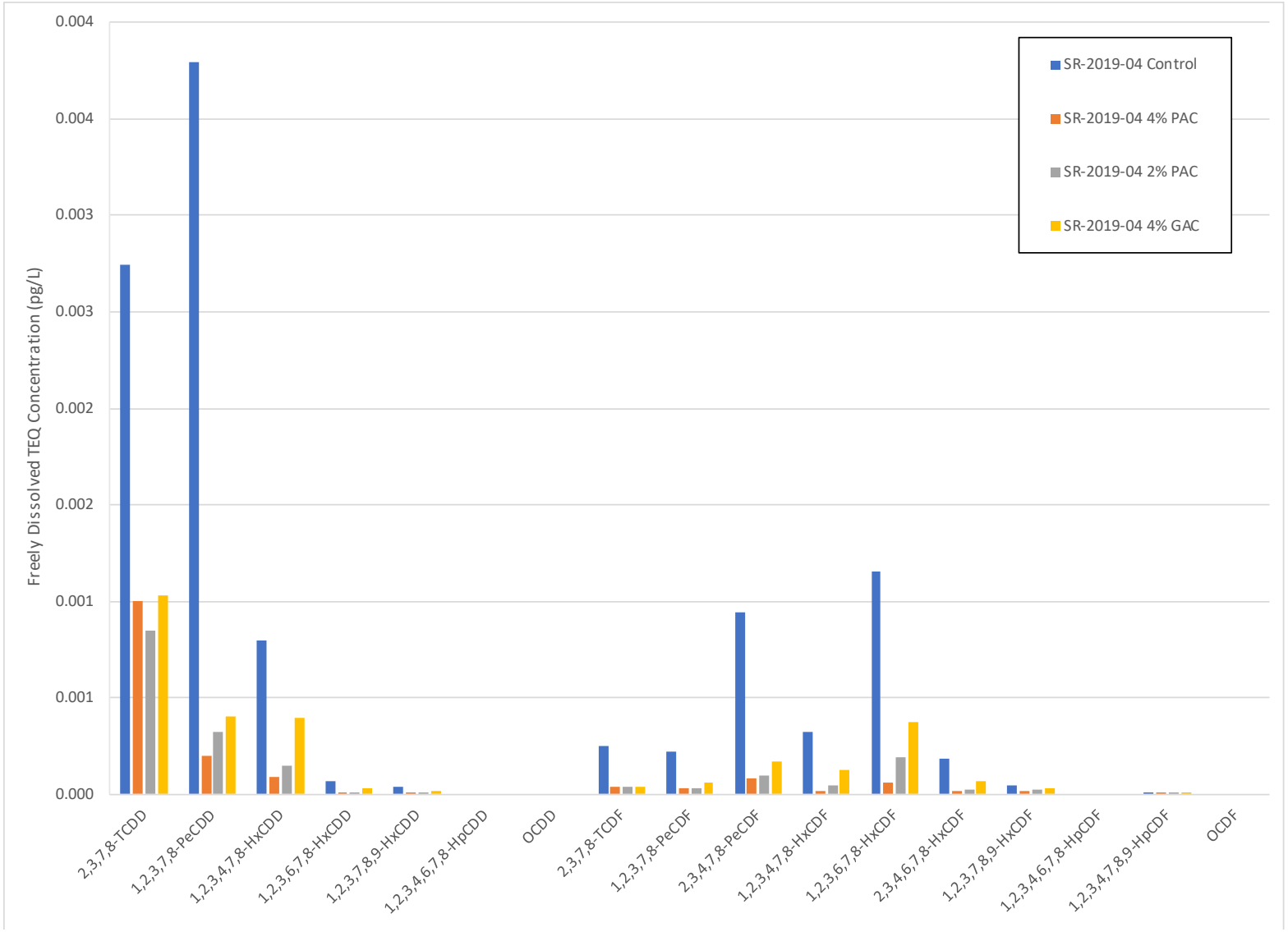
Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design

Appendix A
Congener Distributions



Note:

1. The data presented in these figures have non-detect congeners reported at the analytical laboratory detection limit.



Note:

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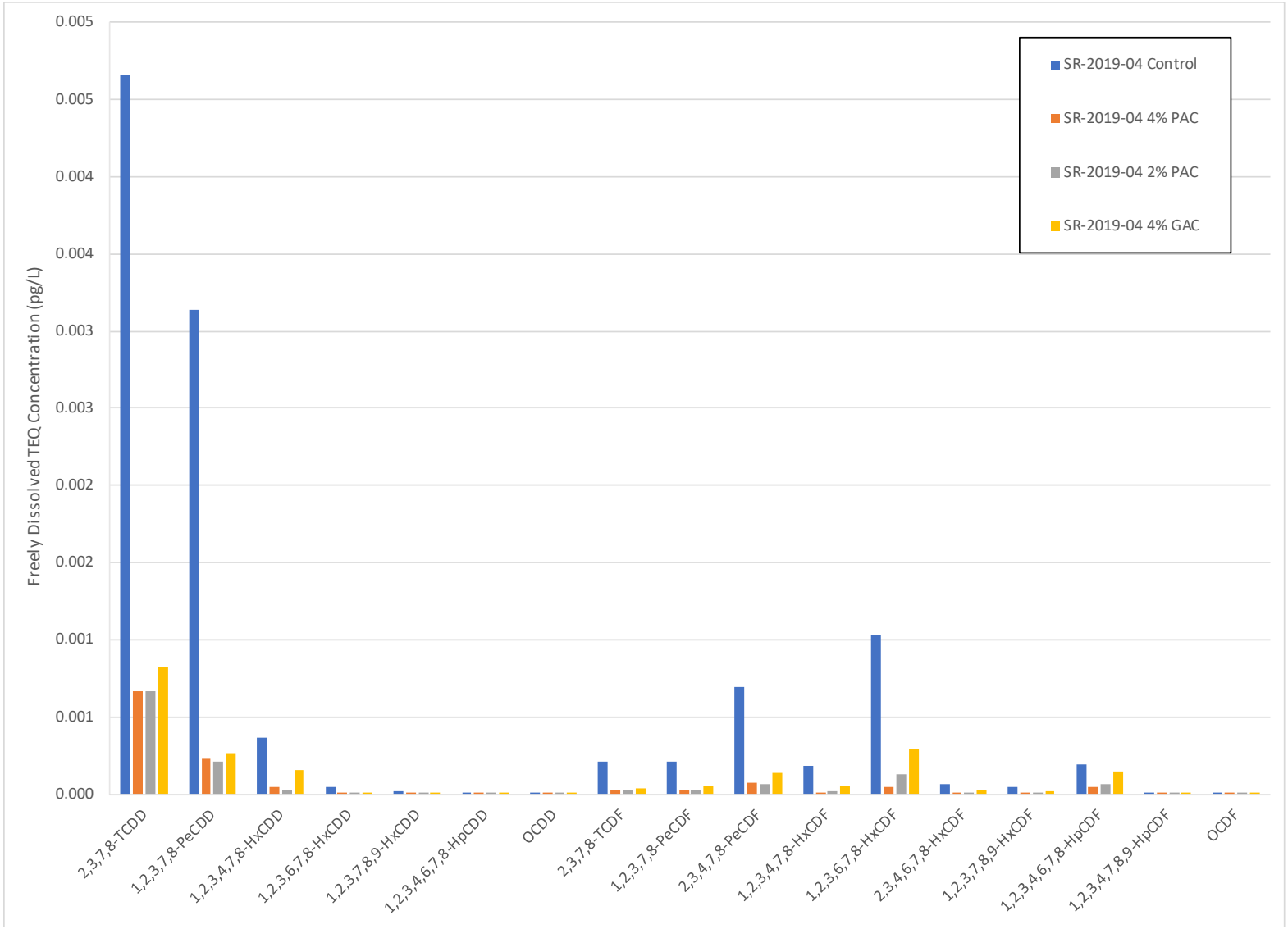

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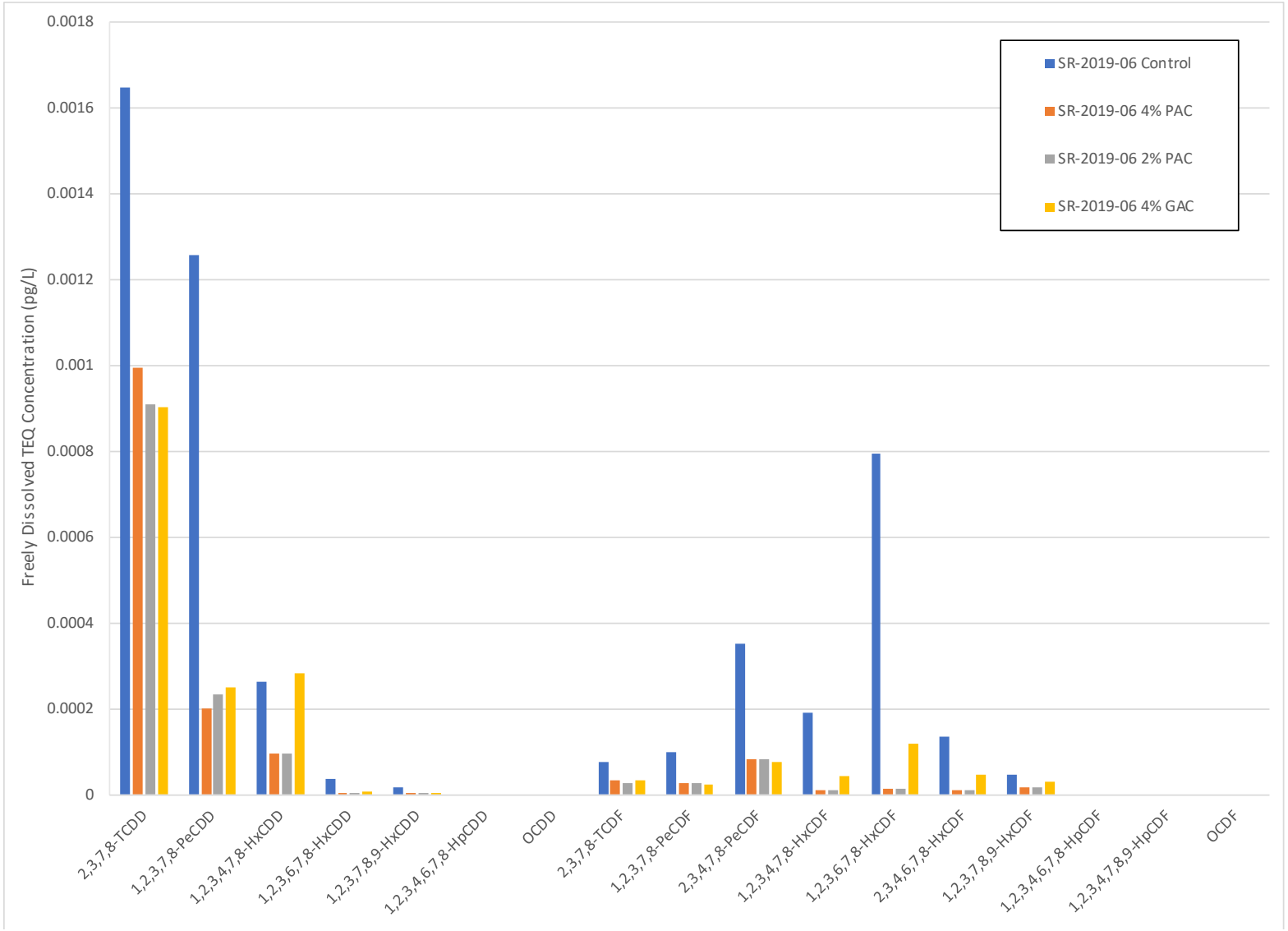

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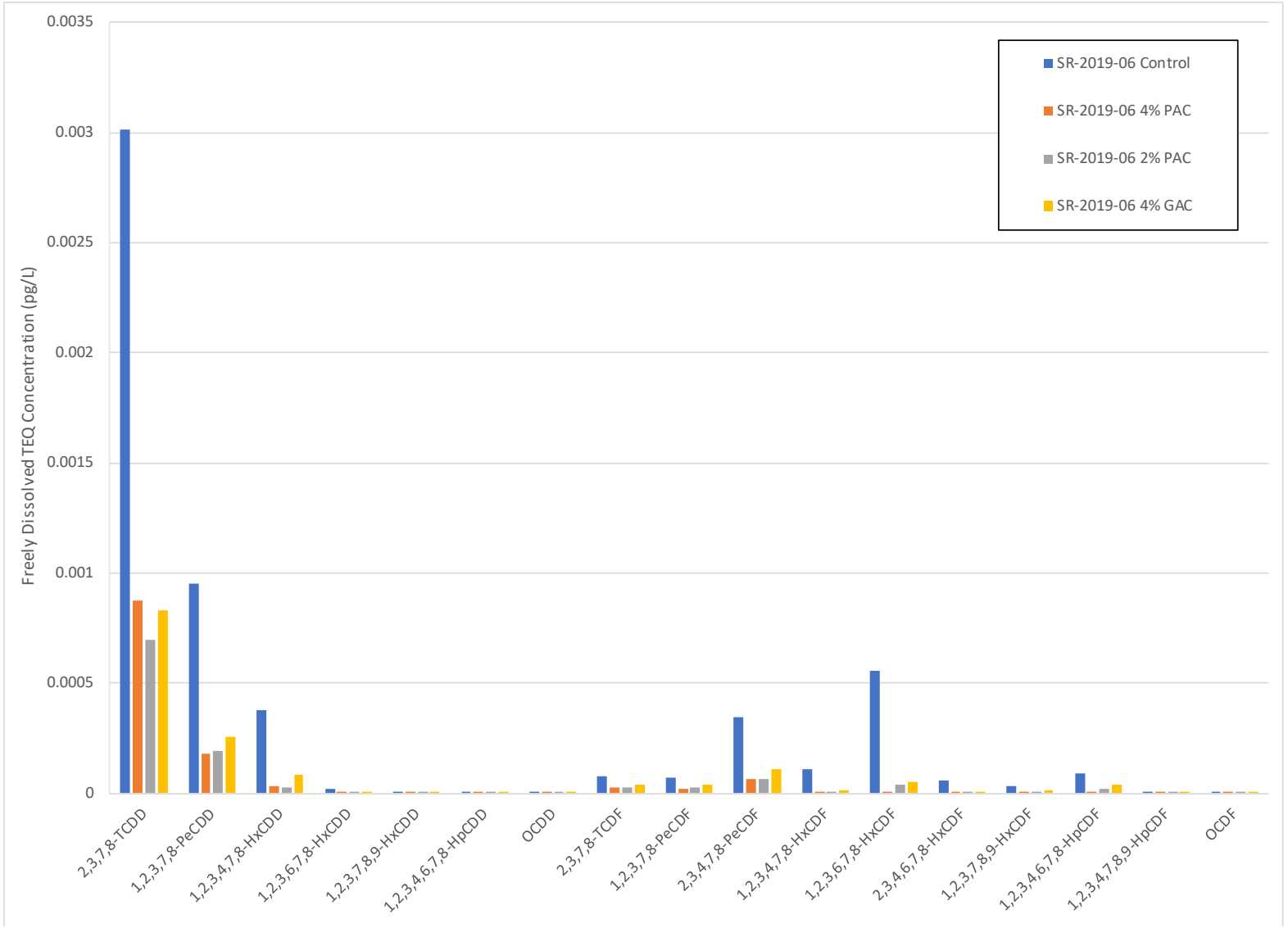

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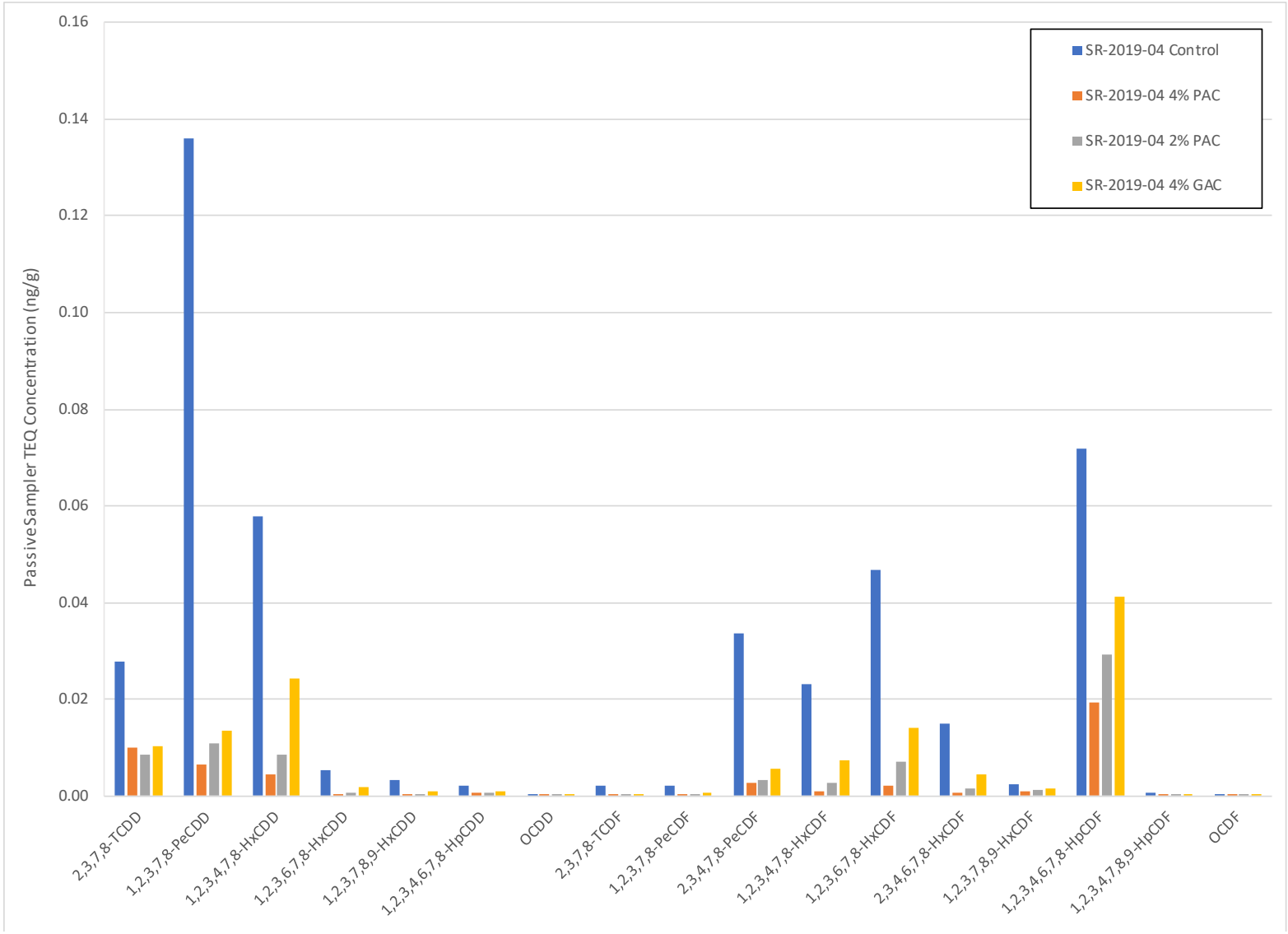

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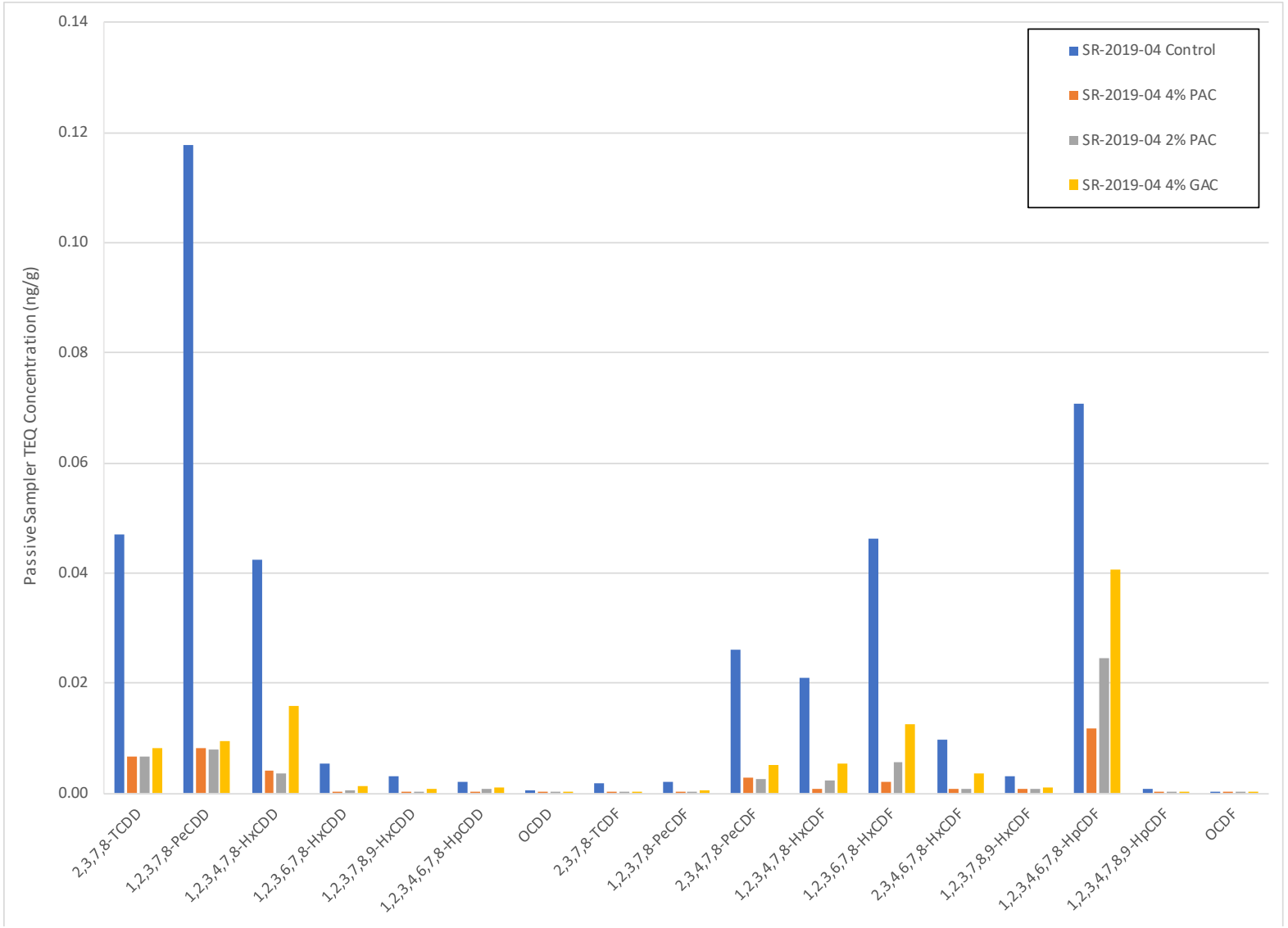

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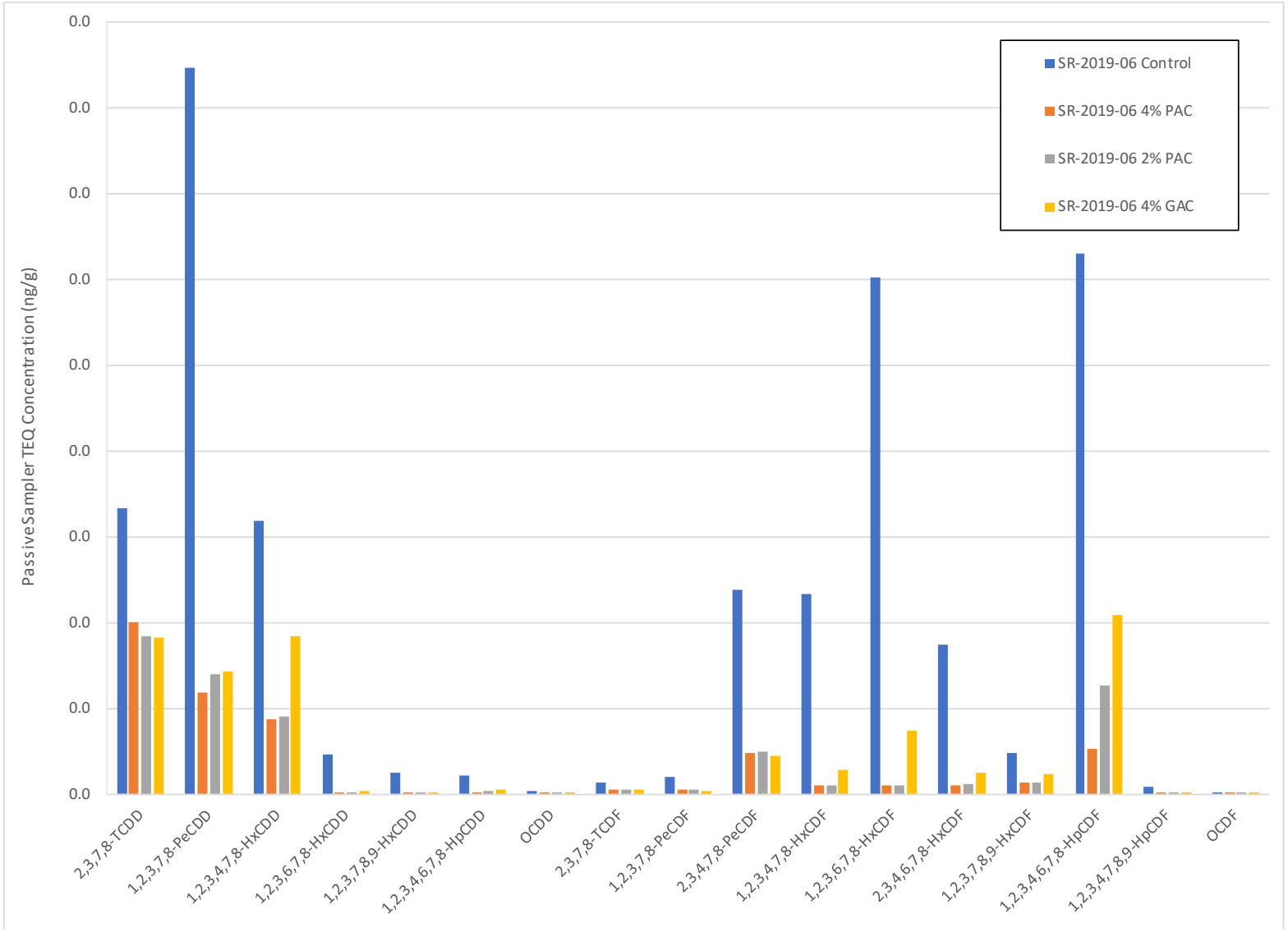

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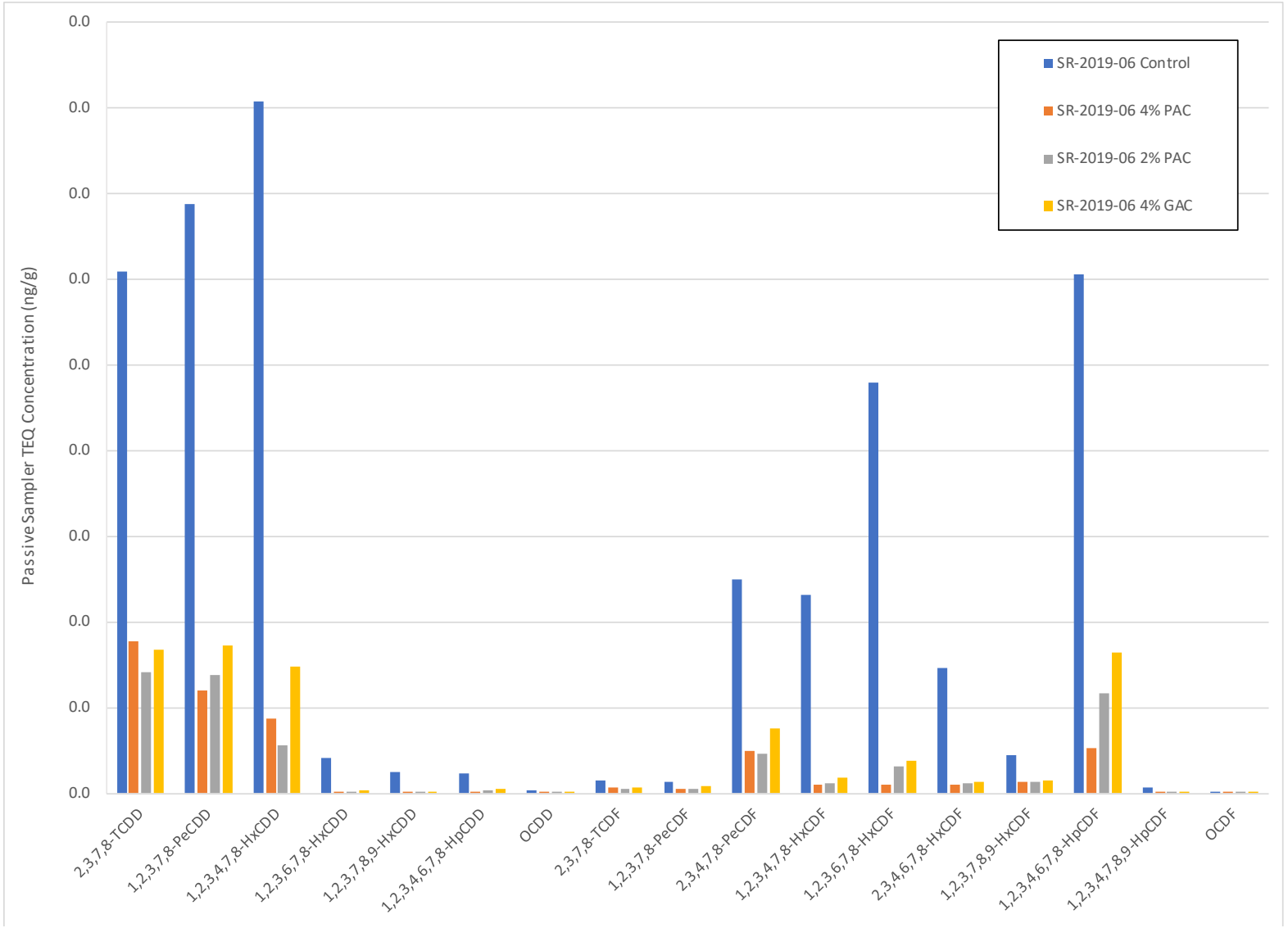

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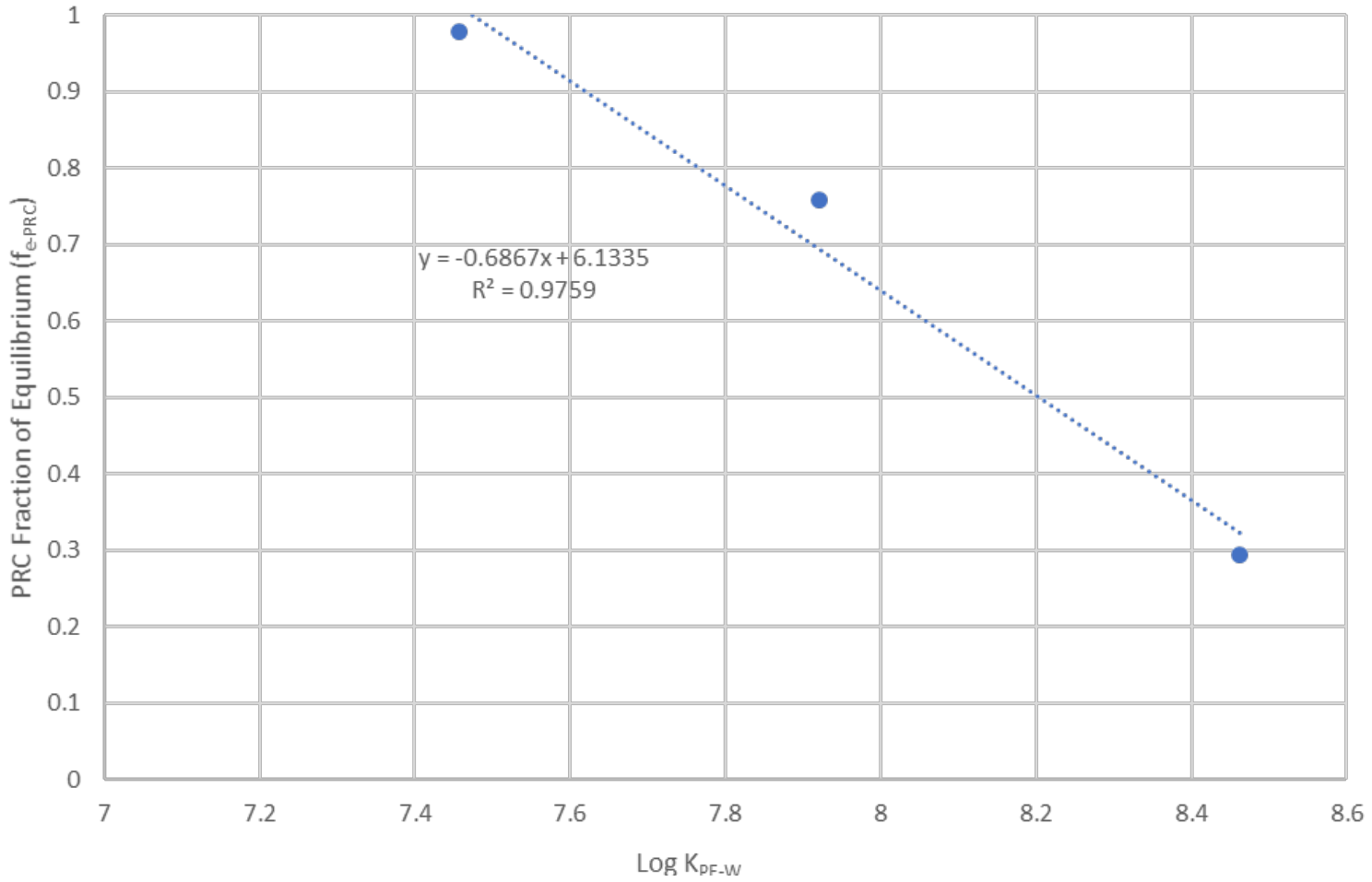
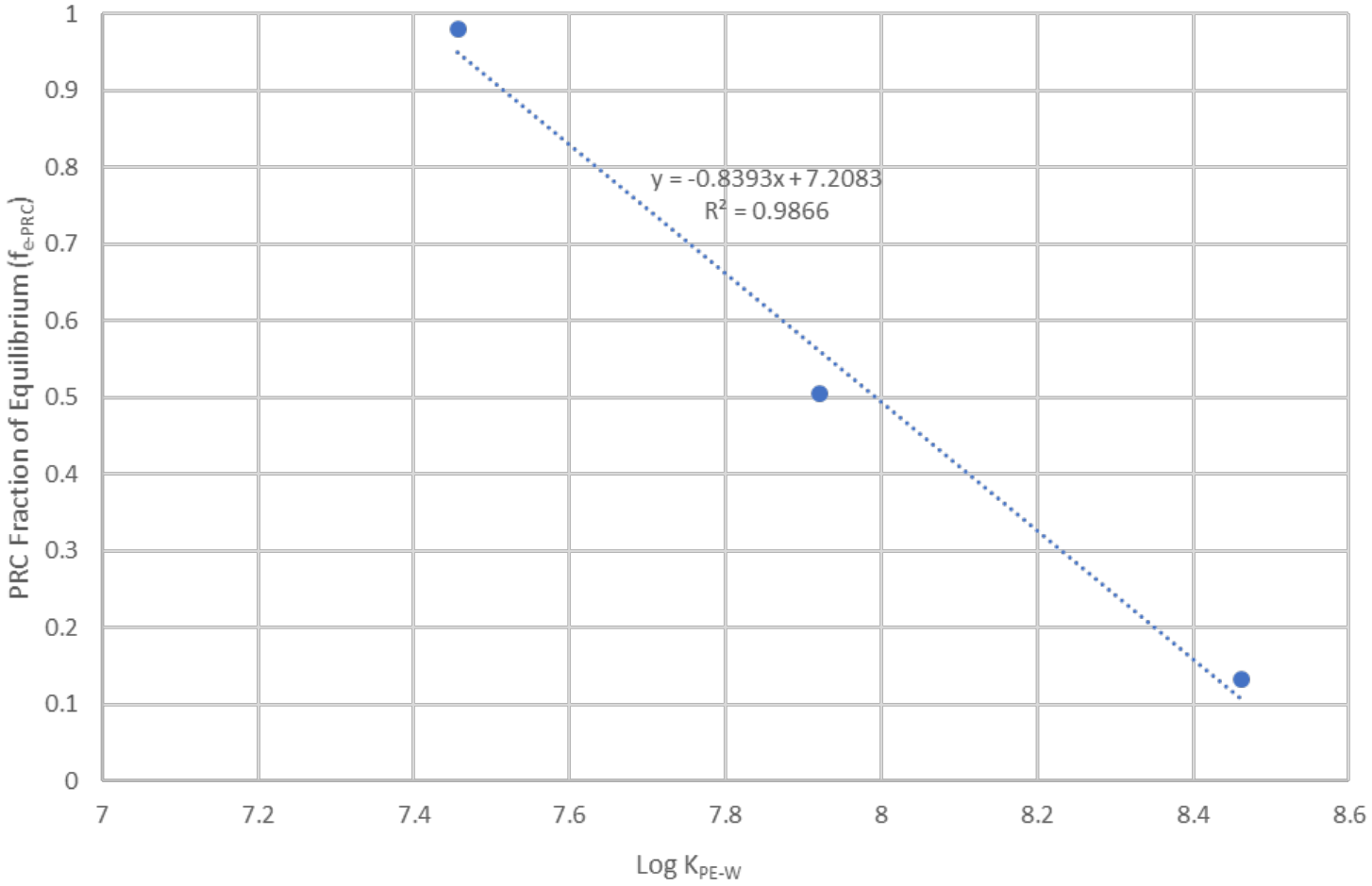
Appendix B
Fraction of Equilibrium Regressions



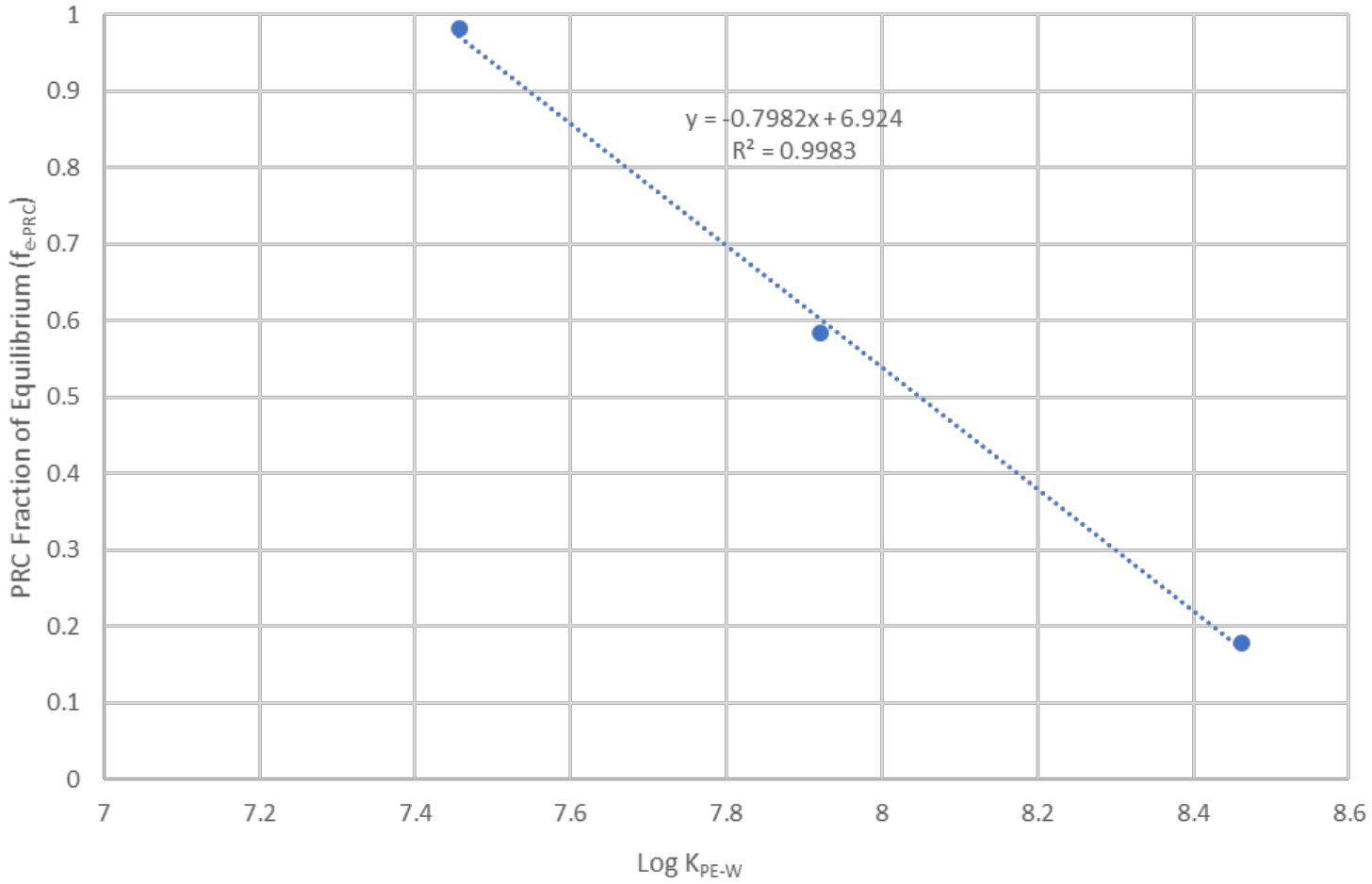
Notes:

1. Calculated $f_{e,PRC}$ and corresponding log $K_{PE\text{-}W}$ values are plotted for ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD. Since ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from LDPE samplers after 31 days of deployment, it was removed from the plot to improve the fit of the linear regression.
2. The linear regression of this plot follows the equation $f_{e,\,PRC} = a \times \log K_{PE\text{-}W} + b$, where "a" is the slope and "b" is the y-intercept.
3. Isotropic exchange kinetics are assumed (i.e., the depletion rate of a PRC on an LDPE sampler reflects the uptake rate of a target analyte). Therefore, the linear regression line represents the possible fraction of equilibrium of target dioxins/furans (f_e). The slope (i.e., "a") and y-intercept (i.e., "b") from this linear regression are utilized to calculate the f_e of each target compound.



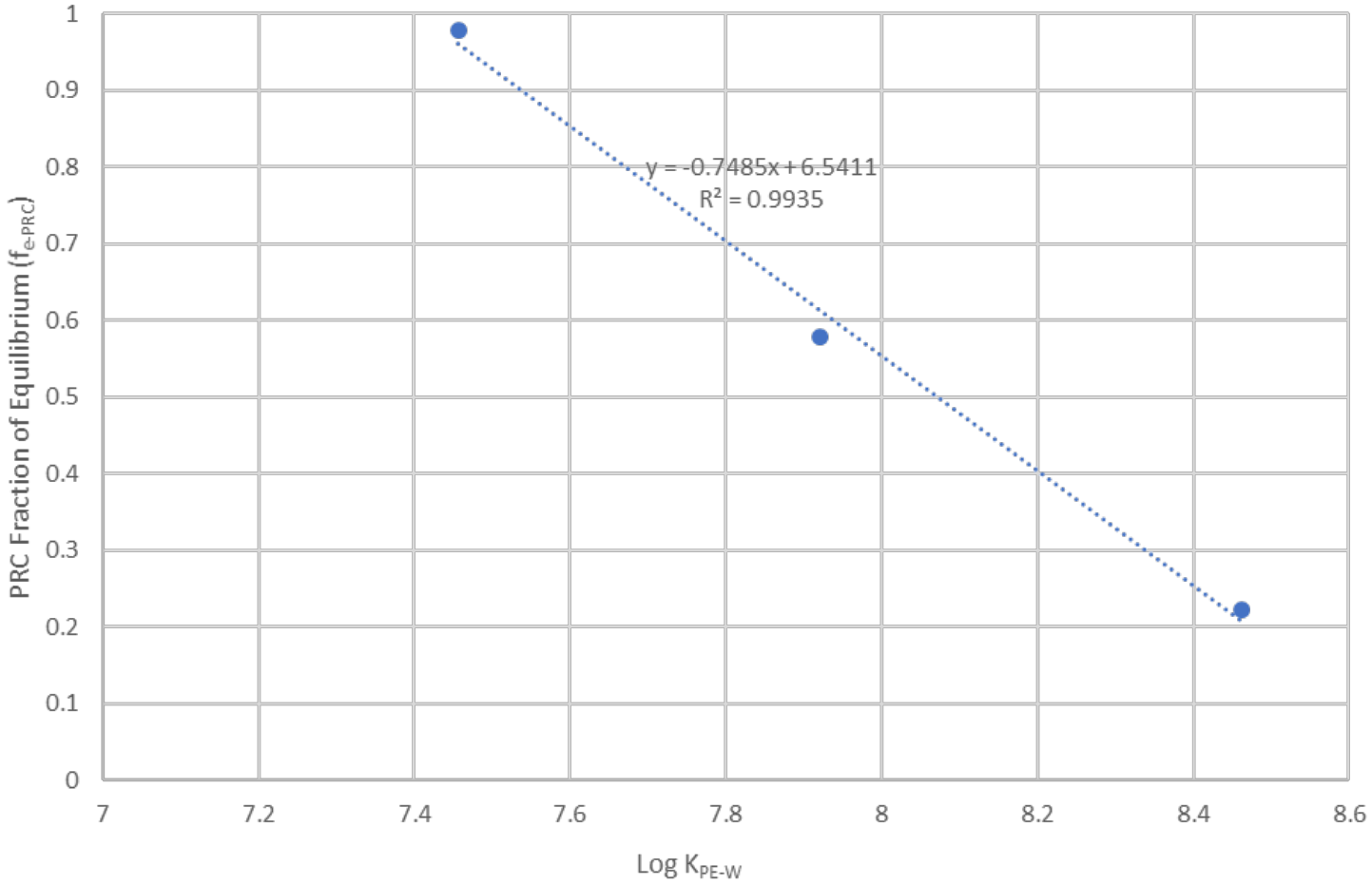
Notes:

1. Calculated $f_{e,PRC}$ and corresponding log K_{PE-W} values are plotted for ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD. Since ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from LDPE samplers after 31 days of deployment, it was removed from the plot to improve the fit of the linear regression.
2. The linear regression of this plot follows the equation $f_{e,PRC} = a \times \log K_{PE-W} + b$, where "a" is the slope and "b" is the y-intercept.
3. Isotropic exchange kinetics are assumed (i.e., the depletion rate of a PRC on an LDPE sampler reflects the uptake rate of a target analyte). Therefore, the linear regression line represents the possible fraction of equilibrium of target dioxins/furans (f_e). The slope (i.e., "a") and y-intercept (i.e., "b") from this linear regression are utilized to calculate the f_e of each target compound.



Notes:

1. Calculated $f_{e,PRC}$ and corresponding log K_{PE-W} values are plotted for ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD. Since ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from LDPE samplers after 31 days of deployment, it was removed from the plot to improve the fit of the linear regression.
2. The linear regression of this plot follows the equation $f_{e, PRC}$=a x log K_{PE-W} + b, where "a" is the slope and "b" is the y-intercept.
3. Isotropic exchange kinetics are assumed (i.e., the depletion rate of a PRC on an LDPE sampler reflects the uptake rate of a target analyte). Therefore, the linear regression line represents the possible fraction of equilibrium of target dioxins/furans (f_e). The slope (i.e., "a") and y-intercept (i.e., "b") from this linear regression are utilized to calculate the f_e of each target compound.



Notes:

1. Calculated $f_{e,PRC}$ and corresponding log K_{PE-W} values are plotted for ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD. Since ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from LDPE samplers after 31 days of deployment, it was removed from the plot to improve the fit of the linear regression.
2. The linear regression of this plot follows the equation $f_{e, PRC} = a \times \log K_{PE-W} + b$, where "a" is the slope and "b" is the y-intercept.
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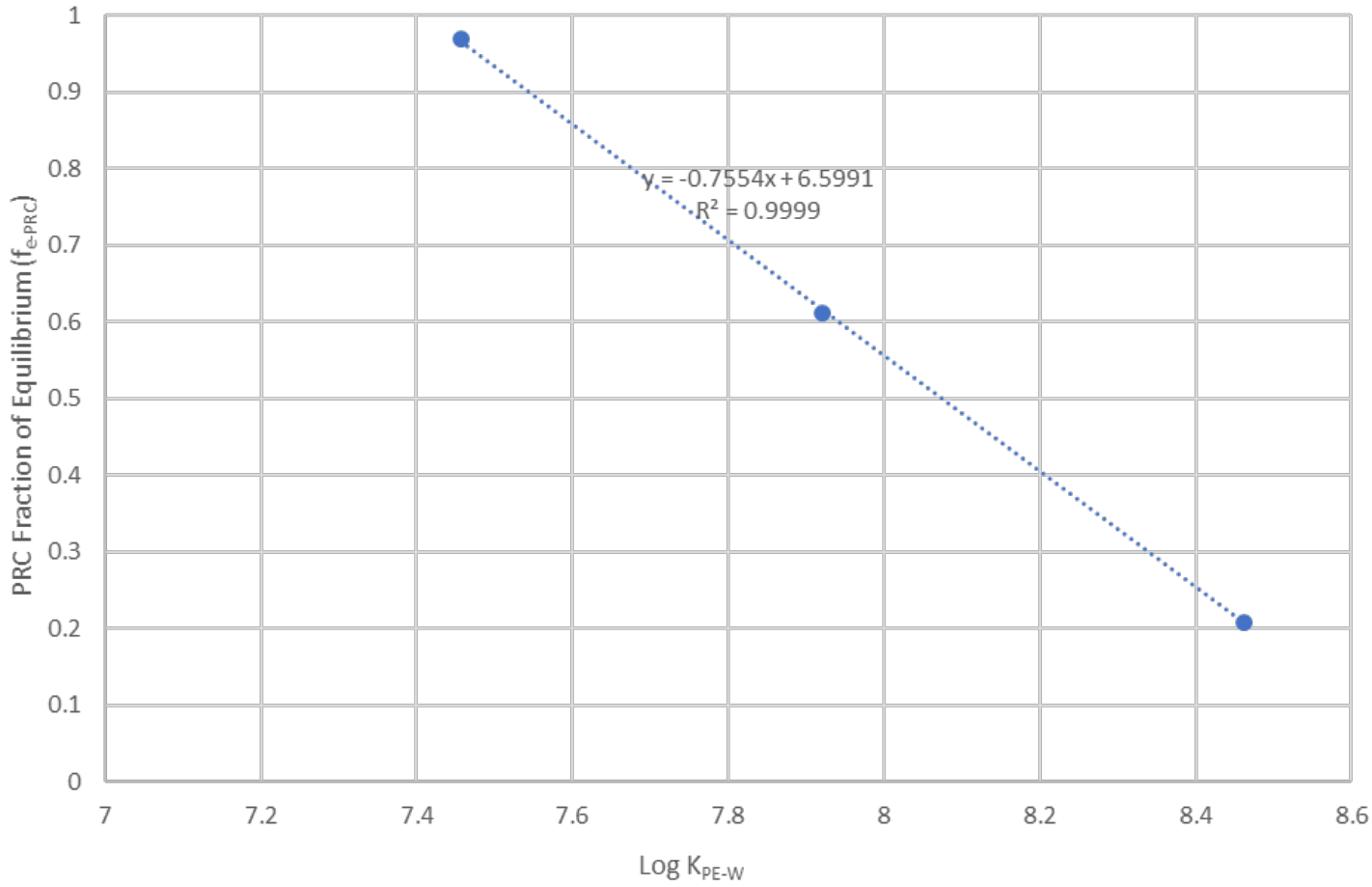

Notes:

1. Calculated $f_{e,PRC}$ and corresponding log K_{PE-W} values are plotted for ^{13}C-1,2,4,7,8-PeCDD, ^{13}C-1,2,3,4,6,8-HxCDD, and ^{13}C-1,2,3,4,6,7,9-HpCDD. Since ^{13}C-1,2,7,8-TCDD achieved approximately 100% depletion from LDPE samplers after 31 days of deployment, it was removed from the plot to improve the fit of the linear regression.
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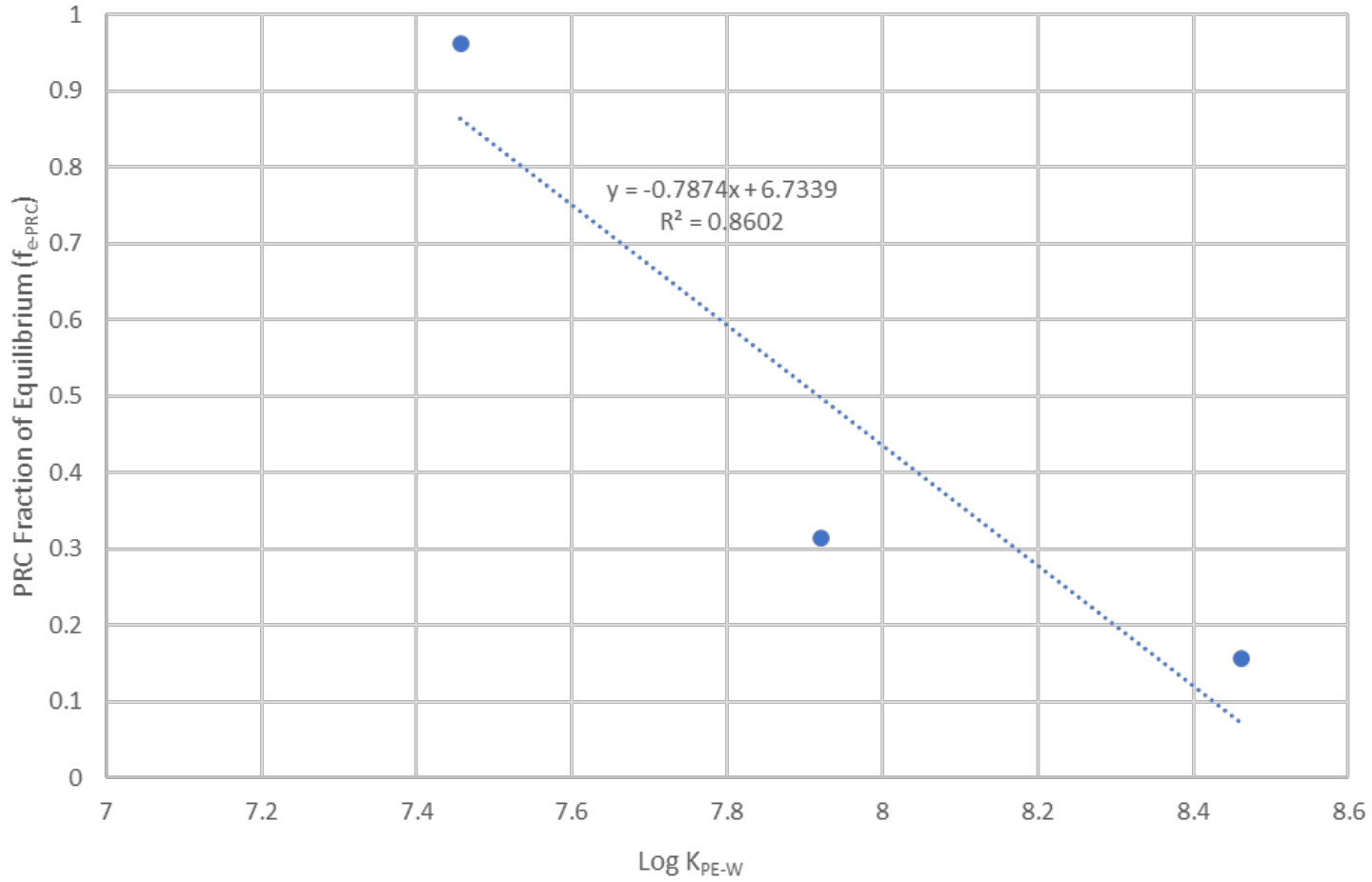

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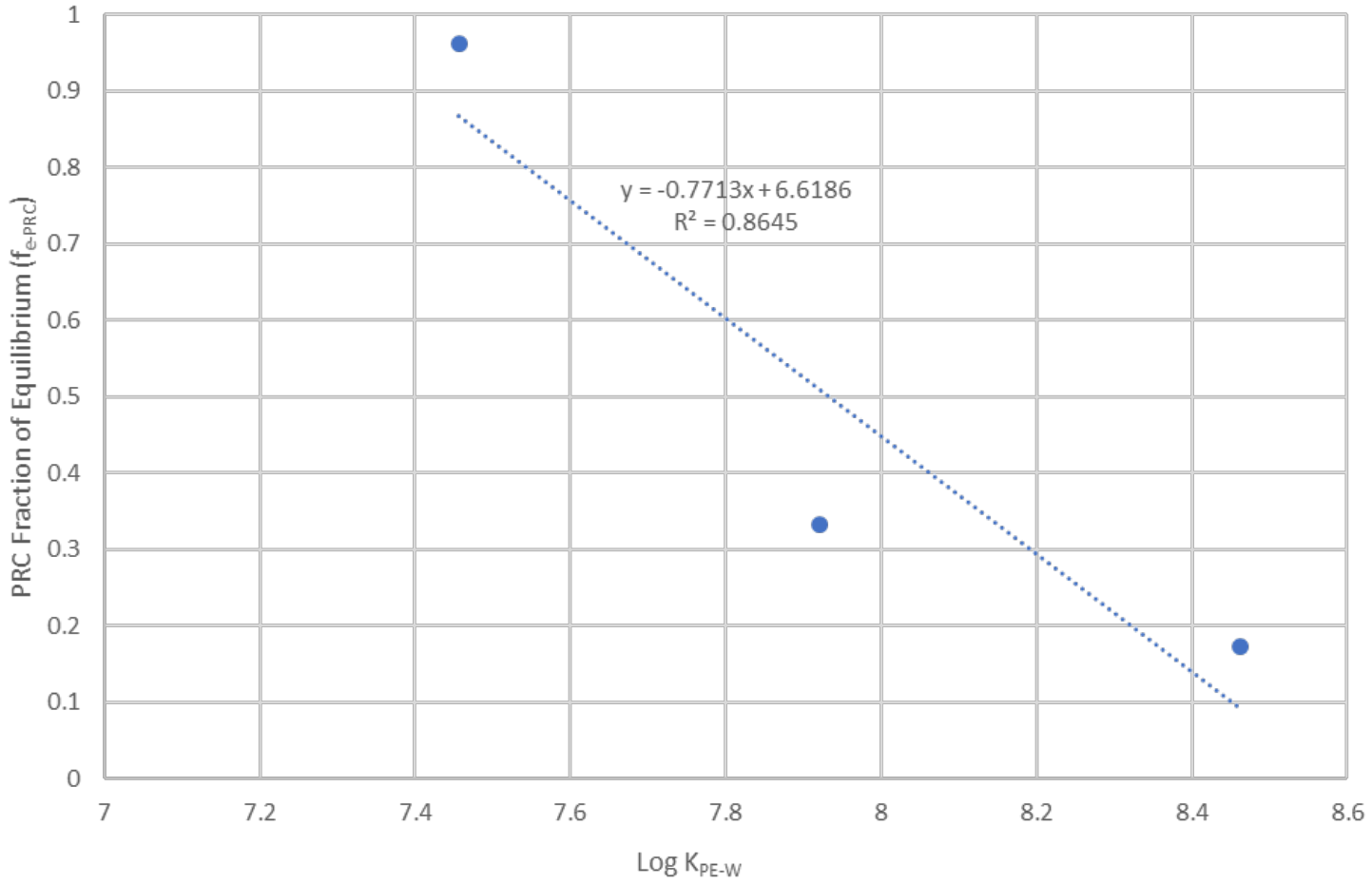

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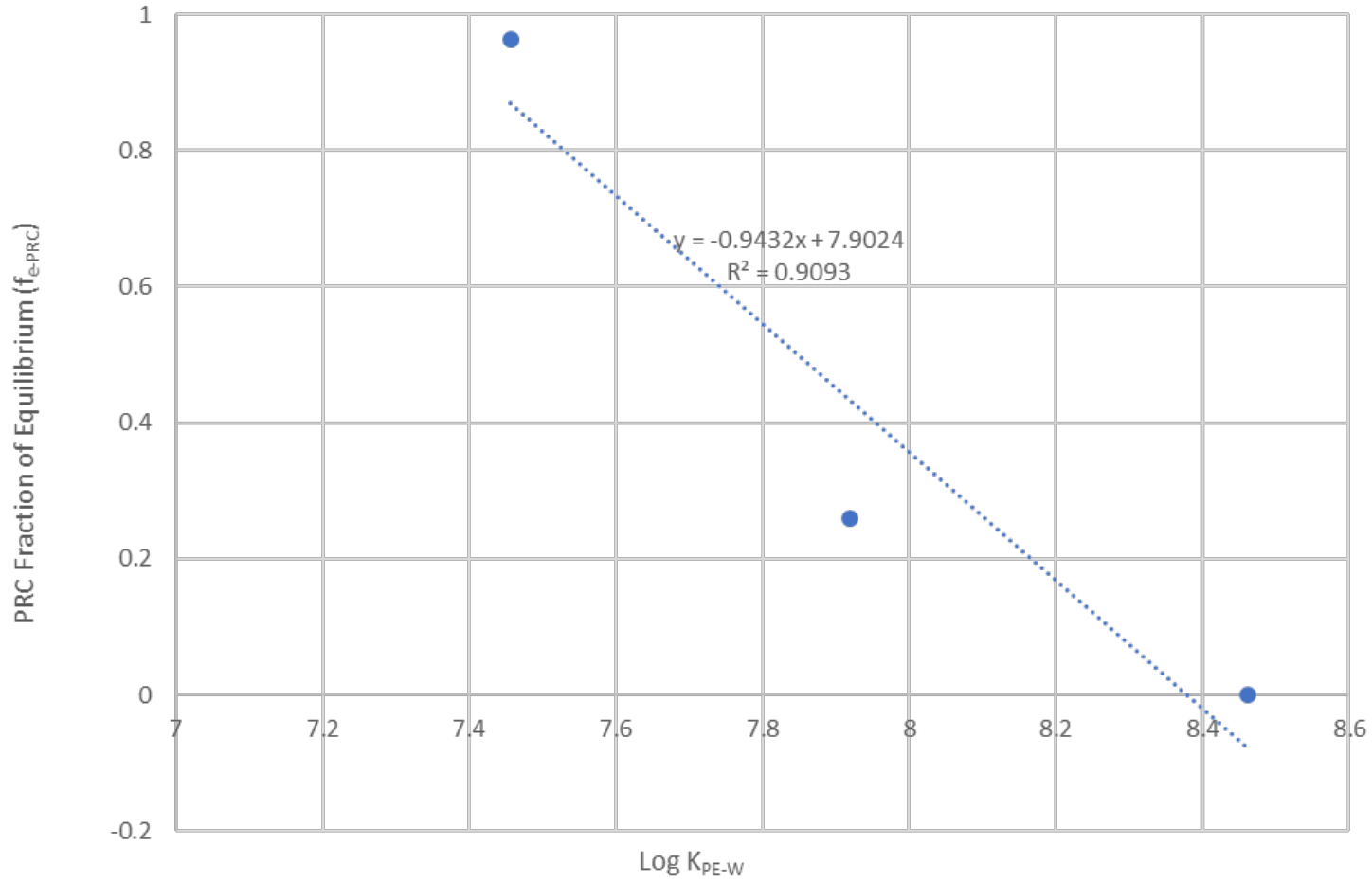

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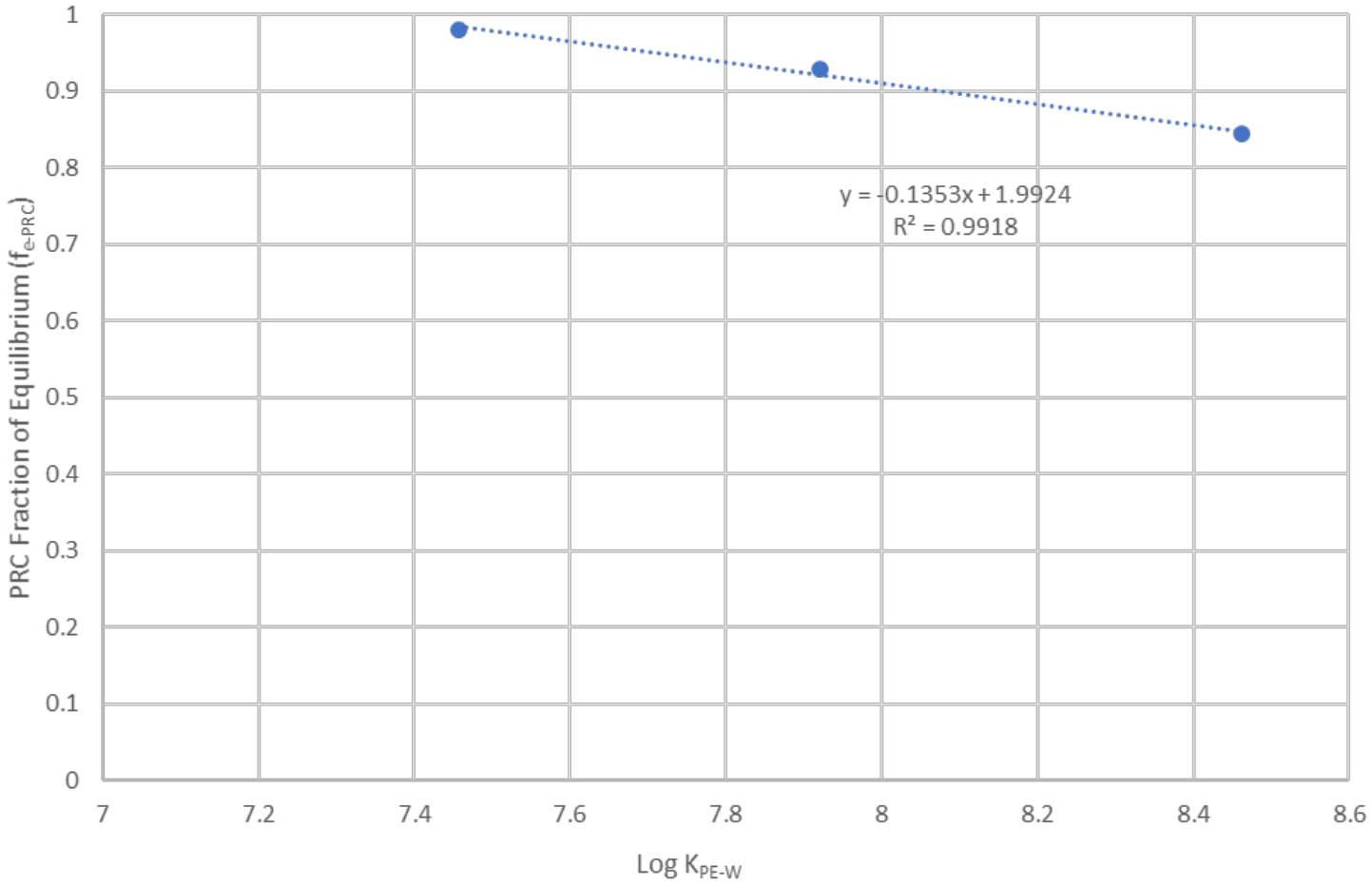

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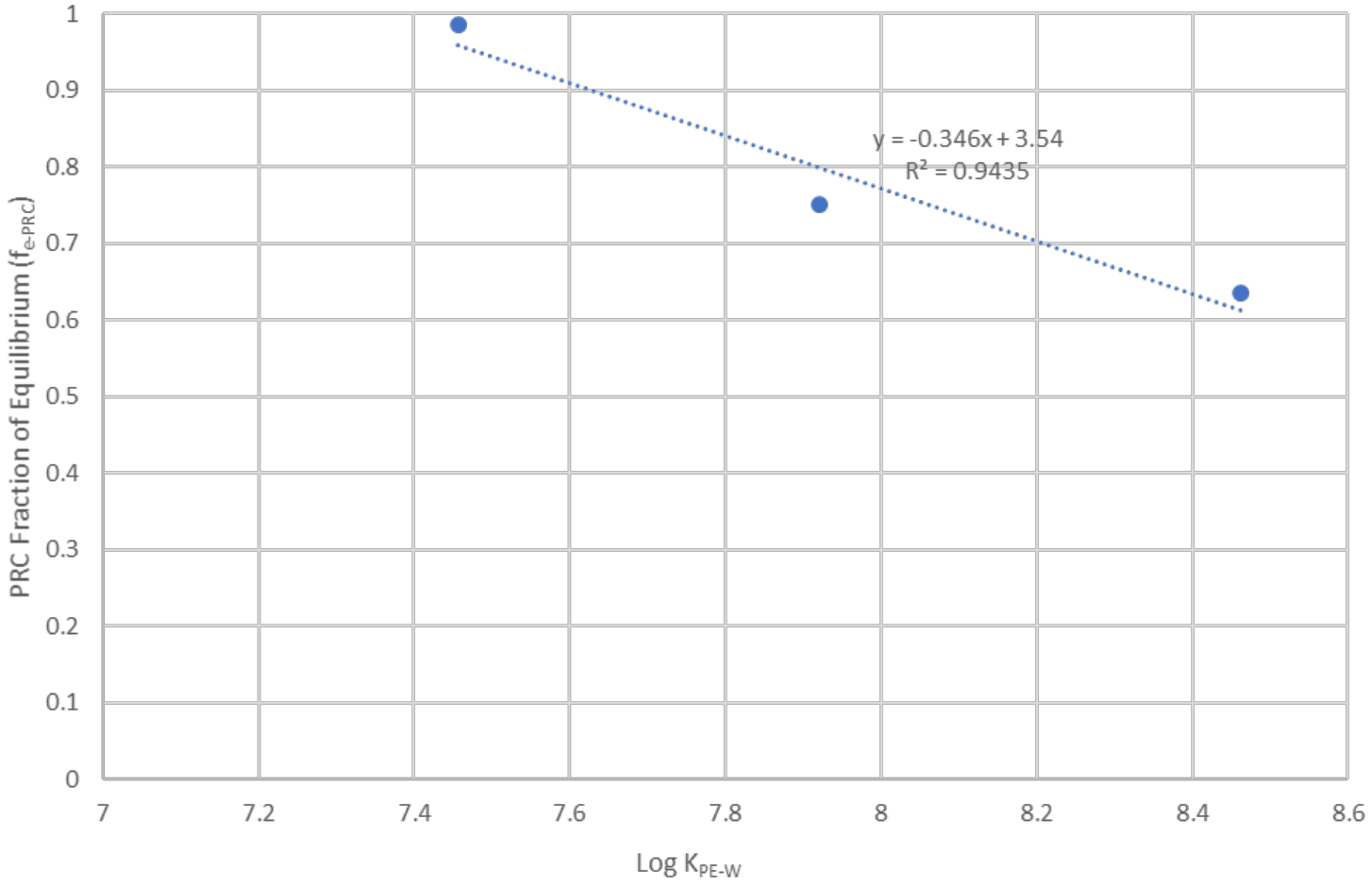

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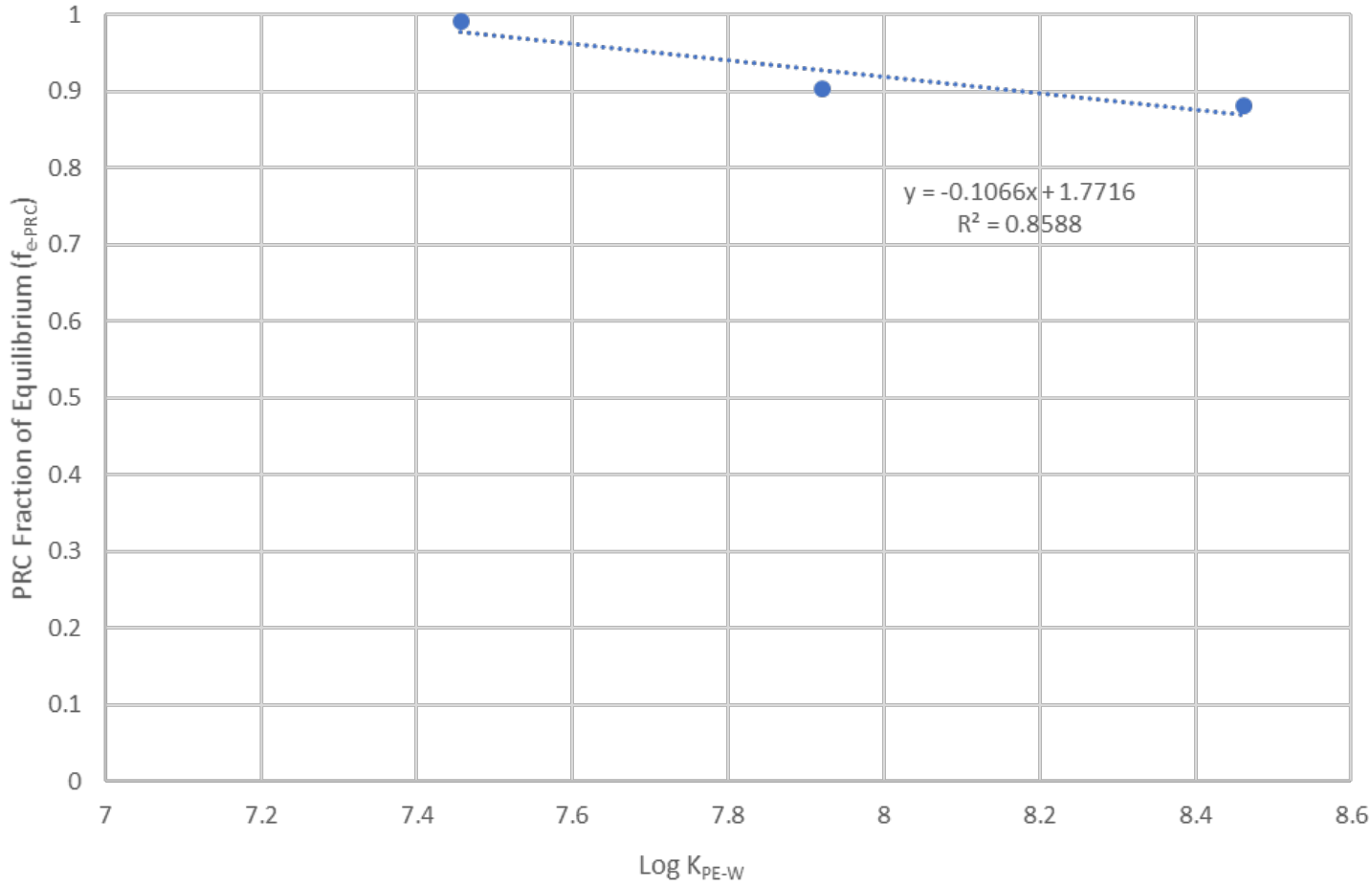

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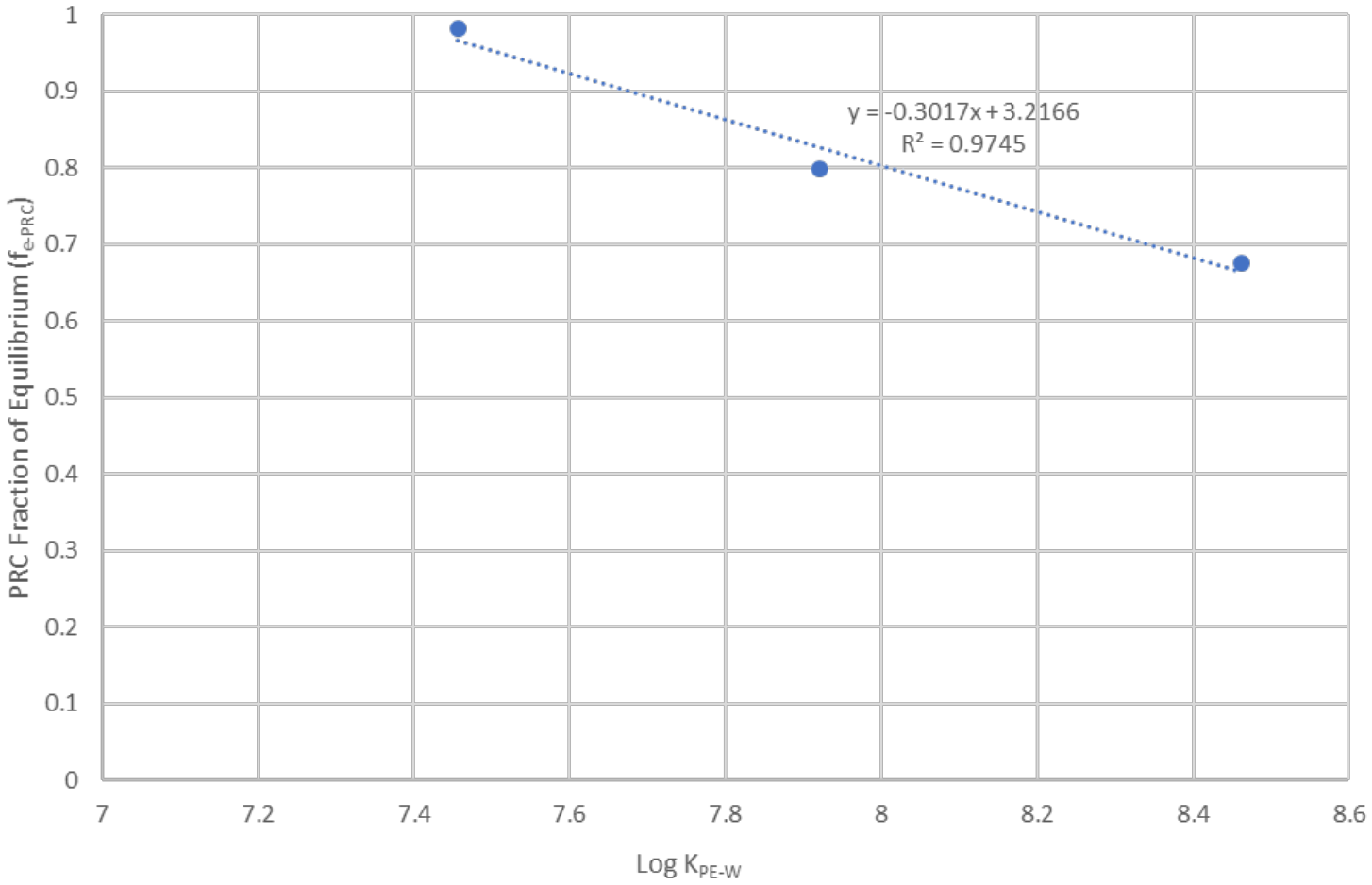

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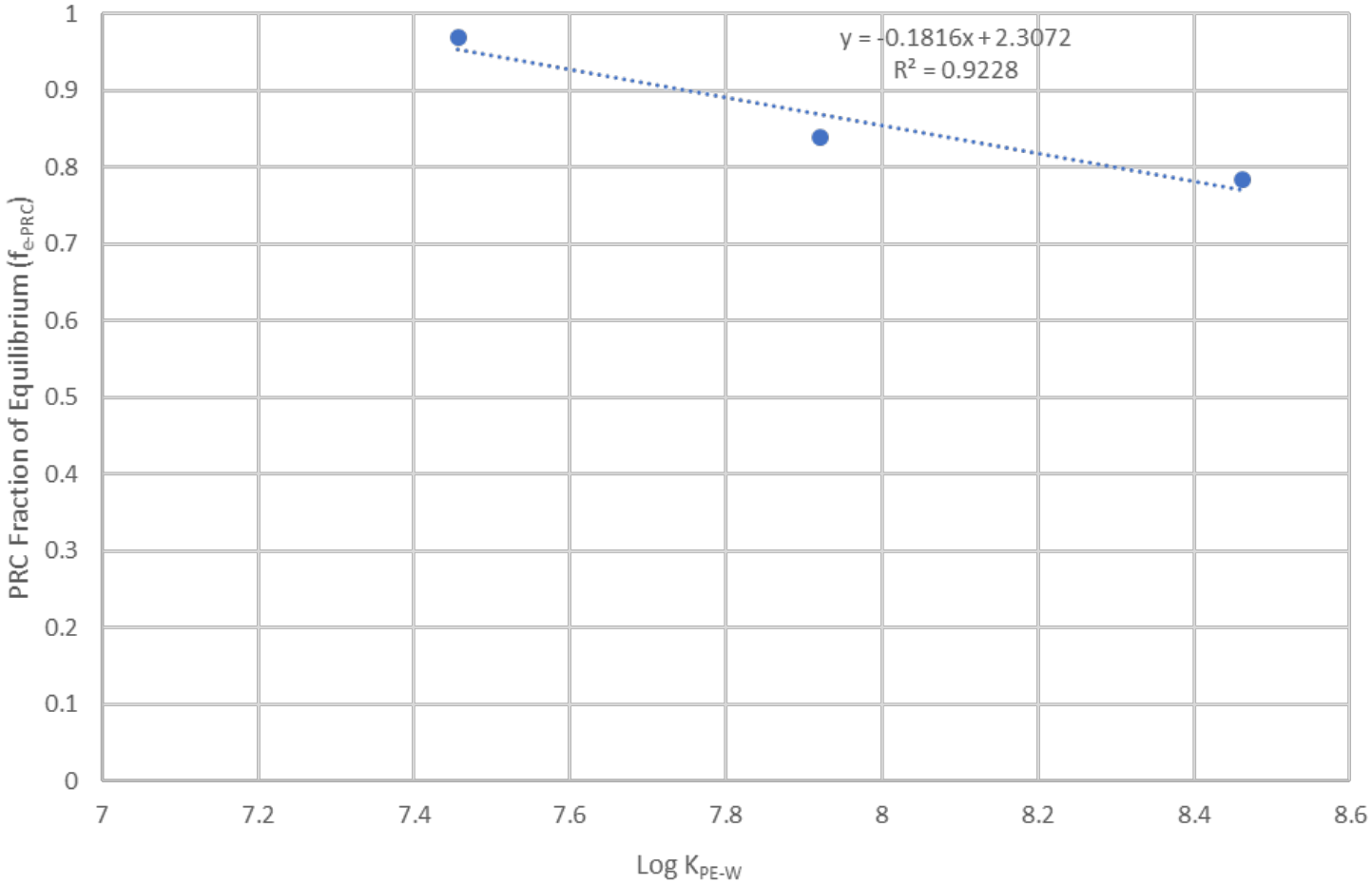

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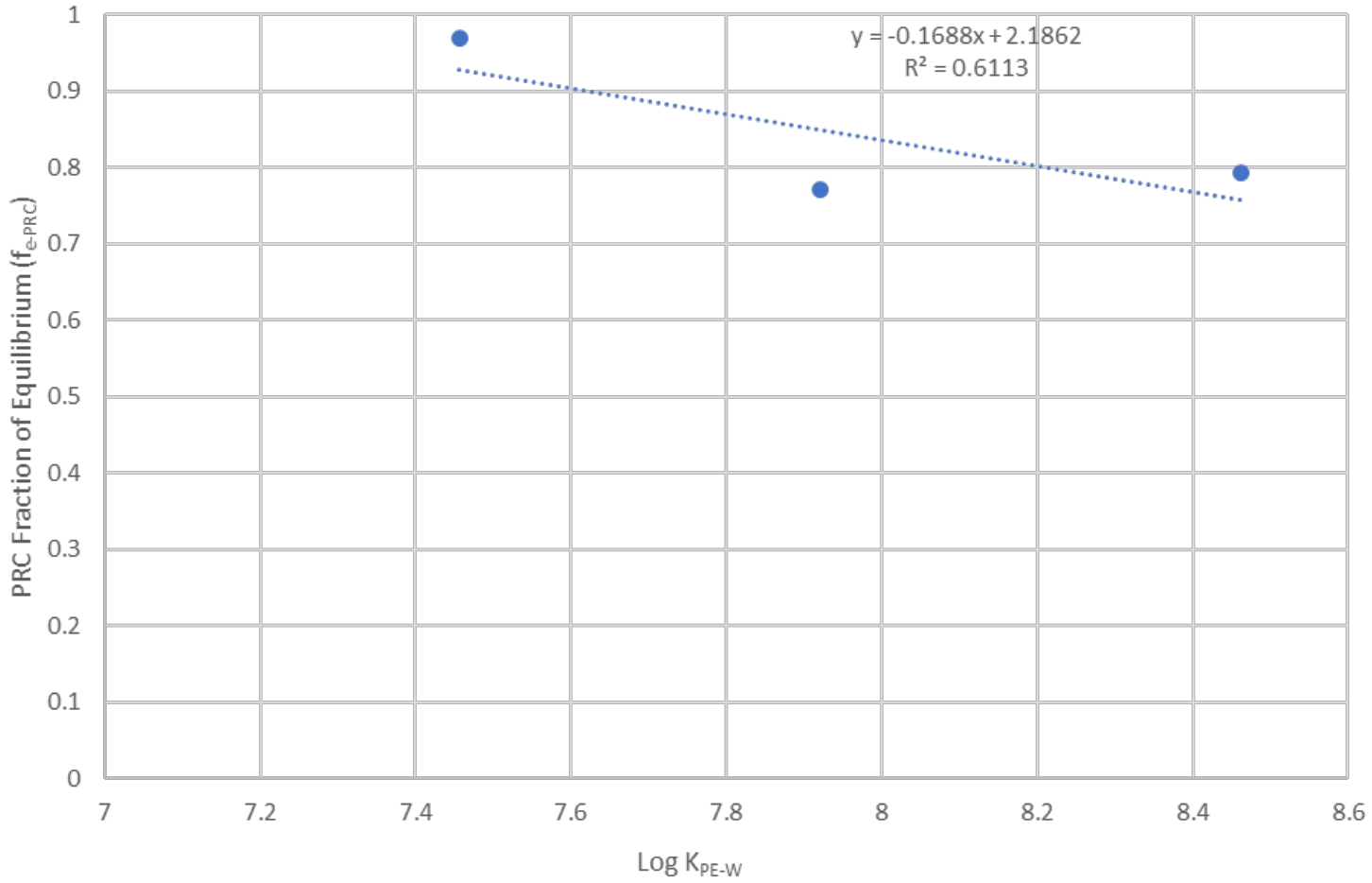

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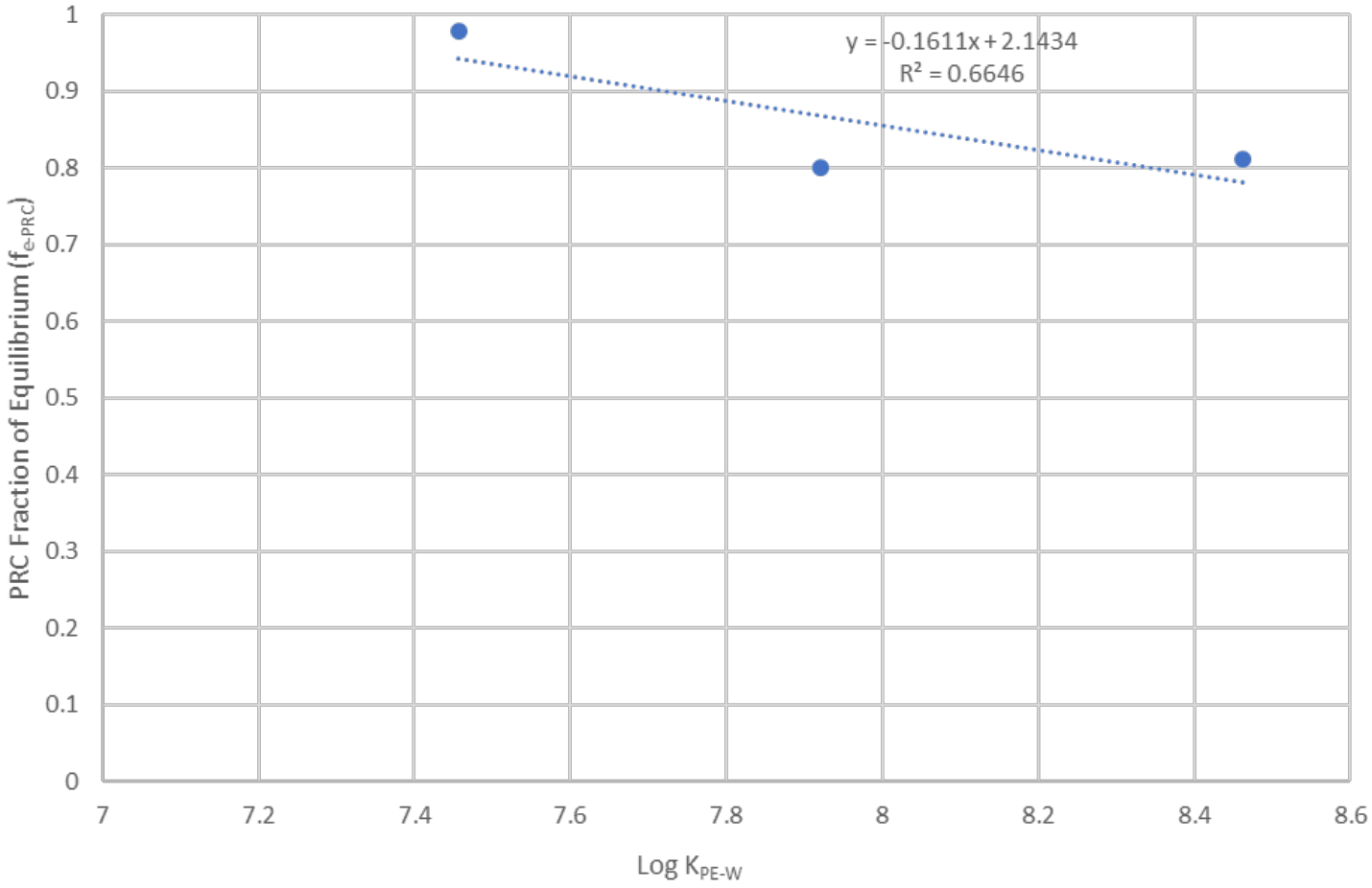

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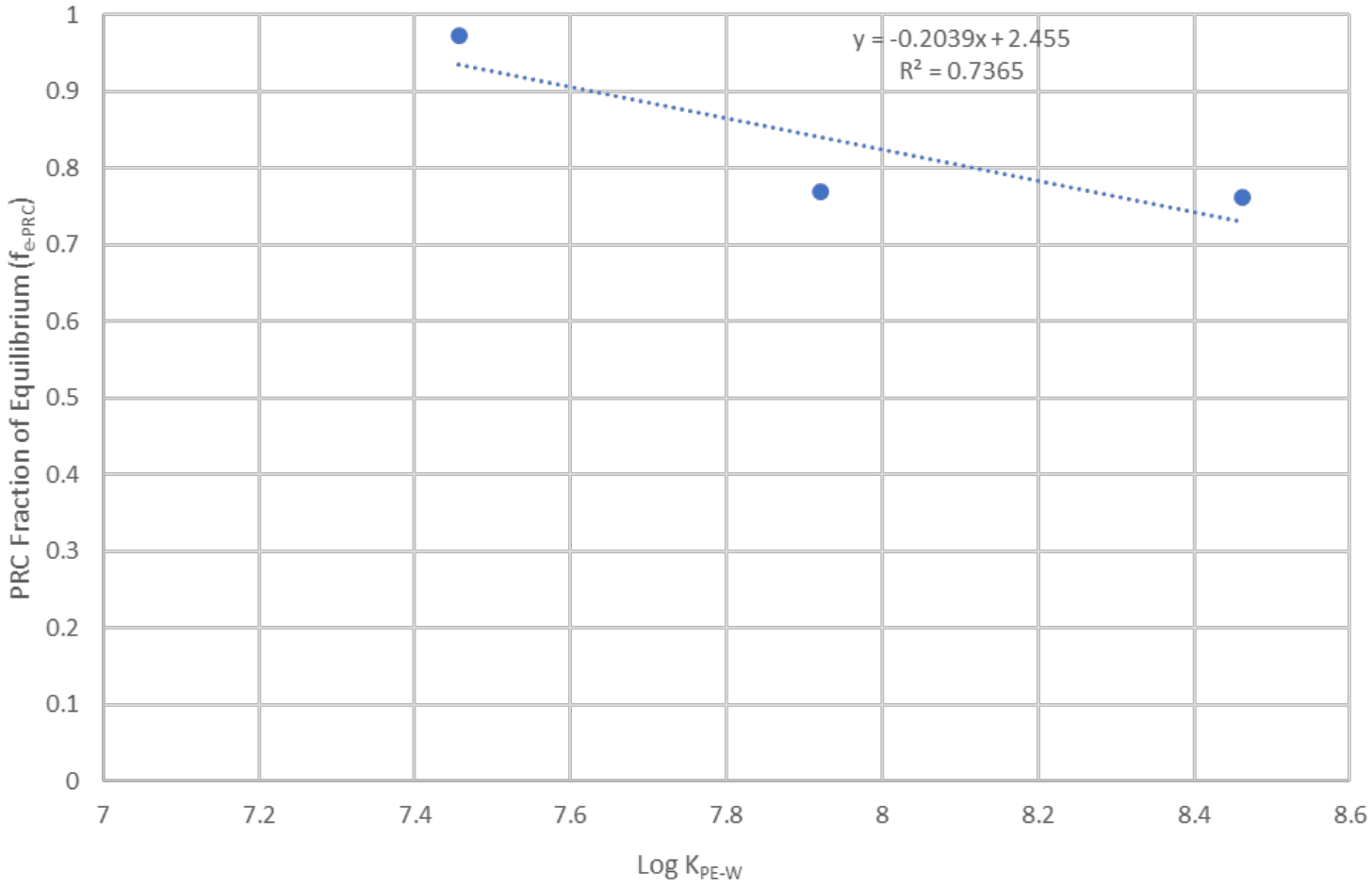

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Appendix C
Data Validation Reports

DRAFT

Data Validation Report – EPA Stage 2A

December 20, 2019

Project: Scanlon Reservoir

Project Number: 191473-02.01

This report summarizes the review of analytical results for 12 low-density polyethylene (LDPE) samples and one field duplicate sample collected on October 28 and November 25, 2019, in Scanlon Reservoir. The samples were collected by the Anchor QEA-Baird Joint Venture (JV) and submitted to SGS North America Inc. (SGS) in Wilmington, North Carolina. The following analytical parameter results were reviewed in this report:

- Polychlorinated dibenzo dioxin and furan (dioxin/furan or PCD/F) by the U.S. Environmental Protection Agency (EPA) Method 1613B

SGS sample delivery group (SDG) numbers B3820 and B3916 were reviewed in this report. Sample IDs, SDGs, matrices, and analyses are presented in Table 1.

Table 1
Sample IDs, SDGs, Matrices, and Analyses

Sample ID	Laboratory Sample ID	Matrix	Analysis
SR-PE-MB-20191028	B3820_17099_DF_001	LDPE	PCD/F
SR-PE-PRC-C0-1-20191028	B3820_17099_DF_002	LDPE	PCD/F
SR-PE-PRC-C0-2-20191028	B3820_17099_DF_003	LDPE	PCD/F
SR-PE-PRC-C0-3-20191028	B3820_17099_DF_004	LDPE	PCD/F
SR-2019-04-PE-CTRL	B3916_17162_DF_001	LDPE	PCD/F
SR-2019-04-PE-4PAC	B3916_17162_DF_002	LDPE	PCD/F
SR-2019-04-PE-4PAC-D	B3916_17162_DF_003	LDPE	PCD/F
SR-2019-04-PE-2PAC	B3916_17162_DF_004	LDPE	PCD/F
SR-2019-04-PE-4GAC	B3916_17162_DF_005	LDPE	PCD/F
SR-2019-06-PE-CTRL	B3916_17162_DF_006	LDPE	PCD/F
SR-2019-06-PE-4PAC	B3916_17162_DF_007	LDPE	PCD/F
SR-2019-06-PE-2PAC	B3916_17162_DF_008	LDPE	PCD/F
SR-2019-06-PE-4GAC	B3916_17162_DF_009	LDPE	PCD/F

Data Validation and Qualifications

The following comments refer to the laboratory's performance in meeting the quality assurance (QA)/quality control (QC) guidelines outlined in the analytical procedures. Laboratory results were reviewed using the following guidelines:

- *Quality Assurance Project Plan* (QAPP) for the Research and Development Pilot Project Design for Remediation of Contaminated Sediments at the Scanlon Reservoir (JV 2019)

- *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods* (EPA 1986)
- *National Functional Guidelines for High Resolution Superfund Methods Data Review* (EPA 2016)

Unless noted in this report, laboratory results for the samples listed above were within QC criteria.

Field Documentation

Field documentation was checked for completeness and accuracy. The chain-of-custody forms were signed by SGS at the time of sample receipt. Samples were received in good condition and within the recommended temperature range.

Sample Preservation and Holding Times

Samples were appropriately preserved and analyzed within holding times.

Laboratory Method Blanks

Laboratory method blanks were analyzed at the required frequencies. All blanks were free of target analytes, with the exception of Method Blank B3916_17162, which had detected results for 11 analytes. Associated sample results that were not significantly greater than (greater than five times) the levels found in the blank were qualified as non-detected.

Field Quality Control

Field Duplicate

One field duplicate was collected in association with this sample set. Detected results are summarized in Table 2.

Table 2
Field Duplicate Summary

Analyte	SR-2019-04-PE-4PAC	SR-2019-04-PE-4PAC-D	RPD	Difference	Control Limit
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	185 pg	98.7 pg	--	86.3 pg	100 pg
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	1,080 pg	556 pg	64.1%	--	--
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	484 pg	348 pg	32.7%	--	--
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	141 pg	90.2 pg	--	50.8 pg	50 pg
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	2.52J pg	3.39J EMPC pg	--	0.87 pg	50 pg
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	6.97J pg	6.02J pg	--	0.95 pg	50 pg
Total Heptachlorodibenzofuran (HpCDF)	894 pg	637 pg	33.6%	--	--
Total Heptachlorodibenzo-p-dioxin (HpCDD)	330 pg	207 pg	45.8%	--	--
Total Hexachlorodibenzofuran (HxCDF)	137EMPC pg	138EMPC pg	0.7%	--	--
Total Hexachlorodibenzo-p-dioxin (HxCDD)	65.1EMPC pg	58.7 pg	--	6.4 pg	50 pg

Notes:
pg: picogram
RPD: relative percent difference
EMPC: estimated maximum possible concentration

Results less than five times the method reporting limit (RL) may have exaggerated relative percent difference (RPD) values; therefore, if the sample or field duplicate result was less than five times the RL, the sample result is evaluated by the difference between them using ± 2x the RL as the control limit.

All RPD and/or difference values were within control limits, with the exception of the 1,2,3,4,6,7,8-octachlorodibenzo-p-dioxin (OCDD) RPD value and the 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD) difference value for samples SR-2019-04-PE-4PAC and SR-2019-04-PE-4PAC-D, which were above the control limits. These results have been qualified "J" to indicate they are estimated.

Qualified data are summarized in Table 3.

Labeled Standard Recoveries

All labeled standards were recovered within laboratory control limits in the PCD/F analyses.

Laboratory Control or Ongoing Precision and Recovery Samples

Laboratory control samples and ongoing precision and recovery (OPR) samples were analyzed at the required frequency and resulted in recoveries within project-required control limits.

Matrix Spike and Matrix Spike Duplicate Samples

Matrix spike samples were not required to be analyzed with this sample set.

Laboratory Duplicates

Laboratory duplicates were not required to be analyzed with this sample set.

Estimated Maximum Potential Concentration

The laboratory qualified PCD/F results that did not meet ion-abundance ratio requirements as estimated maximum possible concentration (EMPC); these results have been qualified "J" to indicate they are estimated.

Method Reporting Limits

RLs were acceptable as reported. All values were reported using the laboratory RLs. Values were reported as undiluted. Some RLs were slightly elevated above the target limits listed in the QAPP due to sample aliquot size and moisture content. Data quality objectives are not expected to be impacted.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods, and all requested sample analyses were completed. Accuracy was acceptable as demonstrated by the OPR recovery values. Precision was acceptable as demonstrated by the field duplicate RPD or difference values, with the exceptions noted above. Most data are acceptable as reported, and all other data are acceptable as qualified. Table 3 summarizes the qualifiers applied to the sample results reviewed in this report.

Data Qualifier Definitions

J Indicates an estimated value.

U Indicates the compound or analyte was analyzed for but not detected at or above the specified limit.

Table 3
Data Qualification Summary

Sample ID	Parameter	Analyte	Reported Result	Qualified Result	Reason
SR-2019-04-PE-CTRL	PCD/F	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	10.3J B pg	10.3U pg	Method blank contamination
		1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	14.2J EMPC pg	14.2J pg	EMPC
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	612EMPC pg	612J pg	
		Total Pentachlorodibenzofuran (PeCDF)	173EMPC pg	173J pg	
		Total Pentachlorodibenzo-p-dioxin (PeCDD)	181EMPC pg	181J pg	
		Total Tetrachlorodibenzofuran (TCDF)	30EMPC pg	30J pg	
		Total Tetrachlorodibenzo-p-dioxin (TCDD)	28.5EMPC pg	28.5J pg	
SR-2019-04-PE-4PAC	PCD/F	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	1080 pg	1080J pg	Field duplicate RPD above control limit
		1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	141 pg	141J pg	Field duplicate difference value above control limit
		1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	5.21J B EMPC pg	5.21U pg	Method blank contamination
		1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	6.19J B pg	6.19U pg	
		Total Hexachlorodibenzofuran (HxCDF)	137EMPC pg	137J pg	EMPC
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	65.1EMPC pg	65.1J pg	
SR-2019-04-PE-4PAC-D	PCD/F	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	556 pg	556J pg	Field duplicate RPD above control limit
		1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	90.2 pg	90.2J pg	Field duplicate difference value above control limit
		1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	3.39J EMPC pg	3.39J pg	EMPC
		Total Hexachlorodibenzofuran (HxCDF)	138EMPC pg	138J pg	
		1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	5.36J B pg	5.36U pg	Method blank contamination
		1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	4.51J B pg	4.51U pg	
		2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	1.65J B EMPC pg	1.65U pg	

Sample ID	Parameter	Analyte	Reported Result	Qualified Result	Reason
SR-2019-04-PE-2PAC	PCD/F	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	5.85J EMPC pg	5.85U pg	Method blank contamination
		2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	4.21J pg	4.21U pg	
		1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	6.71J EMPC pg	6.71J pg	EMPC
		1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	15.1J EMPC pg	15.1J pg	
		Total Heptachlorodibenzofuran (HpCDF)	1410EMPC pg	1410J pg	
		Total Hexachlorodibenzofuran (HxCDF)	461EMPC pg	461J pg	
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	213EMPC pg	213J pg	
SR-2019-04-PE-4GAC	PCD/F	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	7.35J pg	7.35U pg	Method blank contamination
		1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	34.7EMPC pg	34.7J pg	EMPC
		Total Hexachlorodibenzofuran (HxCDF)	941EMPC pg	941J pg	
		Total Pentachlorodibenzofuran (PeCDF)	40.8EMPC pg	40.8J pg	
		Total Pentachlorodibenzo-p-dioxin (PeCDD)	32.9EMPC pg	32.9J pg	
SR-2019-06-PE-CTRL	PCD/F	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	5.71J pg	5.71U pg	Method blank contamination
		1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	2.57J EMPC pg	2.57J pg	EMPC
		1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	5.51J EMPC pg	5.51J pg	
		Total Pentachlorodibenzofuran (PeCDF)	83.7EMPC pg	83.7J pg	
		Total Pentachlorodibenzo-p-dioxin (PeCDD)	81.1EMPC pg	81.1J pg	
		Total Tetrachlorodibenzofuran (TCDF)	4.7EMPC pg	4.7J pg	
		Total Tetrachlorodibenzo-p-dioxin (TCDD)	4.3EMPC pg	4.3J pg	
SR-2019-06-PE-4PAC	PCD/F	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	14.6J EMPC pg	14.6U pg	Method blank contamination
		Total Heptachlorodibenzo-p-dioxin (HpCDD)	37.4EMPC pg	37.4J pg	EMPC
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	9.67EMPC pg	9.67J pg	
SR-2019-06-PE-2PAC	PCD/F	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	3.8J EMPC pg	3.8J pg	EMPC
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	48.7EMPC pg	48.7J pg	
SR-2019-06-PE-4GAC	PCD/F	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	3.41J EMPC pg	3.41J pg	EMPC
		1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	5.56J EMPC pg	5.56J pg	
		Total Hexachlorodibenzofuran (HxCDF)	208EMPC pg	208J pg	
		Total Hexachlorodibenzo-p-dioxin (HxCDD)	84.2EMPC pg	84.2J pg	
		1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	3.12J EMPC pg	3.12U pg	Method blank contamination
		2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	3.09J EMPC pg	3.09U pg	

Notes:
EMPC: estimated maximum possible concentration
pg: picogram
RPD: relative percent difference

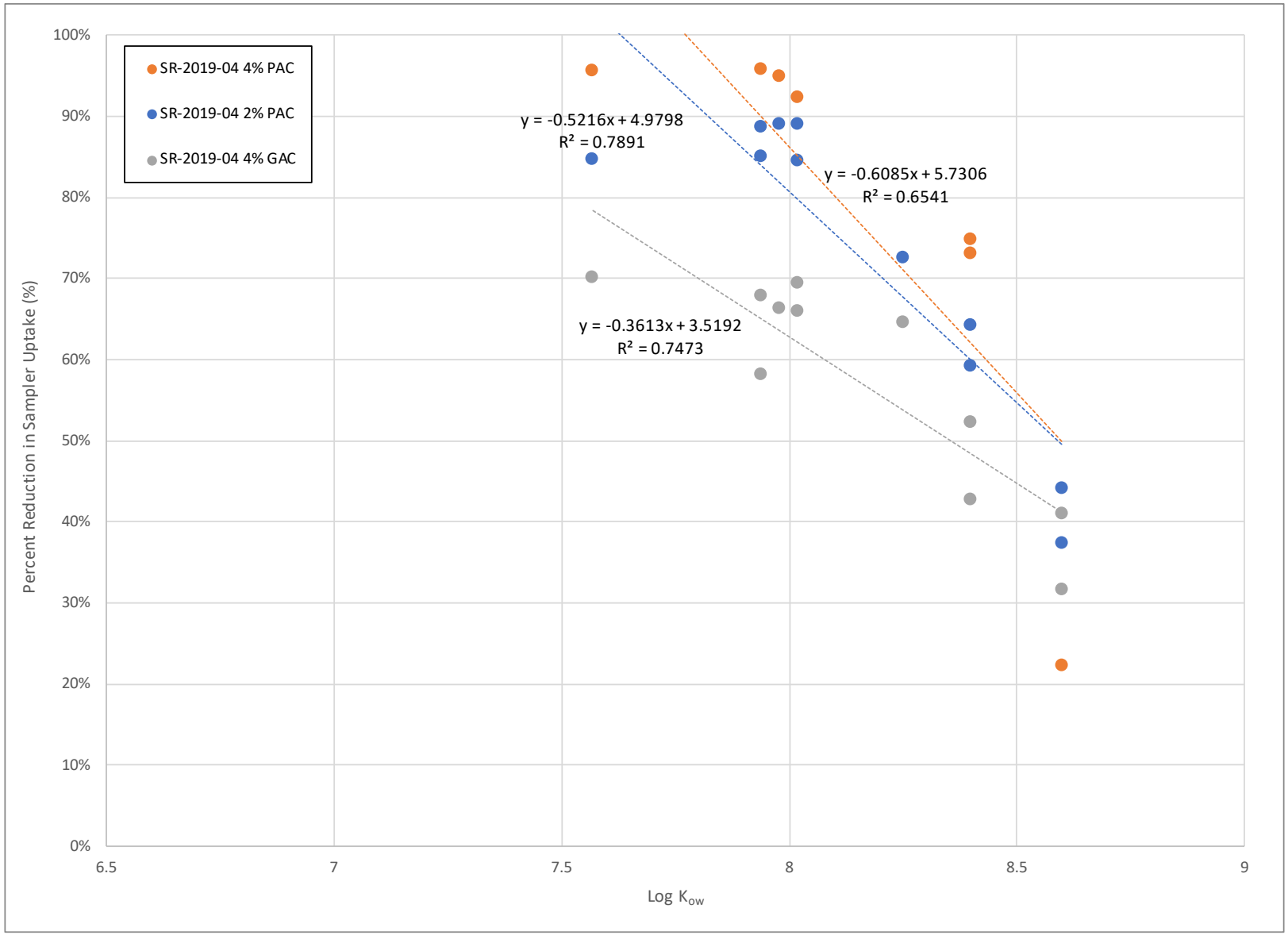
References

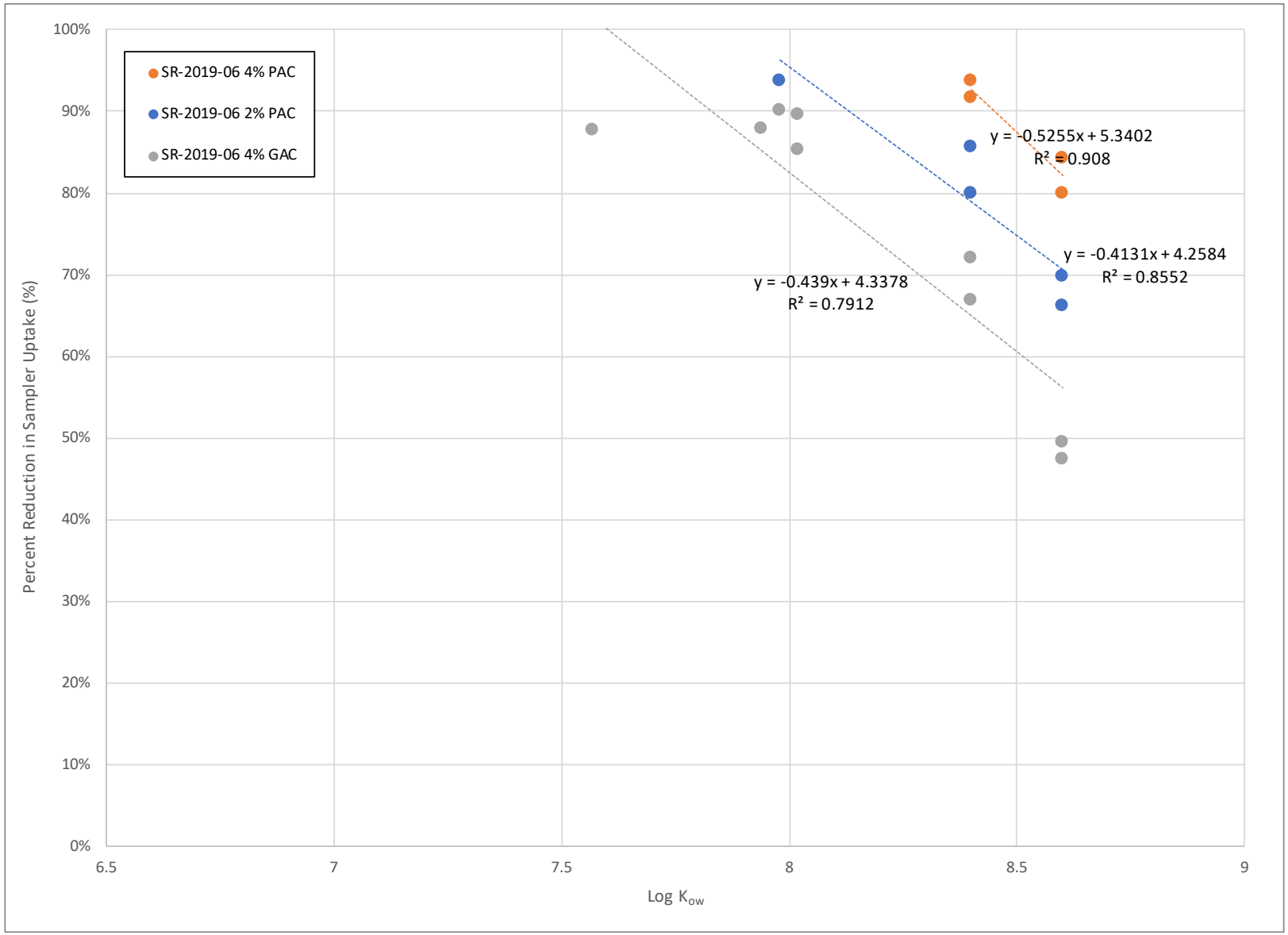
EPA (U.S. Environmental Protection Agency) 1986. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. EPA 530/SW-846.

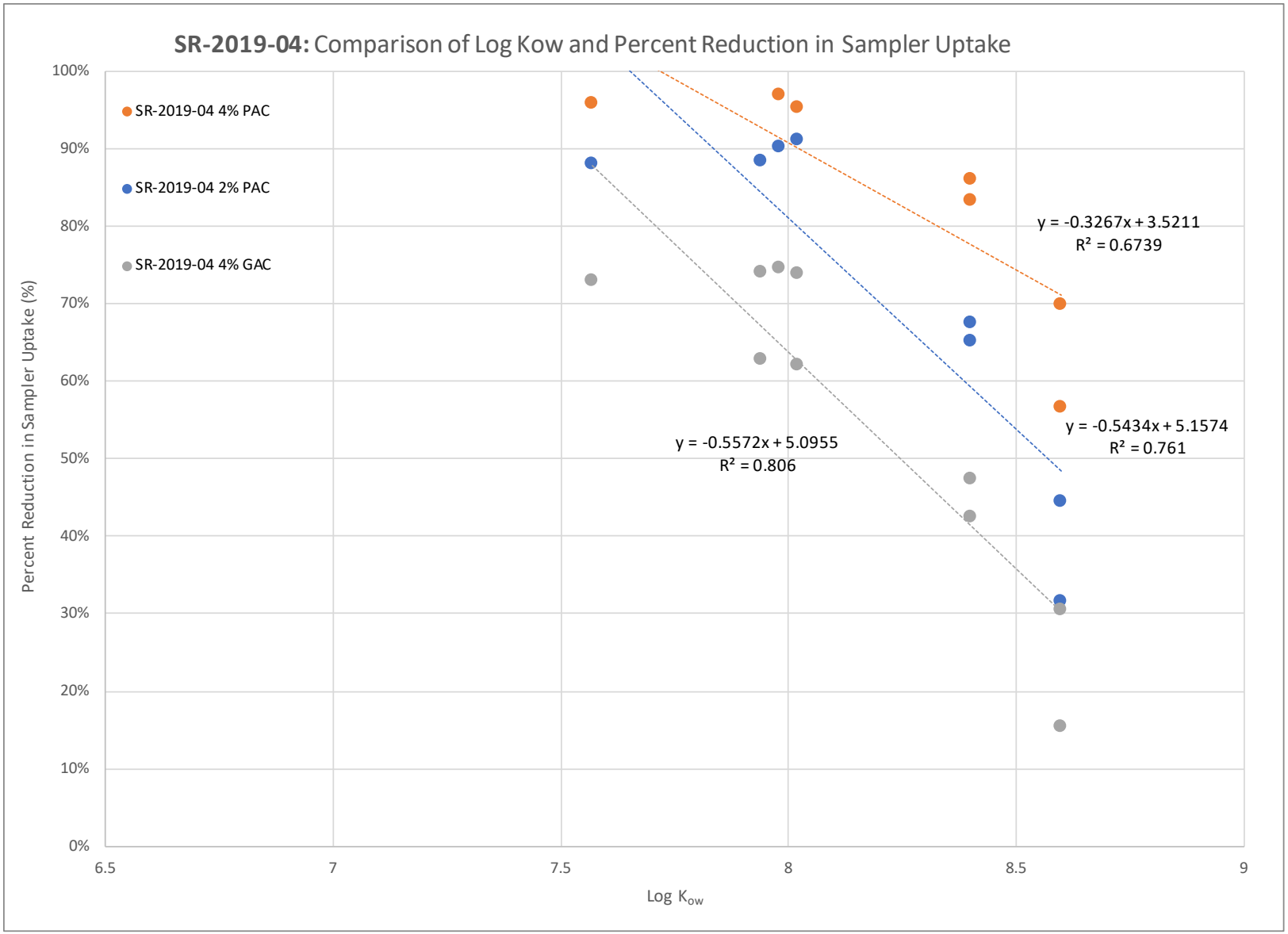
EPA 2016. *National Functional Guidelines for High Resolution Superfund Methods Data Review*. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation (OSRTI). EPA 542-B-16-001. April 2016.

JV (Anchor QEA-Baird Joint Venture), 2019. *Quality Assurance Project Plan*. Research and Development Pilot Project Design for Remediation of Contaminated Sediments at the Scanlon Reservoir, Scanlon, Minnesota. Contract Number W912P4-D-0001. Prepared for U.S. Army Corps of Engineers – Detroit District. September 2019.

Appendix D
Effect of K_{ow} on Reduction in Sampler Uptake


SR-2019-04 4% PAC
SR-2019-04 2% PAC
SR-2019-04 4% GAC
y = -0.5216x + 4.9798
R² = 0.7891
y = -0.6085x + 5.7306
R² = 0.6541
y = -0.3613x + 3.5192
R² = 0.7473
Percent Reduction in Sampler Uptake (%)
100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
6.5
7
7.5
8
8.5
9
Log Kow


SR-2019-06 4% PAC
SR-2019-06 2% PAC
SR-2019-06 4% GAC
y = -0.5255x + 5.3402
R² = 0.908
y = -0.4131x + 4.2584
R² = 0.8552
y = -0.439x + 4.3378
R² = 0.7912
Percent Reduction in Sampler Uptake (%)
Log Kow
100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
6.5
7
7.5
8
8.5
9



ANCHOR QEA Baird Joint Venture

Appendix D - Figure 3
Set 2 Effect of Kow on Percent Reduction – SR-2019-04
Benchscale Treatability Testing Report
Scanlon Reservoir Pilot Remediation Project Design