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Revision 2
Dredged Material Management Program
Dredged Material Characterization Report
8 January 2019

8 January 2019

Ms. Lauran Cole-Warner
Dredged Material Management Office
U.S. Army Corps of Engineers, Seattle District
P.O. Box 3755
Seattle, WA 98124

Subject: Dredged Material Characterization
Proposed Grays Harbor Potash Export Facility
Hoquiam, Washington

Dear Ms. Cole-Warner:

On behalf of BHP Billiton Canada Inc. (BHP), BergerABAM is pleased to submit our report "Revised Dredged Material Characterization, Proposed Grays Harbor Potash Export Facility, Hoquiam, Washington." This characterization was conducted to evaluate the potential suitability of dredged material for open-water placement. BHP requests that the attached data be evaluated relative to Dredged Material Management Program and Washington State Sediment Management Standards criteria for open-water placement and beneficial use of dredged materials. The previously submitted report (dated 17 May 2018) has been updated to include the follow-up sampling and characterization of the proposed dredged material that was completed in August 2018.

We appreciate the guidance and assistance that the Dredged Material Management Office provided throughout this project. Please contact us if you have questions regarding this report.

Sincerely,

Victoria R. England, LG
Senior Environmental Scientist

April Ryckman

April R. Ryckman
Environmental Scientist

VRE:ARR:nb
Attachment

cc w/attach: Valerie Bond, BHP Billiton Canada Inc.

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
Hoquiam, Washington

Prepared for

U.S. Army Corps of Engineers, Seattle District
Dredged Material Management Office
Seattle, Washington

Attention: Lauran Cole-Warner

31 October 2018

Prepared by

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A17.0202.00

REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY

U.S. Army Corps of Engineers, Seattle District
Dredged Material Management Office
Seattle, Washington

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LIST OF ACRONYMS AND ABBREVIATIONS

BHP	BHP Billiton Canada Inc.
COCs	chemicals of concern
DMMO	Dredged Material Management Office
DMMP	Dredged Material Management Program
DMMU	dredged material management unit
EPA	Environmental Protection Agency
GPS	global positioning system
MLLW	mean lower low water
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
Port	Port of Grays Harbor
PSEP	Puget Sound Estuary Program
QA/QC	quality assurance/quality control
RSS	Research Support Services
SAP	Sampling and Analysis Plan
SAPA	Sampling and Analysis Plan Addendum
SL	screening level
SMS	Sediment Management Standards
SVOC	semi-volatile organic compounds

REVISED DREDGED MATERIAL CHARACTERIZATION
PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
HOQUIAM, WASHINGTON

1.0 INTRODUCTION

This report presents the results of sediment sampling activities for the characterization of proposed dredged material at the BHP Billiton Canada Inc. (BHP) proposed new potash export facility (also referred to as the "project site") located in Hoquiam, Washington. BHP proposes to construct a new marine berth and support facilities west of and adjacent to the Port of Grays Harbor (Port) Terminal 3 (Sheet 1). Dredging will be required to accommodate the potash export vessels that will be using the berth and the shiploading facilities at the site.

The purpose of this characterization is to evaluate the suitability of the proposed dredged material for open-water disposal. The sampling plans were in accordance with the Dredged Material Management Program (DMMP) User's Manual dated August 2016. The SAP and SAPA were approved by the Dredged Material Management Office (DMMO) in emails dated 23 February and 30 August 2018, respectively. The SAP, SAPA, and the DMMO approval emails are included as Appendices A and C.

1.1 Project Description

BHP proposes to dredge approximately 110,000 cubic yards of sediment to provide a new berth to support a dual-quadrant shiploader capable of servicing bulk material export vessels. The proposed dredging activities will commence during the 2019/2020 in-water work window.

The Port's Terminal 3 berth is currently authorized to a depth of -41 feet mean lower low water (MLLW) with 2 feet of overdredge allowance. The proposed depth for the new potash export facility berth is -43 feet MLLW plus 2 feet of allowable overdredge to -45 feet MLLW.

The new berth, west of and adjacent to existing Terminal 3 berth, will extend from the edge of the navigation channel to the face of the proposed shiploader trestle (see Sheet 2). The footprint of the new berth will be approximately 4.72 acres, and the dredge area is approximately 7.49 acres. The total volume to be dredged is approximately 110,000 cubic yards.

Existing bathymetry in the proposed dredge area ranges from -1 to -41 feet MLLW. The dredge prism cut will range in thickness from 3 to 23 feet below mudline (including overdredge allowance). The number of dredged material management units (DMMUs) and associated proposed subsamples are based upon the DMMP ranking of low-moderate. The boundaries of each DMMU are based on existing bathymetry, volume, and proposed dredge depth.

2.0 PREVIOUS DREDGING – TERMINAL 3 BERTH

The Terminal 3 berth has a low ranking based on the Port's previous completed studies and the DMMP's determination. Maintenance dredging of the proposed berth is expected to be completed at the same (or greater) frequency as for the Terminal 3 berth, which is maintenance dredged every four years on average. Three rounds of maintenance dredging have been completed at Terminal 3 between 2001 and 2012 with volumes ranging from 22,000 to 66,000 cubic yards.

The DMMP has issued four suitability determinations for material dredged for maintenance and/or deepening at the adjacent Terminal 3 berth from 2008 through 2015. All of the suitability determinations issued for Terminal 3 determined all of the characterized material to be suitable for open-water disposal.

3.0 SAMPLING AND ANALYSIS PROGRAM

The objectives of this sediment characterization report are to evaluate the following.

- Suitability of the proposed dredged material for disposal at a DMMP unconfined (dispersive) open-water disposal site (Washington State Department of Natural Resources managed South Jetty and Point Chehalis disposal sites)
- Suitability of the proposed dredged material for open-water placement for beneficial reuse
- Sediment quality of the newly exposed post-dredge surface relative to Washington State Sediment Quality Standards and antidegradation policy.
- Upland disposal options for dredged material that is not suitable for DMMP open-water disposal (if needed)

3.1 Sampling Activities – February 2018

The initial sampling of the proposed dredged material was performed on 26 through 28 February 2018. Sampling activities were conducted in accordance with the DMMO-approved SAP with deviations as described in Section 3.2.

Dredged volume, dredge prism configuration, and sampling frequency are based on the DMMP-approved ranking of low-moderate, typical cross sections and conditions within the proposed dredging areas as described in the approved SAP (Appendix A).

A total of six cores were collected within the proposed dredge prism at the locations shown on Sheet 3. The samples were collected with a MudMole pneumatic corer owned and operated by AMEC/WOOD of Lynwood, Washington, with dive support provided by Research Support Services (RSS) of Bainbridge Island, Washington. The sampler is a 4-inch-square aluminum core tube with a pneumatic driving assembly attached on top. The sampler is operated by personnel on the sampling vessel in shallow waters and by a diver in deeper waters. A global positioning system (GPS) was used for positioning at each sample location. Sample depths were adjusted for sample compression in the core tube based on diver-collected recovery measurements during sampling (Appendix D).

BergerABAM representatives monitored sampling activities and processed the samples. Sediment samples were examined, screened for indications of contamination[1], and logged immediately after collection. The collected cores were processed (e.g., subsample collection and compositing) onshore in a Port building located on the Terminal 3 pier. The bore logs and photographs of each sample are presented in Appendix E.

A stainless steel trowel was used to remove sediment sample material from the 4-inch-square aluminum core tube. Samples were homogenized in a stainless steel bowl prior to placing into laboratory-supplied sample containers. Samples were placed into a cooler with ice and submitted under chain-of-custody procedures to Analytical Resources Inc. (Tukwila, Washington) for chemical analytical testing. Archive samples were collected every 2 feet from each sampling location for potential follow-up analysis and/or bioassays.

Sample material was composited into four DMMU samples (DMMU-1 through -4). Table 1 includes a summary of the sample compositing scheme, sample coordinates, tide-corrected mudline elevations, and the sample recovery length. Grain-size data and approximate volumes for each DMMU are in Tables 2 and 3, respectively.

3.2 SAP Deviations – February 2018

Field activities and the analytical program were conducted in accordance with the DMMO-approved SAP, with the exception of the deviations summarized in this section.

Current and wind conditions made it difficult for the sampling support vessels and equipment to maintain positions at the identified core locations. Sample station locations were moved, sample recovery was difficult, cores were not as deep as planned, and cores from two of the eight sample locations (SS-4 and SS-7) could not be collected because of the site conditions.

- The revised sample locations as described below (Sheet 3) were discussed with Lauran Cole-Warner of the DMMO during field activities prior to sample collection.
    - Sample SS-3 moved approximately 29 feet east
    - Sample SS-6 moved approximately 36 feet west
    - Sample SS-8 moved approximately 14 feet north
- Two attempts were made at SS-3 in order to achieve sample recovery.
- Sampling at SS-4 and SS-7 was not tried for due to difficult sampling conditions and subcontractor schedule constraints.

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[1] Field screening included sheen testing, observing evidence of staining or deleterious material (if any), and odor observations. The field screening results are included in the sample logs.

DMMU composites 2 and 4 were composited from fewer subsample locations than planned. DMMU-2 consisted of a composite of subsamples SS-3 and SS-8. DMMU-4 consisted of a composite of subsample SS-8.

The site was revisited in August 2018 to attempt sample collection that is more representative of DMMU-2 and DMMU-4 at the direction of the DMMO and as discussed in Section 3.3.

3.3 Sampling Activities – August 2018

Sampling of the proposed dredged material was performed on 14 through 16 August 2018. Sampling activities were conducted in accordance with the DMMO-approved SAPA (July 2018) with deviations as described in Section 3.4.

A total of four cores were collected within the proposed dredge prism at the locations shown on Sheet 3. The samples were collected with a vibracore owned and operated by RSS of Bainbridge Island, Washington. The sampler is a 4-inch-round core tube with a butyrate liner with an electric vibracorer assembly attached on top. The sampler was operated by personnel on the sampling vessel. A GPS was used for positioning at each sample location.

BergerABAM representatives monitored sampling activities and processed the samples. Sediment samples were examined, screened for indications of contamination, and logged immediately after collection. The collected cores were processed (e.g., subsamples collected and composited) on RSS's boat while docked at the City of Hoquiam's marina. The bore logs and photographs of each sample core are presented in Appendix E.

A stainless steel trowel was used to remove sediment sample material from the 4-inch-round core tube with a liner. Samples were homogenized in a stainless steel bowl prior to placing into laboratory-supplied sample containers. Samples were placed into a cooler with ice and submitted under chain-of-custody procedures to Analytical Resources Inc. (Tukwila, Washington) for chemical analytical testing. Archive samples were collected every 2 feet from each sampling location for potential follow-up analysis and/or bioassays.

Sample material was composited into two DMMU samples (DMMU-2A and DMMU-4A). Table 1 includes a summary of the sample compositing scheme, sample coordinates, tide-corrected mudline elevations, and the sample recovery depth. Grain-size data and approximate volumes for each DMMU are in Tables 2 and 3, respectively.

3.4 SAPA Deviations – August 2018

Field activities and the analytical program were conducted in accordance with the DMMO-approved SAPA (July 2018), with the exception of the deviations summarized in this section.

The current made it difficult for the sampling support vessel and equipment to maintain positions at the identified core locations. Slack tide was needed in order for successful core retrieval.

- The revised sample locations as described below and shown on Sheet 3 were discussed with and approved by Lauran Cole-Warner of the DMMO during field activities prior to sample collection.
  - Sample SS-3A moved approximately 30 feet east
  - Sample SS-3B moved approximately 24 feet east
  - Sample SS-4B moved approximately 560 feet east
- Four attempts were made at SS-7. Only surface sediment (from mudline to 4 feet below mudline) was successfully collected.
- Two attempts at SS-3 (SS-3A and SS-3B) were made very near to each other in an attempt to collect deeper samples from the subsurface material. The samples were near each other due to the short window of slack tide when successful sampling was possible. Though neither core extended into the subsurface, it was thought at the time that use of the surface material for composite grain size and TOC analyses in DMMU 2A was justified. The decisions to collect the samples as described were approved in the field by Lauran Cole-Warner of the DMMO.
- SS-4B is 5 feet north of the DMMU-4 boundary as shown on Sheet 7. The location was approved by Lauran Cole-Warner of the DMMO prior to retrieving the core.
- Composite sample DMMU-2A is composed of surface sediment from SS-3A, SS-3B, and SS-7, also per approval by Lauran Cole-Warner of the DMMO.
- Composite sample DMMU-4A is composed of subsurface sediment from SS-3A, SS-3B, and SS-4B.Material from the bottom 1 foot of recovered material from all three cores was added to the composite sample to ensure adequate sample volume for the chemical analyses. A representative subsample volume was collected from the subsurface material in each core.
- #230 sieve (or 62.5 microns) was not used as the cutoff between fines and sand, a #200 sieve (75 microns) was used by the laboratory instead.

4.0 PHYSICAL AND CHEMICAL ANALYTICAL PROGRAM

4.1 DMMU Samples

Four composite samples, DMMU-1 through DMMU-4, and two composite samples, DMMU-2A and DMMU-4A, were submitted to Analytical Resources Inc. in Tukwila, Washington, for physical and chemical analyses in February and August 2018, respectively.

Analyses were performed in accordance with applicable Environmental Protection Agency (EPA) methodology along with DMMP and Puget Sound Estuary Program (PSEP) protocols as appropriate, including the following.

- Total organic carbon by SM5310B/EPA Method 9060 (modified for sediments)
- Total solids by PSEP/SM2540G
- Ammonia by Plumb (1981)
- Sulfides by PSEP and Plumb (1981)
- Grain size by PSEP/ASTM D-422 (modified)
- Total metals and mercury using EPA Methods 6010/6020/7440/7471
- Semi-volatile organic compounds (SVOCs) using EPA Method 8270D
- Polycyclic aromatic hydrocarbons (PAHs) using EPA Method 8270D
- Chlorinated hydrocarbons using EPA Methods 8260B/8270D/8081
- Phthalates, phenols, and miscellaneous extractables using EPA Methods 8270D/8081
- Pesticides using EPA Method 8081
- Polychlorinated biphenyls (PCBs) using EPA Method 8082
- Bulk tributyltin using PSEP, Krone (1989), and Unger (1986)
- Dioxins/furans using EPA Method 1613B

DMMU-2A, collected in August 2018, was only analyzed for total organic carbon by SM5310B/EPA Method 9060 and grain size by PSEP/ASTM D-422 per the DMMO-approved SAPA.

The results of the grain-size analyses are summarized in Table 2. The chemical analytical program consists of the DMMP and Sediment Management Standard (SMS) chemicals of concern (COCs) shown in Tables 4 and 5. The chemical analytical results are shown relative to DMMP criteria in Table 4 and SMS criteria in Table 5.

4.2 Quality Assurance and Quality Control

The laboratory reports are included as Appendices F and G. Review of the data quality of the chemical analytical results indicates that laboratory goals were achieved based on the results of quality assurance/quality control (QA/QC) parameters, including surrogates, spikes, replicates, and method blanks except where noted and as flagged by the analytical laboratory. The QA/QC review summary is included as Appendix H.

5.0 ANALYTICAL RESULTS

5.1 Grain Size Characteristics

The grain size results from the dredge prism samples are summarized in Table 2. The proposed dredged material primarily consists of fine and very fine sand interbedded with silt and clay.

The additional grain size analysis of DMMU-2A (August 2018) shows that material to be similar to DMMUs-1, -3, -4, and -4A with the previously analyzed DMMU-2 (February 2018) having a relatively low percentage of fines. The grain size variability in DMMU-2 is likely a result of localized variability of grain size from location to location within the dredge prism and potentially may be affected by seasonal variations in sedimentation.

5.2 Chemical Data Relative to DMMP and SMS Criteria - DMMUs

The chemical analytical results from the sediment characterization are summarized relative to DMMP criteria and SMS criteria in Tables 4 and 5, respectively. Contaminants of concern were either not detected or were detected at concentrations less than the applicable DMMP and SMS criteria in the DMMU composite sediment samples collected from DMMU-1 through DMMU-4, and DMMU-4A in February and August 2018. DMMU-2A in August 2018 was analyzed for TOC and grain size only.

6.0 SUMMARY AND CONCLUSIONS

6.1 Data Gaps

Based on analytical results and coordination with the DMMP, we believe that the data collected to date should be sufficient to evaluate the suitability of the proposed dredge material and data did not indicate increasing contamination with depth. While the available data indicate the proposed dredged material is suitable for open-water placement, we acknowledge the following data gaps.

6.1.1 February 2018

- Full penetration (including Z-layer) relative to the SAP was only achieved in core SS-6; see Table A below for depths achieved).
- Planned cores at two locations were not completed because of site conditions and schedule restraints in February 2018. This resulted in fewer subsampling locations than planned in DMMUs-2 and -4. DMMU-2 consisted of subsamples SS-3 and SS-8, and DMMU-4 consisted of subsample SS-8.
- Follow-up sampling was completed in August 2018 to collect additional samples in DMMU-4 material and to reanalyze DMMU-2 material for grain size.

6.1.2 August 2018

- Full penetration (including Z-layer) relative to the SAPA was not achieved in any of the cores. The bottom 1 foot of the core was used to represent the Z-layer (see Table B below for depths achieved).
- Subsurface material from subsample SS-7 was not collected as noted in Section 3.4.
- Three subsample locations (SS-3A, SS-3B, and SS-4B) were moved with the approval of Lauran Cole-Warner of the DMMO as noted in Section 3.4.

Table A: Planned versus Actual Compositing Scheme – February 2018

DMMU ID	Core Station	Date Sampled	Planned Sample Elevation (MLLW)	Actual Sample Elevation (MLLW)	Planned Z-Layer Elevation (MLLW)	Actual Bottom of the Core Elevation (MLLW)	Planned Total Core Length with Z-Layer	Actual Total Core Length
DMMU-1 (surface)	SS-1	2/26/18	-32 to -36	-26 to -30	See Below			
	SS-2	2/26/18	-25 to -29	-15 to -19	See Below			
	SS-5	2/27/18	-27 to -31	-24 to -29	See Below			
	SS-6	2/27/18	-0 to -4	-0.6 to -4.6	See Below			
DMMU-2 (surface)	SS-3	2/27/18	-36 to -40	-33 to -37	See Below			
	SS-4	NA	-35 to -39	NA	See Below			
	SS-7	NA	-37 to -41	NA	See Below			
	SS-8	2/26/18	--37 to --41	-35 to -39	See Below			
DMMU-3 (subsurface)	SS-1	2/26/18	-36 to -38	-30 to -34	-38 to -40	-32 to -34	8	8
	SS-2	2/26/18	-29 to -33	-19 to -26	-33 to -35	-24 to -26	10	11
	SS-5	2/27/18	-31 to -45	-29 to -33	-45 to -47	-31 to -33	20	9
	SS-6	2/27/18	-4 to -10	-4.6 to -12	-10 to -12	-10 to -12	12	11
DMMU-4 (subsurface)	SS-3	2/27/18	-40 to -45	NS	-45 to -47	-33 to -37	11	4
	SS-4	NA	-39 to -45	NS	-45 to -47	NS	12	NS
	SS-7	NA	-41 to -45	NS	-45 to -47	NS	10	NS
	SS-8	2/26/18	-41 to -45	-39 to -46	-45 to -47	-44 to -46	10	11

Notes: NS= Not Sampled; NA=Not Applicable; MLLW=Mean Lower Low Water

Table B: Planned versus Actual Compositing Scheme – August 2018

DMMU ID	Core Station	Date Sampled	Planned Sample Elevation (MLLW)	Actual Sample Elevation (MLLW)	Planned Z-Layer Elevation (MLLW)	Actual Bottom of the Core Elevation (MLLW)	Planned Total Core Length with Z-Layer	Actual Total Core Length
DMMU-2A (surface)	SS-3A	8/15/18	-36 to -40	-35 to -39	See Below			
	SS-3B	8/15/18	-36 to -40	-35 to -39	See Below			
	SS-4B	8/15/18	-35 to -39	NS	See Below			
	SS-7	8/15/18	-37 to -41	-36 to -39	See Below	-36 to -39	10	3
DMMU-4A (subsurface)	SS-3A	8/15/18	-40 to -45	-39 to -41	-45 to -47	-41 to -42	11	7
	SS-3B	8/15/18	-36 to -40	-39 to -41	-45 to -47	-41 to -42	11	7
	SS-4B	8/15/18	-39 to -45	-25 to -29	-45 to -47	-29 to -35	12	10
	SS-7	8/15/18	-41 to -45	NS	-45 to -47	NA	10	NA

Notes: NS= Not Sampled; NA=Not Applicable; MLLW=Mean Lower Low Water

6.2 Summary of Sampling Representativeness

6.2.1 Penetration

The data indicates that the sampled sediment does not contain COCs at concentrations exceeding DMMP/SMS criteria. Full penetration (relative to the SAP and SAPA) was not achieved in the majority of the cores. Based on samples that were obtained, it appears that underlying material is not likely to be impacted by historical contamination, as there did not appear to be an increase in contaminant concentrations with depth.

6.2.2 DMMU Composition

DMMUs-1 and -3, representing approximately 53,770 cubic yards of the dredge prism, were characterized fully in accordance with the approved SAP in February 2018.

The surface DMMU-2 was composited from two subsamples, two less than required by the guidance based on the volume during the February 2018 sampling event. The results from DMMU-2 analyses showed no exceedances of SMS or DMMP criteria.

Follow-up sampling and grain size analysis of the surface material in DMMU-2 (sample DMMU-2A, collected in August 2018) show a grain size distribution similar to the grain size distribution in the other DMMUs , although fines were substantially higher than the original samples. There were no exceedances of DMMP/SMS criteria in any of the material characterized from the dredge prism during the February and August 2018 sampling events and, thus, a low potential for the material where DMMU-2A was collected to represent an environmental concern.

The south subsurface DMMU-4, collected in February 2018, only consisted of material from one subsample. Follow-up sampling in August 2018 successfully collected additional subsurface material in DMMU-4 from two subsamples/core locations. That material was composited and submitted for chemical analyses as discussed previously. Together, these analyses were considered sufficiently representative of DMMU-4 material by the DMMP agencies.

6.2.3 Conclusions

Cores were collected from six locations within the proposed dredge prism and composited to create four composite DMMU samples (DMMU-1 through DMMU-4) in February 2018. An additional four cores were collected from three locationsand composited to create two composite DMMU samples (DMMU-2A and DMMU-4A) during follow-up sampling completed at the site in August 2018 (see Section 3.4). The DMMU samples from both sampling events were submitted for chemical analysis of DMMP and SMS COCs (DMMU-2A, collected in August 2018, was only analyzed for total organic carbon by SM5310B/EPA Method 9060 and grain size by PSEP/ASTM D-422 per the DMMO-approved SAPA). The proposed dredged material was sampled and analyzed in accordance with the DMMP-approved project SAP except for the deviations noted in Sections 3.2 and 3.4. Despite issues encountered during sampling, data was deemed sufficient by the DMMO for regulatory decision-making.

COCs either were not detected or were detected at concentrations less than the DMMP and SMS screening levels (SLs) in the sample composites for DMMU-1 through DMMU-4 and DMMU-4A. The data indicate that dredged material from DMMU-1 through DMMU-4 and DMMUs-2A and -4A (Sheet 3) is suitable for open-water placement based on the coordination with the DMMO during sampling and the chemical analytical results summarized in Tables 4 and 5.

7.0 LIMITATIONS

This report has been prepared for BHP and the U.S. Army Corps of Engineers DMMO for their use in evaluating and documenting the suitability of the proposed dredged material for open-water disposal.

This study is based on sampling and analyses conducted in accordance with the guidelines of the DMMP at specific sampling locations. It is possible that sediment quality may vary over time and/or between sampling locations.

Within the limitations of scope, schedule, and budget, our services have been executed in accordance with the generally accepted environmental science practices for dredged material characterization in this area at the time this report was prepared. No warranty or other conditions, express or implied, should be understood.

8.0 BIBLIOGRAPHY

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Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Tables

TABLE 1. SUMMARY OF SAMPLE COORDINATES, ADJUSTED MUDLINE ELEVATIONS AND SAMPLE LENGTHS

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

DMMU ID	Sample ID[1]	Date Sampled	Northing[2]	Easting[2]	Water Depth (feet)[3]	Adjusted Mudline Elevation (ft MLLW)[4]	Sample Length Recovered (feet)
February-18							
1	SS-1	2/26/2018	615440.00	787469.00	26	-25.90	3.92
	SS-2	2/26/2018	615425.00	787743.00	15	-15.00	3.5
	SS-5	2/27/2018	615366.00	788095.00	33	-24.00	4.52
	SS-6	2/27/2018	615431.00	788362.00	5.6	-0.6	3.23
2	SS-3	2/27/2018	615258.00	787616.00	43	-33.00	4.2
	SS-4	Not Applicable					
	SS-7	Not Applicable					
	SS-8	2/26/2018	615266.00	788387.00	45	-35.00	3.67
3	SS-1	2/26/2018	615440.00	787469.00	26	-25.90	3.96
	SS-2	2/26/2018	615425.00	787743.00	15	-15.00	7.75
	SS-5	2/27/2018	615366.00	788095.00	33	-24.00	4.53
	SS-6	2/27/2018	615431.00	788362.00	5.6	-0.6	7.71
4	SS-3	2/27/2018	615258.00	787616.00	43	-33.00	No Recovery
	SS-4	Not Applicable					
	SS-7	Not Applicable					
	SS-8	2/26/2018	615266.00	788387.00	45	-35.00	7.01
August-18							
2A	SS-3A	8/15/2018	615259.136	787616.620	35.1	-35.01	7
	SS-3B	8/15/2018	615253.052	787609.583	35.1	-35.01	8.3
	SS-7	8/15/2018	615249.803	787998.719	46.3	-36.00	2.9
4A	SS-3A	8/15/2018	615259.136	787616.620	35.1	-35.01	7
	SS-3B	8/15/2018	615253.052	787609.583	35.1	-35.01	8.3
	SS-4B	8/15/2018	615309.747	788448.942	30.2	-24.57	10.5

Notes:

[1] See Sheet 3 for sample locations

[2] Northing and easting are based on the North American Datum of 1983 (NAD83) State Plane Coordinate System, Washington South, Survey Feet.

[3] Depth finder on vessel was used to measure water depth.

[4] Adjusted Mudline Elevation = Water Depth + Tidal Stage

TABLE 2. SUMMARY OF GRAIN SIZE DATA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

Description (Sieve Size - microns)	**DMMU-1**	**DMMU-2**	**DMMU-3**	**DMMU-4**	**DMMU-2A**	**DMMU-4A**
Gravel (3" - 3/8")	0.0%	0.2%	0.0%	0.0%	0.0%	0.0%
Coarse Sand (#4 - #10)	0.4%	3.6%	0.1%	0.0%	0.0%	0.1%
Medium Sand (#10 - #40)	6.8%	41.4%	1.6%	2.0%	0.7%	0.8%
Fine Sand (#40 - #100)	21.0%	21.9%	14.4%	22.7%	10.6%	16.7%
Very Fine Sand (#100 - #200)	18.7%	6.1%	21.1%	23.9%	14.2%	31.9%
Coarse Silt (#200 - 32)	24.7%	20.6%	28.5%	21.3%	39.7%	27.0%
Medium Silt (32 - 13)	5.8%	0.6%	9.4%	7.3%	11.9%	5.4%
Fine Silt (13 - 7)	11.0%	3.0%	10.4%	10.8%	9.2%	6.8%
Very Fine Silt (7 - 3.2)	3.9%	1.2%	4.7%	4.2%	4.6%	4.1%
Clay (3.2 - <1.3)	7.7%	1.2%	9.9%	7.8%	9.2%	7.2%
Total Fines (#200)	53.1%	26.6%	62.9%	51.5%	74.5%	50.5%

TABLE 3. SUMMARY OF DMMU VOLUMES

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
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DMMU ID	Core Station	Dredge Depth Elevation + 2' OD (ft MLLW)	Assumed Elevation (ft MLLW)	Approximate Total DMMU Volume (cy)
DMMU 1	SS-1	N/A	-32	22,616
	SS-2	N/A	-25	
	SS-5	N/A	-27	
	SS-6	N/A	0	
DMMU 2/2a	SS-3	N/A	-36	22,675
	SS-4	N/A	-35	
	SS-7	N/A	-37	
	SS-8	N/A	-41	
DMMU 3	SS-1	-40	-36	31,154
	SS-2	-35	-29	
	SS-5	-47	-31	
	SS-6	-12	-4	
DMMU 4/4a	SS-3	-47	-40	31,161
	SS-4	-47	-39	
	SS-7	-47	-41	
	SS-8	-47	-41	
Total				107,606

TABLE 4. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO DMMP CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
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CHEMICAL	**Sample ID**	**DMMU-1**	**DMMU-2**	**DMMU-3**	**DMMU-4**	**DMMU-2A**	**DMMU-4A**	**DMMP Criteria (dry weight)**		
	Sample date	**2/27/2018**	**2/27/2018**	**2/27/2018**	**2/27/2018**	**8/15/2018**	**8/15/2018**	**SL**	**BT**	**ML**
CONVENTIONALS (mg/kg dry weight)										
Ammonia		**128 D**	**187 D**	**209 D**	**163 D**	**---**	**169 D**	---	---	---
Total sulfides		**291.00**	**85.20**	**88.80**	**249.00**	**---**	**66.02**	---	---	---
GENERAL CHEMISTRY (percent)										
Total solids		**64.07**	**63.83**	**65.39**	**60.77**	**66.50**	**64.55**	---	---	---
Total volatile solids		**4.75**	**10.05**	**4.81**	**5.22**	**---**	**4.62**	---	---	---
Total organic carbon		**1.65**	**1.02**	**0.98**	**1.26**	**1.02**	**1.46**	---	---	---
METALS (mg/kg dry weight)										
Antimony		**0.03 J**	**<0.29**	<0.29	<0.33	---	<0.28	150	---	200
Arsenic		**5.89**	**5.70**	**6.30**	**5.69**	---	**5.59**	57	507.1	700
Cadmium		**0.14 J**	**0.17**	**0.13 J**	**0.1 J**	---	**0.11J**	5.1	---	14
Chromium		**32.1**	**29.4**	**30.8**	**30.6**	---	**25.9**	260	---	---
Copper		**37.2**	**42.8**	**38.5**	**46.9**	---	**35.5**	390	---	1,300
Lead		**5.70**	**6.04**	**5.92**	**6.32**	---	**4.72**	450	975	1,200
Mercury		<0.0341	**0.0311**	**0.0316**	**0.0322**	---	**0.0281**	0.41	1.5	2.3
Selenium		**1.36**	**1.13**	**1.20**	**1.42**	---	**2.21**	---	3	---
Silver		**0.18 J**	**0.18 J**	**0.17 J**	**0.18 J**	---	**0.15J**	6.1	---	8.4
Zinc		**82.2**	**70.2**	**62.5**	**73.9**	---	**59.8**	410	---	3,800
PAHs (µg/kg dry weight)										
Naphthalene		**11.1 J**	<19.4	**9.0 J**	**15.2 J**	**---**	**26.9**	2,100	---	2,400
Acenaphthylene		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	560	---	1,300
Acenaphthene		<19.4	<19.4	<19.1	**5.4 J**	**---**	**39.60**	500	---	2,000
Fluorene		<19.4	<19.4	<19.1	**6.2 J**	**---**	**35.9**	540	---	3,600
Phenanthrene		**12.8 J**	**7.3 J**	**12.4 J**	**17.4 J**	**---**	**65.00**	1,500	---	21,000
Anthracene		<19.4	<19.4	<19.1	<19.7	**---**	**7.0J**	960	---	13,000
2-Methylnaphthalene[1]		**7.3 J**	<19.4	**6.3 J**	**7.1 J**	**---**	**22.4**	670	---	1,900
Total LPAH		**23.9**	**7.3**	**21.4**	**44.2**	**---**	**169.4**	5,200	---	29,000
Fluoranthene		**14.2 J**	**8.1 J**	**13.2 J**	**20.9**	**---**	**31.2**	1,700	4,600	30,000
Pyrene		**15.4 J**	**6.8 J**	**11.8 J**	**17.5 J**	**---**	**22.9**	2,600	11,980	16,000
Benzo(a)anthracene		<19.4	<19.4	<19.1	<19.7	**---**	**7.8J**	1,300	---	5,100

TABLE 4. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO DMMP CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
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CHEMICAL	Sample ID	DMMU-1	DMMU-2	DMMU-3	DMMU-4	DMMU-2A	DMMU-4A	DMMP Criteria (dry weight)		
	Sample date	2/27/2018	2/27/2018	2/27/2018	2/27/2018	8/15/2018	8/15/2018	SL	BT	ML
Chrysene		**5.3 J**	**6.4 J**	**5.1 J**	**8.2 J**	**---**	**8.7J**	1,400	---	21,000
Benzofluoranthenes (b, j ,k)		<38.7	<38.9	<38.1	**10.8 J**	**---**	<39.5	3,200	---	9,900
Benzo(a)pyrene		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	1,600	---	3,600
Indeno(1,2,3-c,d)pyrene		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	600	---	4,400
Dibenz(a,h)anthracene		<4.8	<4.9	<4.8	**1.2 J**	**---**	<19.8	230	---	1,900
Benzo(g,h,i)perylene		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	670	---	3,200
Total HPAH		**34.9**	**21.3**	**30.1**	**58.6**	**---**	**40.7**	12,000	---	69,000
CHLORINATED HYDROCARBONS (µg/kg dry weight)										
1,4-Dichlorobenzene		<4.8	<4.9	<4.8	<4.9	---	<4.9	110	---	120
1,2-Dichlorobenzene		<4.8	<4.9	<4.8	<4.9	---	<4.9	35	---	110
1,2,4-Trichlorobenzene		<4.8	<4.9	<4.8	<4.9	---	<4.9	31	---	64
Hexachlorobenzene (HCB)		<0.49	<0.49	<0.49	**1.28**	---	<4.9	22	168	230
PHTHALATES (µg/kg dry weight)										
Dimethyl phthalate		<19.4	<19.4	<19.1	<19.7	---	<19.8	71	---	1,400
Diethyl phthalate		<19.4	<19.4	<19.1	<19.7	---	<19.8	200	---	1,200
Di-n-butyl phthalate		<19.4	<19.4	<19.1	<19.7	---	<19.8	1,400	---	5,100
Butyl benzyl phthalate		<4.8	<4.9	<4.8	<4.9	---	<4.9	63	---	970
Bis(2-ethylhexyl) phthalate		<48.4	<48.6	<47.7	<49.2	---	<49.4	1,300	---	8,300
Di-n-octyl phthalate		<19.4	<19.4	<19.1	<19.7	---	<19.8	6,200	---	6,200
PHENOLS (µg/kg dry weight)										
Phenol		<19.4	<19.4	<19.1	**18.4 J**	**---**	**23.4**	420	---	1,200
2-Methylphenol		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	63	---	77
4-Methylphenol		<19.4	<19.4	<19.1	<19.7	**---**	<19.8	670	---	3,600
2,4-Dimethylphenol		<24.2	<24.3	<23.8	<24.6	**---**	<24.7	29	---	210
Pentachlorophenol		<96.8	<97.2	<95.3	<98.3	**---**	<98.8	400	504	690
MISCELLANEOUS EXTRACTABLES (µg/kg dry weight)										
Benzyl alcohol		<19.4	<19.4	<19.1	<19.7	---	<19.8	57	---	870
Benzoic acid		<194	<194	<191	<197	---	<198	650	---	760
Dibenzofuran		<19.4	<19.4	<19.1	<19.7	---	**31.1**	540	---	1,700
Hexachlorobutadiene		<0.49	<0.49	<0.49	<0.50	---	<0.49	11	---	270
N-Nitrosodiphenylamine		<4.8	<4.9	<4.8	<4.9	---	<4.9	28	---	130

TABLE 4. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO DMMP CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

CHEMICAL	Sample ID	DMMU-1	DMMU-2	DMMU-3	DMMU-4	DMMU-2A	DMMU-4A	DMMP Criteria		
	Sample date	**2/27/2018**	**2/27/2018**	**2/27/2018**	**2/27/2018**	**8/15/2018**	**8/15/2018**	**SL**	**BT**	**ML**
PESTICIDES & PCBs (µg/kg dry weight)										
4,4’-DDD		<0.97	<0.97	<0.99	<1.00	---	<0.98	16	---	---
4,4’-DDE		<0.97	<0.97	<0.99	<1.00	---	<0.98	9	---	---
4,4’-DDT		<0.97	<0.97	<0.99	<0.50	---	<0.98	12	---	---
sum of 4,4’-DDD, 4,4’-DDE and 4,4’-DDT		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	50	69
Aldrin		<0.49	<0.49	<0.49	<0.50	---	<0.49	9.5	---	---
Total Chlordane		<0.97	<0.97	<0.99	<1.00	---	<0.99	2.8	37	---
cis-chlordane		<0.49	<0.49	<0.49	<0.50	---	<0.49			
trans-chlordane		<0.49	<0.49	<0.49	<0.50	---	<0.49			
cis-nonachlor		<0.97	<0.97	<0.99	<1.00	---	<0.98			
trans-nonachlor		<0.97	<0.97	<0.99	<1.00	---	<0.98			
oxychlordane		<0.97	<0.97	<0.99	<1.00	---	<0.98			
Dieldrin		<0.97	<0.97	<0.99	<1.00	---	<0.98	1.9	---	1,700
Heptachlor		<0.49	<0.49	<0.49	<0.50	---	<0.49	1.5	---	270
Total PCBs Aroclors (Sum of: 1016, 1221, 1242, 1248, 1254, 1260, 1268)		<3.9	<3.9	<3.9	<4.0	---	<3.8	130	--	3,100
Total PCBs (mg/kg OC)								--	38[2]	--
ORGANOMETALLIC COMPOUNDS										
Tributyltin ion (bulk, ug/kg)		<3.73	<3.75	---	---	---	<3.66	---	73	---
DIOXINS/FURANS (ng/kg)										
2,3,7,8-TCDD		**0.78 J**	**0.54 J**	**0.767 J**	**1.22**	**---**	**0.83 J**	5	---	---
Total TEQ of dioxins/furans[3]		**2.81**	**1.48**	**2.61**	**4.52**	**---**	**2.42**	15	---	---

TABLE 4. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO DMMP CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

Notes:

DMMP = Dredged Material Management Program (User's Manual, August 2016)

Total LPAH = The sum of acenaphthylene, acenaphthene, anthracene, fluorene, naphthalene and phenanthrene.

Total HPAH = The sum of benzo(a)anthracene, benzo(a)pyrene, total benzofluoranthenes, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, indeno(1,2,3,-c,d)pyrene and pyrene.

Total benzofluoranthenes = the sum of the "b," "j" and "k" isomers. The "j" isomer co-elutes with the "k" isomer, thus the concentration of the "j" isomer is included in the "k" isomer concentration.

[1] 2-Methylnaphthalene is not included in the summation for total LPAH.

[2] This value is normalized to total organic carbon, and is expressed in mg/kg organic carbon.

SL = Screening Level

BT = Bioaccumulation Trigger

ML = Maximum Level

LPAH = low molecular weight polynuclear aromatic hydrocarbon compounds

HPAH = high molecular weight polynuclear aromatic hydrocarbon compounds

D = The reported value is from a dilution

J = Estimated concentration when the value is less than ARI's established reporting limits

--- = not analyzed

0.46 Indicates an exceedance of DMMP SL Criteria

0.46 Indicates an exceedance of DMMP BT Criteria

0.46 Indicates an exceedance of DMMP ML Criteria

<0.94 = the target analyte was not detected at the reported concentration

2,3,7,8-TCDD - Screening level based on *Dredged Material Evaluation and Disposal Proceedures Users Manual* Guidelines for Dioxin Evaluation in Grays Harbor (Section 8.3.3) Dredged Material Management Office, U.S. Army Corps of Engineers, Seattle District

Total TEQ of dioxins/furans Screening level based on Dredged Material Evaluation and Disposal Proceedures Users Manual Guidelines for Dioxin Evaluation in Grays Harbor (Section 8.3.3) Dredged Material Management Office, U.S. Army Corps of Engineers, Seattle District

TABLE 5. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO SMS CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

CHEMICAL	**Sample ID**	**DMMU-1**	**DMMU-2**	**DMMU-3**	**DMMU-4**	**DMMU-2A**	**DMMU-4A**	**SMS Criteria**	
	Sample date	**2/27/2018**	**2/27/2018**	**2/27/2018**	**2/27/2018**	**8/15/2018**	**8/15/2018**	**SQS**	**CSL**
CONVENTIONALS (mg/kg dry weight)									
Ammonia		**128 D**	**187 D**	**209 D**	**163 D**	**---**	**169 D**	---	---
Total sulfides		**291.00**	**85.2**	**88.8**	**249**	**---**	**66.02**	---	---
GENERAL CHEMISTRY (percent)									
Total solids		**64.07**	**63.83**	**65.39**	**59.40**	**66.50**	**64.55**	---	---
Total volatile solids		**4.75**	**10.05**	**4.81**	**5.22**	**---**	**4.62**	---	---
Total organic carbon		**1.65**	**1.02**	**0.98**	**1.26**	**1.02**	**1.46**	---	---
METALS (mg/kg dry weight)									
Antimony		**0.03 J**	<0.29	<0.29	<0.33	---	<0.03	---	---
Arsenic		**5.89**	**5.70**	**6.30**	**5.69**	---	**5.59**	57	93
Cadmium		**0.14 J**	**0.17**	**0.13 J**	**0.1 J**	---	**0.11J**	5.1	6.7
Chromium		**32.1**	**29.4**	**30.8**	**30.6**	---	**25.9**	260	270
Copper		**37.2**	**42.8**	**38.5**	**47**	---	**35.5**	390	390
Lead		**5.70**	**6.04**	**6**	**6.32**	---	**4.72**	450	530
Mercury		<0.0341	**0.0311**	**0.32**	**0.03**	---	**0.0281**	0.41	0.59
Selenium		**1.36**	**1.13**	**1.20**	**1.42**	---	**2.21**	---	---
Silver		**0.18**	**0.18 J**	**0.17 J**	**0.18 J**	---	**0.15J**	6.1	6.1
Zinc		**82**	**70.2**	**62.5**	**73.9**	---	**59.8**	410	960
PAHs (mg/kg Organic Carbon)									
Naphthalene		**0.7**	<1.9	**0.9**	**1.2**	**---**	**1.8**	99	170
Acenaphthylene		<1.2	<1.9	<1.9	<1.6	**---**	0.3	66	66
Acenaphthene		<1.2	<1.9	<1.9	**0.4**	**---**	**2.7**	16	57
Fluorene		<1.2	<1.9	<1.9	**0.5**	**---**	**2.5**	23	79
Phenanthrene		**0.8**	**0.7**	**7.4**	**1.4**	**---**	**4.5**	100	480
Anthracene		<1.2	<1.9	<1.9	<1.6	**---**	**0.5**	220	1,200
2-Methylnaphthalene[1]		**0.4**	<1.9	**0.6**	**0.0**	**---**	**1.5**	38	64
Total LPAH		**1.4**	**0.7**	**8.3**	**3.5**	**---**	**11.6**	370	780
Fluoranthene		**0.9**	**0.8**	**1.3**	**1.7**	**---**	**2.1**	160	1,200
Pyrene		**0.9**	**0.7**	**1.2**	**1.4**	**---**	**1.6**	1,000	1,400
Benzo(a)anthracene		<1.2	<1.9	<1.9	<1.6	**---**	**0.5**	110	270
Chrysene		**0.3**	**0.6**	**0.5**	**0.7**	**---**	**0.6**	110	460
Benzofluoranthenes (b, j ,k)		<2.3	<3.8	<3.9	**0.9**	**---**	<0.7	230	450
Benzo(a)pyrene		<1.2	<1.9	<1.9	<1.6	**---**	<0.4	99	210
Indeno(1,2,3-c,d)pyrene		<1.2	<1.9	<1.9	<1.6	**---**	<0.4	34	88
Dibenz(a,h)anthracene		<0.3	<0.5	<0.5	**0.10**	**---**	<0.1	12	33
Benzo(g,h,i)perylene		<1.2	<1.9	<1.9	<1.6	**---**	<0.4	31	78
Total HPAH		**2.1**	**2.1**	**3.1**	**4.7**	**---**	**2.8**	960	5,300
CHLORINATED HYDROCARBONS (mg/kg Organic Carbon)									
1,4-Dichlorobenzene		<0.3	<0.5	<0.5	<0.4	---	<0.0	3.1	9
1,2-Dichlorobenzene		<0.3	<0.5	<0.5	<0.4	---	<0.0	2.3	2.3
1,2,4-Trichlorobenzene		<0.3	<0.5	<4.9	<0.4	---	<0.0	0.81	1.8
Hexachlorobenzene (HCB)		<0.0	<0.0	<0.1	**0.1**	---	<0.0	0.38	2.3

TABLE 5. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO SMS CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

	Sample ID	**DMMU-1**	**DMMU-2**	**DMMU-3**	**DMMU-4**	**DMMU-2A**	**DMMU-4A**	**SMS Criteria**	
CHEMICAL	**Sample date**	**2/27/2018**	**2/27/2018**	**2/27/2018**	**2/27/2018**	**8/15/2018**	**8/15/2018**	**SQS**	**CSL**
PHTHALATES (mg/kg Organic Carbon)									
Dimethyl phthalate		<1.2	<1.9	<1.9	<1.6	---	<0.4	53	53
Diethyl phthalate		<1.2	<1.9	<1.9	<1.6	---	<1.2	61	110
Di-n-butyl phthalate		<1.2	<1.9	<1.9	<1.6	---	<0.4	220	1,700
Butyl benzyl phthalate		<0.3	<0.5	<0.5	<0.4	---	<0.0	4.9	64
Bis(2-ethylhexyl) phthalate		<2.9	<4.8	<4.9	<3.9	---	<2.0	47	78
Di-n-octyl phthalate		<1.2	<1.9	<1.9	<1.6	---	<0.6	58	4,500
PHENOLS (µg/kg dry weight)									
Phenol		<19.4	<19.4	<19.1	**18.4 J**	---	**23.4**	420	1,200
2-Methylphenol		<19.4	<19.4	<19.1	<19.7	---	<7.7	63	63
4-Methylphenol		<19.4	<19.4	<19.1	<19.7	---	<14.5	670	670
2,4-Dimethylphenol		<24.2	<24.3	<23.8	<24.6	---	<2.1	29	29
Pentachlorophenol		<96.8	<97.2	<95.3	<98.3	---	<30.9	360	690
MISCELLANEOUS EXTRACTABLES (µg/kg dry weight)									
Benzyl alcohol		<19.4	<19.4	<19.1	<19.7	---	<14.7	57	73
Benzoic acid		<194	<194	<191	<197	---	<58.45	650	650
Dibenzofuran (mg/kg Organic Carbon)		<1.2	<1.90	<1.95	<1.56	---	**2.1**	15^{2}	58^{2}
Hexachlorobutadiene (mg/kg Organic Carbon)		<0.03	<0.05	<0.05	<0.04	---	<0.05	3.9^{2}	6.2^{2}
N-Nitrosodiphenylamine (mg/kg Organic Carbon)		<0.3	<0.48	<0.49	<0.39	---	<0.09	11^{2}	11^{2}
PESTICIDES & PCBs (µg/kg dry weight)									
4,4'-DDD		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
4,4'-DDE		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
4,4'-DDT		<0.97	<0.97	<0.99	<0.50	---	<0.98	---	---
sum of 4,4'-DDD, 4,4'-DDE and 4,4'-DDT		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
Aldrin		<0.49	<0.49	<0.49	<0.50	---	<0.49	---	---
Total Chlordane		<0.97	<0.97	<0.99	<1.00	---	<0.99	---	---
cis-chlordane		<0.49	<0.49	<0.49	<0.50	---	<0.49	---	---
trans-chlordane		<0.49	<0.49	<0.49	<0.50	---	<0.49	---	---
cis-nonachlor		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
trans-nonachlor		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
oxychlordane		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
Dieldrin		<0.97	<0.97	<0.99	<1.00	---	<0.98	---	---
Heptachlor		<0.49	<0.49	<0.49	<0.50	---	<0.49	---	---
PCBs (mg/kg Organic Carbon)									
Total PCBs Aroclors (Sum of: 1016, 1221, 1242, 1248, 1254, 1260, 1268)		<0	<0.38	<0.40	<0.32	---	<0.26	12	65

TABLE 5. SUMMARY OF CHEMICAL ANALYTICAL RESULTS COMPARED TO SMS CRITERIA

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
REVISED DREDGED MATERIAL CHARACTERIZATION REPORT
HOQUIAM, WASHINGTON

Notes:

SMS = Sediment Management Standards (December 2017)

Total LPAH = The sum of acenaphthylene, acenaphthene, anthracene, fluorene, naphthalene and phenanthrene.

Total HPAH = The sum of benzo(a)anthracene, benzo(a)pyrene, total benzofluoranthenes, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, indeno(1,2,3,-c,d)pyrene and pyrene.

Total benzofluoranthenes = the sum of the "b," "j" and "k" isomers. The "j" isomer co-elutes with the "k" isomer, thus the concentration of the "j" isomer is included in the "k" isomer concentration.

[1] 2-Methylnaphthalene is not included in the summation for total LPAH.

[2] This value is normalized to total organic carbon, and is expressed in mg/kg organic carbon.

SQS = Sediment Quality Standards

CSL = Cleanup Screening Levels

LPAH = low molecular weight polynuclear aromatic hydrocarbon compounds

HPAH = high molecular weight polynuclear aromatic hydrocarbon compounds

TOC = Total organic carbon

D = The reported value is from a dilution

J = Estimated concentration when the value is less than ARI's established reporting limits

--- = not analyzed

245.2	indicates an exceedance of SMS SQS criteria
180.6	indicates an exceedance of SMS CSL criteria

<0.37 = the target analyte was not detected at the reported concentration

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Figures

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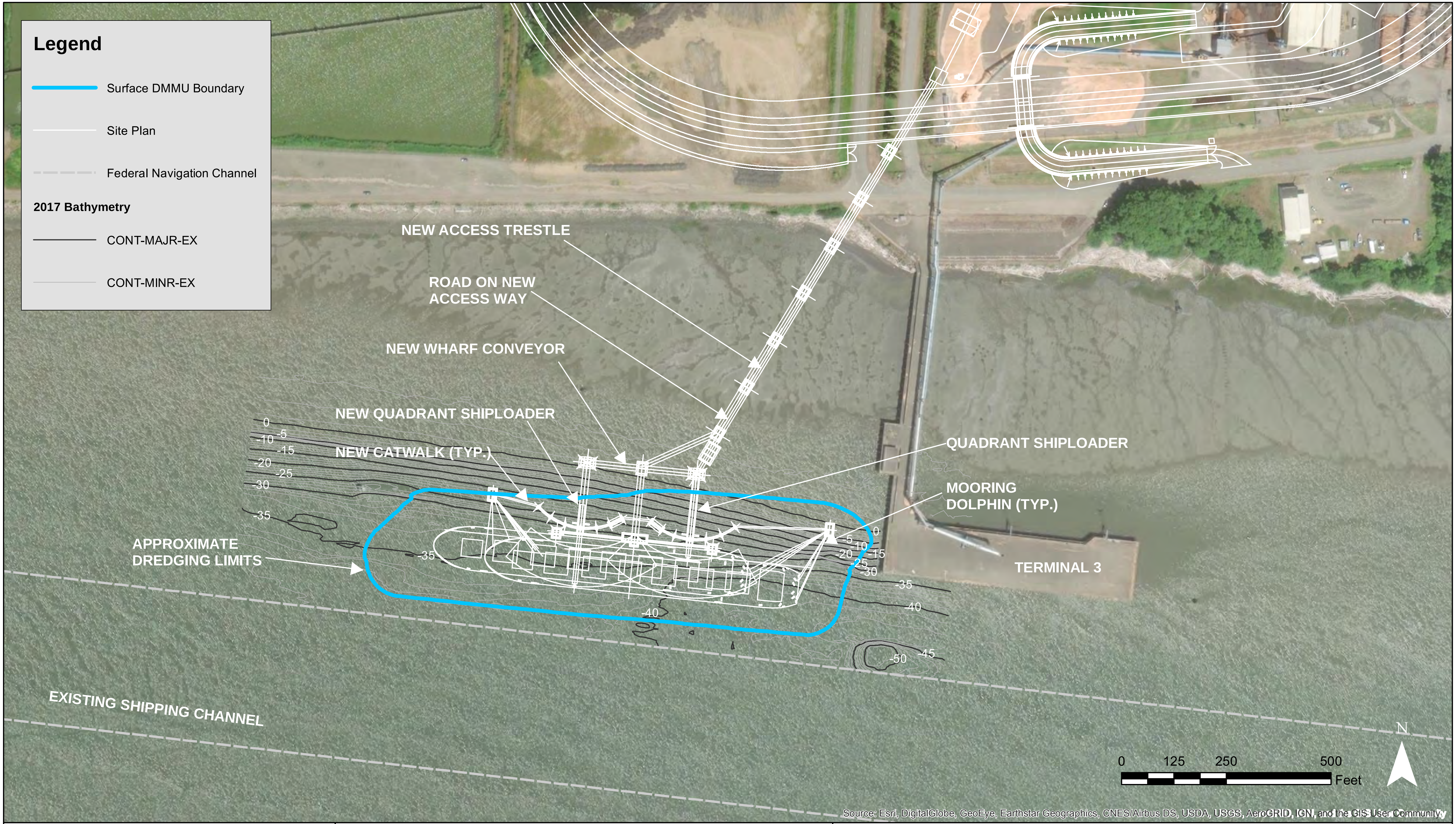
Proposed Grays Harbor Potash Export Facility-
Revised Dredged Material Characterization Report

Sheet 1- Vicinity Map and Project Site Location
September 2018



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Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 2- Proposed Project Features
September 2018

General Notes

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM; WASHINGTON STATE PLANE SOUTH (WSPS), NAV83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING 2017.

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Vancouver, Washington 98660-3231
(360) 823-6100 FAX: (360) 823-6101

Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 3 -Proposed Dredge Prism and Sampling Locations

September 2018

1" = 120'
60 0 60 120
scale feet

GENERAL NOTES:
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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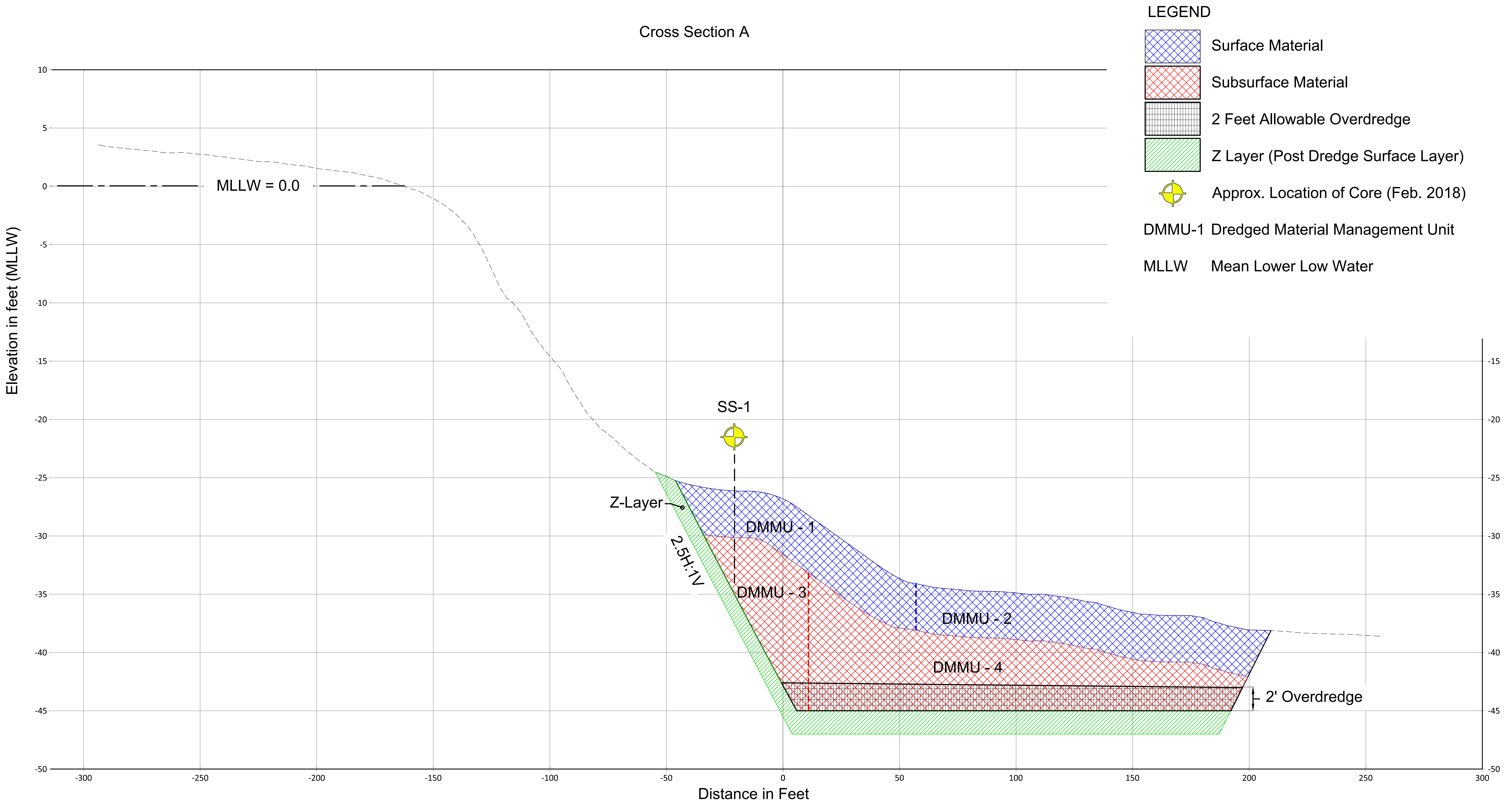

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Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 4 - Cross Section A: STA 3+00

September 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet
Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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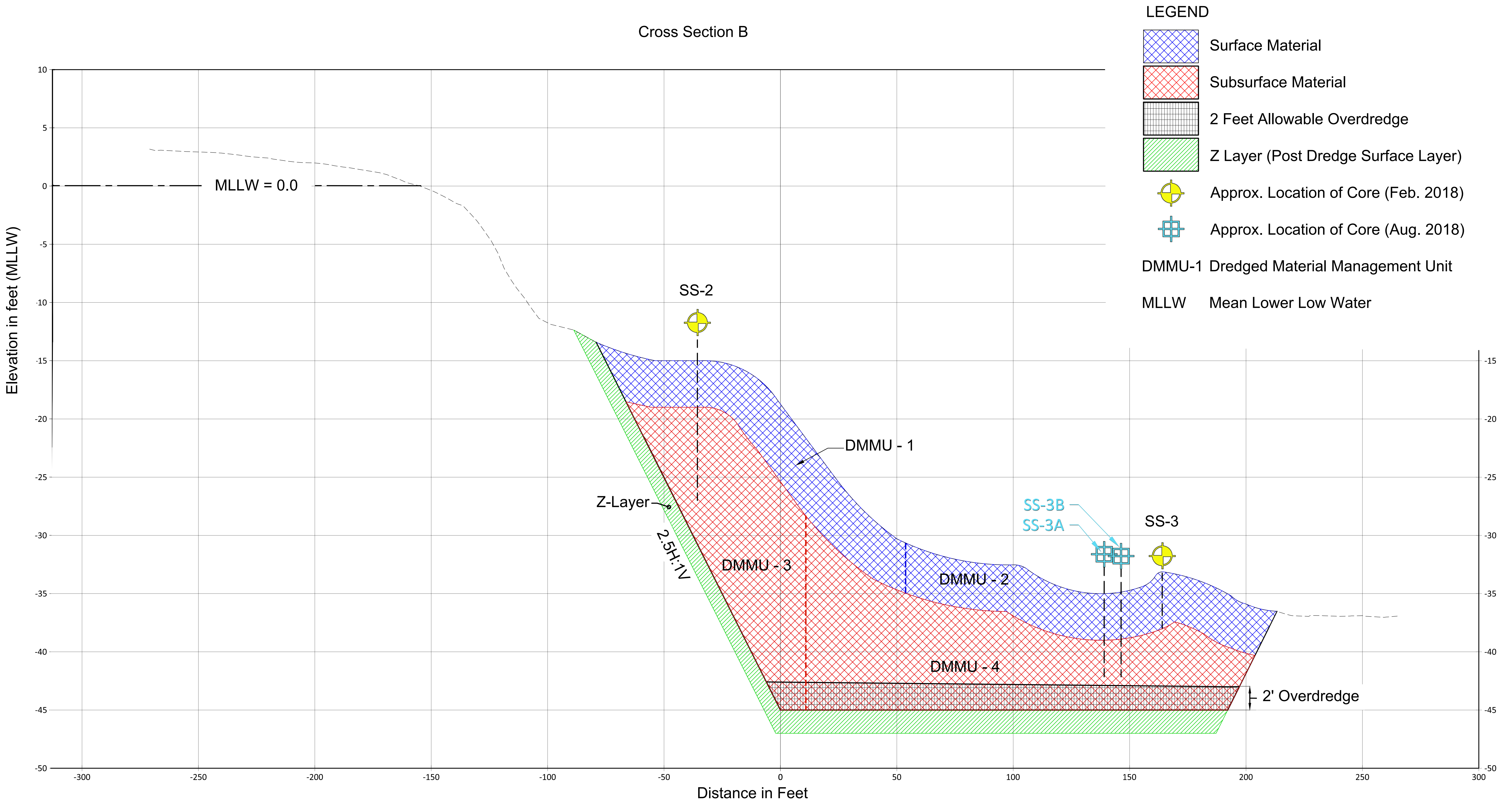

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Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 5 - Cross Section B: STA 5+00

September 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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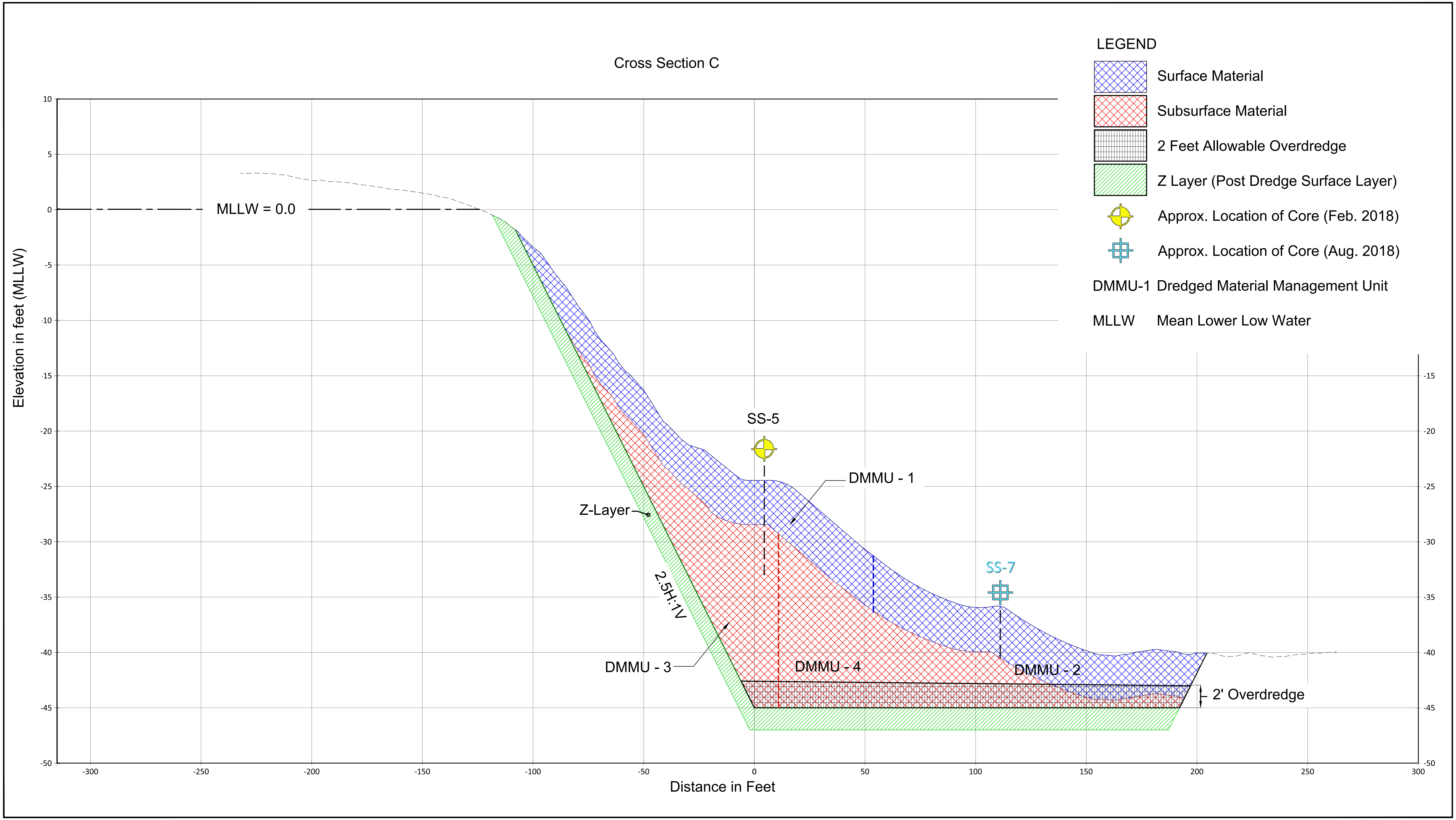

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Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 6 - Cross Section C: STA 9+00

September 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet
Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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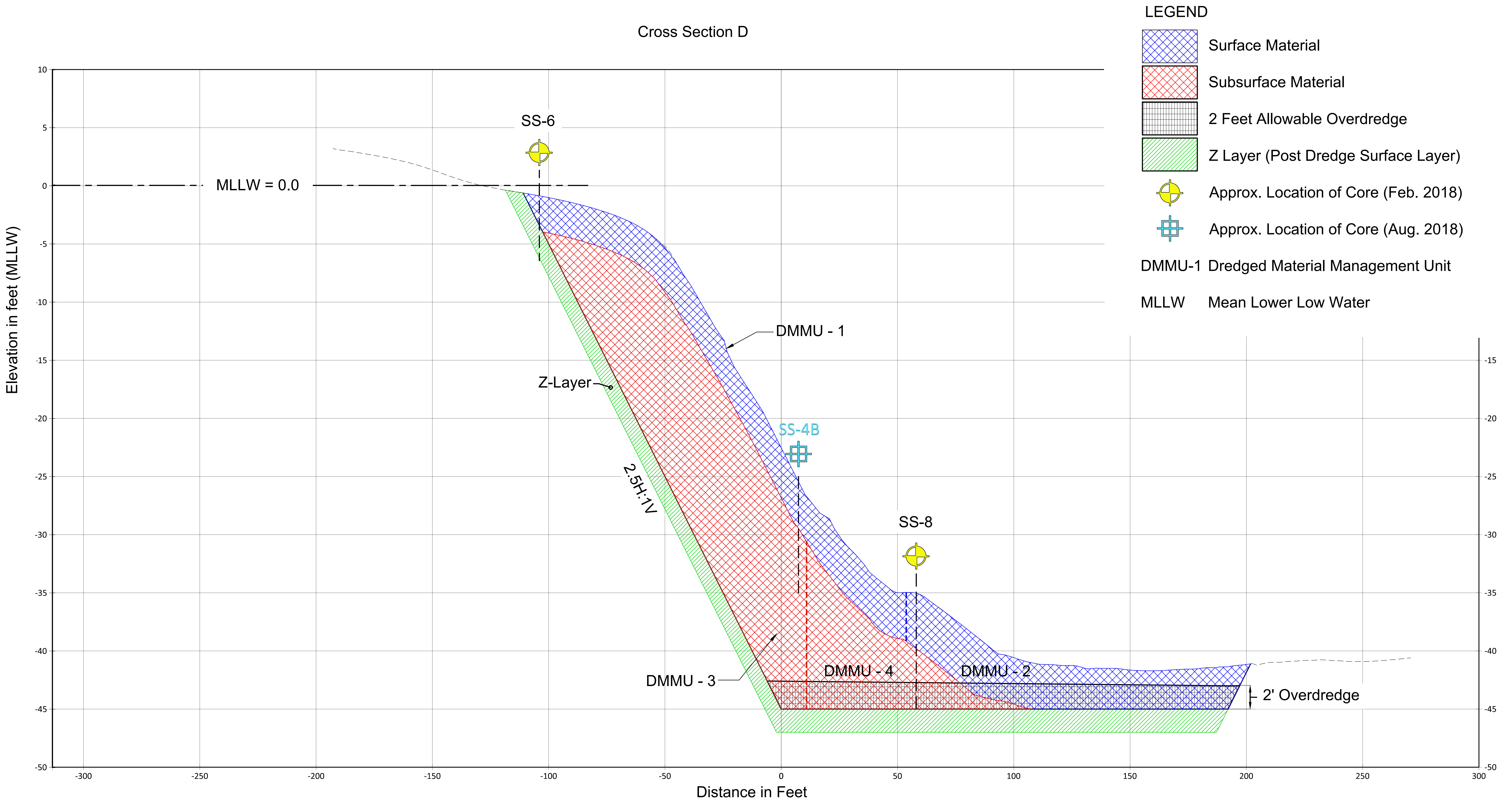
Cross Section D

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Proposed Grays Harbor Potash Export Facility -
Revised Dredged Material Characterization Report

Sheet 7 - Cross Section D: STA 12+00

September 2018

Horizontal Scale 1" = 40' (20 0 20 40 feet)
Vertical Scale 1" = 8' (4 0 4 8 feet)

GENERAL NOTES:
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix A

Sampling and Analysis Plan and DMMO Approval Email

Sampling and Analysis Plan

BHP Billiton Canada, Inc.
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

Prepared for

Ms. Valerie Bond
BHP Billiton Canada, Inc.
130 Third Avenue South
Saskatoon, SK S7H 1L3
Canada

Attention: Dredged Material Management Office (DMMO)

11 October 2017, revised 22 February 2018

Prepared by

BergerABAM
210 East 13th Street, Suite 300
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Job No. A17.0202.00

Sally Fisher

Sally L. Fisher
Senior Project Manager

Victoria R. England, LG
Senior Environmental Scientist

SAMPLING AND ANALYSIS PLAN

BHP Billiton Canada, Inc.
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

TABLE OF CONTENTS

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ACRONYMS AND ABBREVIATIONS

ARI	Analytical Resources, Inc.
ASTM	American Society for Testing and Materials
BHP	BHP Billiton Canada, Inc.
CFR	Code of Federal Regulations
COCs	contaminants of concern
cy	cubic yard(s)
DMMO	Dredged Material Management Office
DMMP	Dredged Material Management Program
DMMU	Dredged Material Management Unit
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management
xxH:xxV	Horizontal to Vertical
kg	kilograms
mg	milligrams
MLLW	mean lower low water
PCBs	polychlorinated biphenyls
PCDD/F	Polychlorinated Dibenzodioxins/Furans
Port	Port of Grays Harbor
PSDDA	Puget Sound Dredged Disposal Analysis
PSEP	Puget Sound Estuary Program
QA/QC	quality assurance/quality control
SAP	Sampling and Analysis Plan
SL	screening level
SMS	Washington State Sediment Management Standards
SMARM	Sediment Management Annual Review Meeting
SQS	Washington State Sediment Quality Standards
SVOCs	semi-volatile organic compounds
USACE	U.S. Army Corps of Engineers
VOCs	volatile organic compounds
WAC	Washington Administrative Code

SAMPLING AND ANALYSIS PLAN
PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
DREDGED MATERIAL CHARACTERIZATION

1.0 INTRODUCTION

The Port of Grays Harbor (Port) Terminal 3 is located on the north shore of Grays Harbor in Hoquiam, Washington (see Figure 1). Terminal 3 has a deep-water berth and a dock supported by concrete and steel piling that is accessible by a single approach. The dock was built in the early 1980s and has been used only for shipping wood and wood products. BHP Billiton Canada, Inc. (BHP) is proposing to construct a new potash export facility (also referred to as the "project site") west of and adjacent to the Port's Terminal 3. The proposed facility includes the construction of a new marine berth and support facilities.

Approximately 110,000 cubic yards of dredging is needed to accommodate a new dual quadrant shiploader and to provide sufficient draft for the vessels that will serve the facility. This Sampling and Analysis Plan (SAP) provides the methods for characterizing the area to be dredged. This SAP describes the site history, potential sources of contaminants, existing data, the proposed project, and associated sampling and analysis of the proposed dredged material.

The analytical results will be used to evaluate the potential suitability of dredged material for in-water disposal or beneficial use, and/or upland placement in accordance with Dredged Material Management Program (DMMP), the Washington State Department of Ecology (Ecology) Sediment Management Standards (SMS) protocols, and Solid Waste Handling Standards.

This SAP is provided to the Dredged Material Management Office (DMMO) for review and approval of the proposed sampling and analysis program for characterizing project sediments and completing both a Suitability Determination for unconfined, open-water disposal or placement, and review of the post-dredge surface.

2.0 PROJECT DESCRIPTION

The proposed project will be located west of and adjacent to the existing Terminal 3 dock as shown on Figure 2. A new berth will be constructed as part of the proposed facility to support a dual-quadrant ship loader capable of servicing bulk material export vessels (Panamax class). Structures to support the loading equipment and moor the shipping vessels will also be constructed. The berth will align with, and overlap, the dredging footprint for the existing Port's Terminal 3 dock and extend to the navigation channel. The face of the new structure will align with the face of the existing dock to minimize dredging and accommodate ship berthing and loading.

2.1 Proposed Dredging

The Port's Terminal 3 berth is currently authorized to a depth of -41 feet mean lower low water (MLLW) with 2 feet of overdredge allowance. The proposed depth for the new potash export facility berth is -43 feet MLLW plus 2 feet of allowable overdredge, to -45 feet MLLW.

The new berth will extend from the edge of the navigation channel to the face of the proposed shiploader trestle. The footprint of the new berth will be approximately 4.72 acres and the footprint of the entire dredge area is approximately 7.49 acres. The total volume to be dredged is approximately 110,000 cubic yards.

Existing bathymetry in the proposed dredge area ranges from -1 to -41 feet MLLW. The dredge prism cut will range in thickness from approximately 3 to 23 feet below mudline (including overdredge allowance—see Figures 5 through 9). The number of dredged material management units (DMMUs) and associated subsamples is based upon the DMMP ranking of low-moderate as described in Section 6.2.2. The boundaries of each DMMU in accordance with DMMP criteria are based on existing bathymetry, volume, and proposed dredge depth.

3.0 EXISTING CONDITIONS

Terminal 3 and the proposed project features are shown on Figure 2. The Port's Terminal 3 and dock were constructed in the early 1980s and used since then for shipping wood and wood products. The adjacent upland property is currently occupied by a wood chip facility (Willis Enterprises) and a lumber facility (Dahlstrom Lumber).

A former wastewater lagoon operated by the city of Hoquiam is located adjacent to the shoreline immediately north of the project site. The lagoon has been cleaned of sludge and is currently partially filled with rainwater. The eastern portion of the lagoon is partially filled with soil excavated from various projects in the area, as well as material generated from landslide cleanup and repair from local sites. The U.S. Army Corps of Engineers (USACE) recently placed unsuitable dredged material from the navigation channel in the lagoon, and they used the Terminal 3 dock as the transloading facility.[1] The filling is permitted and controlled by the City.

The existing Terminal 3 berth has been routinely dredged to remove accumulated material and to maintain the needed draft in the berth. While most of the area of the proposed berth has never been dredged and consists of subtidal marine sediment, a portion of the footprint overlaps with the existing Terminal 3 berth. The Terminal 3 berth was last dredged in 2013 to its authorized depth of -41 feet MLLW plus 2 feet of overdredge.

---

[1] According to Port personnel, no spills occurred during the transloading process.

The proposed berth is shown on Figure 3 with an overlay of the existing Terminal 3 berth boundary. Cross sections of the proposed dredge area are shown on Figures 5 through 9.

3.1 Permit Status

The Port has current federal, state, and local permits to conduct maintenance dredging at Terminal 3 that are valid until 2019 (USACE Permit No. NWS-2008-997). The Terminal 3 berth is characterized to -41 feet plus 2 feet of overdredge allowance. The recency for Terminal 3 expires in December 2021.

3.2 New Zealand Mud Snail

Grays Harbor is not documented as an area known or suspected of harboring the New Zealand mud snail.[2] Equipment brought to Grays Harbor for this sampling event will be washed/prepped to ensure snails will not be transported to Grays Harbor.

4.0 POTENTIAL SOURCES OF CONTAMINATION

4.1 Regulatory Database Search

We reviewed reports and data from Ecology’s Environmental Information Management (EIM) database (Ecology EIM, accessed 6 June 2017) regarding historical investigations at the terminals and on nearby upland properties that may pose an environmental concern to the project site. The completed EIM database review focused on sites with historical environmental concerns within approximately 1/2 mile of the project site.

Historical cleanup sites and former landfill facilities (Apex Environmental, Lamb Grays Harbor Company, and Adams Street Inert Waste Disposal Site) are approximately 1/2 mile upland from the project site (see Figure 1). There are no recorded releases on file associated with the landfill sites, and Ecology’s records indicate that cleanups were completed at all of the sites identified above except Lamb Grays Harbor Company and Apex Environmental. These three sites are approximately 1/2 mile or more away from and upland from the proposed project site berth and are not likely to have impacted the proposed dredged material in the project area.

5.0 AVAILABLE SITE CHARACTERIZATION DATA REVIEW

The DMMP has issued various suitability determinations for material dredged for maintenance and/or deepening at the adjacent Terminal 3 berth from 2008 through 2015. All of the suitability determinations issued for Terminal 3 determined all of the characterized material to be suitable for open-water disposal.

---

[2] Per Ecology’s Invasive Species website: http://www.ecy.wa.gov/programs/eap/Invasivespecies/AIS-publicversion.html and the map located at https://nas.er.usgs.gov/viewer/omap.aspx?SpeciesID=1008, visited on 6 June 2017.

Sediment characterization has not been previously completed within the footprint of the project site except for the western portion of the Terminal 3 berth that overlays the proposed new berth.

6.0 PROGRAM OBJECTIVES AND APPROACH

6.1 Objectives

The objectives of this SAP are to present procedures and methods that will be used to

- Characterize sediment from the existing surface to the proposed authorized depth (to -45 feet MLLW [-43 feet MLLW with 2-foot overdredge allowance]) for the new berth.
- Characterize sediment from the depth representing the surface that will be exposed after dredging ("Z-layer").
- Evaluate suitability of the material for open-water disposal at the Washington Department of Natural Resources managed South Jetty and Point Chehalis disposal sites relative to DMMP and SMS criteria (Washington Administrative Code [WAC] 173-204).
- Evaluate upland disposal options (if the dredged material is not suitable for open-water disposal).

6.2 General Approach

The general approach used to develop this SAP is based on the following.

- Sampling frequency is based on a low-moderate ranking, based on existing site conditions and historical site use.[3]
- The proposed depth for the new berth is -43 feet MLLW with a 2-foot overdredge allowance.
- The characterization will be completed using MudMole sampling equipment. The sampling and analysis will be performed under an in-water work window (2017 to 2018) extension with the approval of WDFW and following submittal and approval of an appropriate SAP to the DMMP.

6.2.1 Site Ranking

The DMMP (DMMO, 2016a) defines site ranking as follows.

- "Low" ranking where there are "few or no sources of chemicals of concern. Data are available to verify low chemical concentrations (below DMMP screening levels) and no significant response in biological tests."
- "Low-moderate" ranking is used where "available data indicate a low rank may be warranted, but there are insufficient data to confirm the ranking."

---

[3] Recency guidelines allow characterization data to be valid for low-moderate ranked sites for six years.

- "Moderate" ranking is used at those sites where "sources exist in the vicinity of the project, or there are present or historical uses of the project site, with the potential for producing chemical concentrations within a range associated historically with some potential for causing adverse biological impacts."
- "High" ranking is used at those sites where there are "many known chemical sources, high concentrations of chemicals of concern, and/or biological testing failures in one or both of the two most recent cycles of testing."

6.2.2 Terminal 3 Sediment

The dredged material in the project area generally consists of sandy silt with clay, based on review of previous characterizations at the existing Terminal 3 berth. The dredging volume estimated for this study is based on current bathymetry (December 2017).

The dredging volume history for the adjacent Terminal 3 berth is shown below.

Historical Dredging Volumes

Dredging Year	Terminal 3 (cubic yards)
December 2001	21,784
January 2011	32,367
January 2012	65,827
January 2013 to depth of -43 MLLW	44,000
Total	163,978
Average	40,995

The Terminal 3 berth has a low ranking based on the Port's previous completed studies and the DMMP's determination. It is likely the adjacent proposed project area will also have a low risk for the presence of contaminated sediment. We have assumed a "low-moderate" ranking for this characterization because there are no data to support a "low" ranking at this time.

7.0 SAMPLE COLLECTION AND HANDLING PROCEDURES

7.1 General

Dredge volume, dredge prism configuration, and sampling frequency are based on typical cross sections and conditions within the project area. The dredge prism for this SAP is based on the following assumptions.

- The existing top of mudline ranges from approximately Elevation + 3 to -41 feet MLLW.
- The design dredging depth will be Elevation –43 feet MLLW with 2 feet of allowable overdredge depth.
- The new slope face will have a 2.5H:1V slope from the top of bank to the design depth.

- The surface material (mudline to 4 feet) and the subsurface material (below 4 feet) in the dredge prism will be ranked low-moderate.

7.2 General Sampling Scheme

The sampling and analyses frequency for DMMP characterization for this project has been determined in accordance with the proposed site ranking of “low-moderate” as discussed above. We expect that this frequency will also be sufficient for evaluating its suitability for beneficial use, compliance with the SMS (WAC 173-204), and/or other disposal options.

The dredge prism depths range from 1 foot to approximately 23 feet (including the 2 feet of allowable overdredge) depending upon the sample location. The potential dredge area has been delineated into two surface DMMUs and two subsurface DMMUs as shown on Figure 3.

Sampling and analysis for this project will be performed in accordance with DMMP/Puget Sound Estuary Program (PSEP) protocols. Samples will be collected using MudMole equipment. Cores will be processed and samples collected either as soon as the core is extruded on the boat/barge or onshore at the end of each sampling day.

Samples will be collected to 2 feet deeper than the proposed dredge depth (including 2 feet of overdredge) at each boring location in order to collect Z-layer samples representing the newly exposed, post-dredge surface conditions. The proposed sample stations will be located approximately as shown on Figure 3.

Samples will be composited from the stations to create samples representative of each DMMU as described in Section 7.3. Each DMMU sample will be analyzed for DMMP/SMS conventional parameters, the full suite of marine DMMP/SMS contaminants of concern (COCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), polychlorinated dibenzodioxins/furans (PCDD/F), pesticides and bulk tributyltin, as shown on Table 2. Sediment reference material will also be submitted for analysis of PCBs and PCDD/F in accordance with DMMP guidance.

7.3 Compositing Scheme

7.3.1 General

Samples will be collected from the subunits in each station and composited to represent DMMUs. Details of the compositing scheme, including depth, subunits, and DMMU volumes, are shown on Table 1. The anticipated compositing schemes are shown schematically on Figures 5 through 9. DMMU samples will be collected and archived for bioassays (see Section 8.6). Subunit samples will be collected and archived at 2-foot intervals for potential future chemical analysis, if needed.

7.3.2 Surface and Subsurface Units

DMMP requirements for sampling and analysis of surface sediment for a low-moderate ranked site consist of one sample per 8,000 cubic yards and one analysis per 32,000 cubic yards. DMMP requirements for sampling and analysis of subsurface sediment for a low-moderate ranked site consist of one sample per 8,000 cubic yards and one analysis per 48,000 cubic yards.

Subunit samples representing approximately 5,804 to 7,942 cubic yards of material at each location will be collected. The subunit samples (e.g., sediment sample [SS] SS-1A, SS-2A, etc.) will be composited to represent the DMMU samples (DMMU-1, DMMU-2, DMMU-3, and DMMU-4) as summarized in Table 1.

7.3.3 Post-dredge Surface Z-layer Samples

Post-dredge surface sediment samples will be collected from depths representing the newly exposed sediment surface from all eight sample stations (representing 2 feet of post-dredge sediment surface) if possible and as shown in Table 1.

The Z-layer samples will be archived pending results from the overlying DMMU samples. The Z-layer samples will be analyzed for any COCs found in the DMMU samples that exceed the associated DMMP screening levels (SLs) and/or SMS sediment quality standards (SQS).

7.4 Sample Collection and Handling Procedures

DMMP-approved sampling requirements, analytical methods, and quality assurance/quality control (QA/QC) criteria are included in Appendix A. Sample volumes, holding times, containers, preservatives, and chemical analytical methods are summarized in Table A-1 in Appendix A. QA/QC criteria are summarized in Tables A-2 through A-3.

7.4.1 Sample Collection

The sediment samples will be collected with a MudMole pneumatic corer. The sampler is a 4-inch-square aluminum core tube with a pneumatic driving assembly attached on top. The sampler will be operated by personnel on the sampling vessel in shallow waters and by a diver in deeper waters.

Existing mudline will be determined at each station (see Section 7.6) prior to sampling to document sampling depths at each location. The core sampler will be lowered to the bottom using a winch after reaching the selected sample station using on vessel GPS equipment to locate each station.

The operator will turn the pneumatic driver on after the core tube has entered the sediment. The operator will pause the driving operation every 2 feet to measure the penetration and recovery of the core. The internal recovery will be measured by lowering a weighted tape measure down the inside of the core tube until the weight contacts the surface of the sediment. The penetration will be measured using a second tape measure and reference marks on the outside of the core tube. The penetration and recovery readings will be relayed to the sampling vessel personnel using a wireless underwater diver communication system and will be recorded. Once the sample depth is achieved, the air hammer will be turned off and the final set of penetration and recovery measurements will be made. A lifting winch will be used to extract the core.

Additional cores may be collected at a station if the depth of penetration or the recovery is insufficient to meet the sampling and analysis needs of the study, including archive material for potential bioassays.

The core tubes will be labeled and will note which end is the top and/or bottom. The cores will be transported to the upland sample processing station and stored in an upright position.

Boring Logs

Penetration and recovery measurements are used to account for thinning and compaction of the sediments during driving. An on-deck top-of-sediment measurement from the top of the core tube to the surface of the sediment within the core tube accounts for any movement or loss of sediment in the core tube as the core catcher closes during extraction. The penetration and recovery data and the on-deck top-of-sediment measurement will be entered into a spreadsheet program to generate a bore log.

Each bore log will include a bore graph of penetration versus recovery that can be used during processing for identifying the in-situ depth of different sediment horizons. The X-axis on the bore graph is the distance to a sediment layer (in feet) referenced to the top of the tube, and the Y-axis is the in-situ depth (feet) below mudline. The solid line is the on-deck recovery (used in the processing of the core), and the gray dashed line is the in-situ recovery.

The bore logs for this project will have predefined in-situ sampling depths entered in the table on the right side of the bore log (see Figures 10 and 11 for an example bore graph and bore log, respectively). The distance that each in situ sampling depth is from the top of the tube will be estimated using the on-deck recovery curve. The in situ depth of a sediment structure or a sampling interval can be interpolated from the on-deck recovery information using the measured distance from the top of the tube.

The bore log with bore graph will be printed either onboard the sampling vessel or in the field laboratory from data recorded during the core driving. The field data form or the completed bore log will be kept with the core during transport and processing.

At a minimum, the following information will also be included in the bore log.

- Elevation of each station sampled as measured from MLLW at the time of sampling
- Station location determined in latitude and longitude using GPS
- Date and time of collection of each sample
- Names of field person(s) collecting and logging in the sample
- Sample characteristics, including grain size, density, and moisture

- Weather conditions
- Tidal conditions and tidal stage
- The sample station number as derived from this sampling plan
- Apparent resistance of the material to sampling based on the depth of penetration of the sampler
- Picture of each core
- Description of debris, including woody debris or logs (if encountered) or makes sampling problematic
- Any deviation from the approved sampling plan

7.4.2 Sampling Equipment Decontamination

All samplers and miscellaneous sampling tools will be thoroughly cleaned prior to use according to the following procedure.

- Water rinse
- Wash with brush and Alconox soap
- Triple rinse with distilled water

All sampling equipment not used immediately after cleaning will be wrapped in aluminum foil. The rule of "potential for contaminants" will be used such that any sampling equipment suspected of contamination will be rejected and decontaminated prior to use.

7.4.3 Sample Handling and Compositing

The core tube will be placed on a sawhorse and oriented with the hinged side of the core catcher to the side. The uppermost side of the core tube is removed using a circular saw. The depth of cut on the saw will be set to just slightly over the wall thickness of the aluminum tube. An approximate 1-cm (0.38-inch) layer will be removed from the exposed sediment surface with a decontaminated stainless-steel scraper. The surface layer of sediment will be removed starting at the bottom of the core tube and moving toward the top. This method minimizes potential contamination of clean, deeper layers with material from shallower, potentially more contaminated layers.

Sediments from each segment will be collected from the center of the core starting at the inserted plate marking the top of the segment. Sediment touching the sides of the core tube will be left in place. Sediment will be collected from each segment starting from below each inserted plate and extending down the core tube until sufficient sample volume is obtained. The distance down the tube that sediment is removed will be recorded to provide information on the actual collection interval for each sample.

The sample material will be composited and thoroughly mixed in stainless-steel bowls. Equal volumes of sample material from each core will be composited into one DMMU sample for analysis. Any sediment remaining after sample collection will be collected as archive material.

One to 2 liters of homogenized sample will be jarred to provide adequate volume for physical and chemical analyses (see Appendix A for sample jar requirements). Approximately 4 liters of the homogenized sample will be jarred (with zero headspace) to provide adequate volume for bioassay testing. The material collected for bioassay testing will be archived pending chemical analytical data results. The composited samples will be stored in iced coolers for transport to the laboratory.

All handwork (extruding, mixing, and compositing) will be performed using stainless-steel spoons and bowls. All sampling, mixing, and compositing equipment will be decontaminated prior to collection at each sampling station. Disposable latex/nitrile gloves will be used and will be rinsed with distilled water before and after handling each individual sample, as appropriate, to prevent sample contamination. Gloves will be disposed of between composites to prevent cross contamination.

7.4.4 Sulfides Subsampling

Sulfide samples will be preserved using 5 milliliters of 2 Normal zinc acetate per 30 grams of composited sediment (DMMP 2016b). The zinc acetate will be placed in a 4-ounce sampling jar and the sample material will be placed in the jar, covered, and shaken vigorously to completely expose the material to the zinc acetate.

7.5 Field Sampling Schedule

The sampling will be performed using sampling equipment owned and operated by Research Support Services of Bainbridge Island, Washington, and Amec Foster Wheeler of Seattle, Washington. The sampling is scheduled for the week of 26 February 2018.

7.6 Positioning

Station positions will be determined in latitude and longitude using a hand-held GPS unit (North American Datum 83/07) to the nearest 0.1 second. The accuracy of measured and recorded horizontal coordinates will be within 3 meters. The GPS receiver will be situated over the sampling gear to achieve the most accurate position for each sample. Sample coordinates are shown on Table 3.

Vertical elevations will be referenced to MLLW-based tidal stage and mudline elevations at the time of sampling. Mudline elevations will be calculated in the field based on elevation of mudline and determined by tide level and lead-line depth to mudline measurements. Real-time corrections will be completed based on depth of water column and National Oceanic and Atmosphere Association tidal gauge data (Aberdeen Station ID 9441187). Depths below mudline can typically be determined within approximately 0.1 foot.

7.7 Sample Transport and Chain-of-Custody Procedures

The samples will be transported to an accredited chemical analytical laboratory when the sampling and compositing is completed. Chain-of-custody procedures will be used to track sample handling from field collection through delivery of the samples to the laboratory. Specific procedures will be as follows.

- Samples will be packaged and shipped in accordance with U.S. Department of Transportation regulations, as specified in 49 CFR 173.6 and 49 CFR 173.24.
- Individual sample containers will be packed to prevent breakage and transported in a sealed ice chest or other suitable container. Ice enclosed in the sample cooler will be double-bagged and well-sealed. A temperature blank will be included in each sample cooler.
- The coolers will be clearly labeled with sufficient information (name of project, time and date container was sealed, person sealing the cooler, and BergerABAM's office name and address) to enable positive identification.
- A sealed envelope containing chain-of-custody forms will be enclosed in a plastic bag and taped to the inside lid of the cooler.
- Signed and dated chain-of-custody seals will be placed on all coolers prior to shipping.
- Sample coolers will be kept to 4 degrees C +/- 2 degrees and transported by vehicle to an accredited chemical analytical laboratory within 24 hours of being sealed.

The chain-of-custody form will be signed by the persons transferring custody of the coolers upon transfer of sample possession to the laboratory. The receiver will break open the shipping container seal and record the condition of the samples upon receipt of samples at the laboratory. Chain-of-custody forms should be used internally in the lab to track sample handling and final disposition.

8.0 LABORATORY PHYSICAL AND CHEMICAL SEDIMENT ANALYSIS

8.1 Analysis Program

The analysis program for this project has been developed primarily to evaluate suitability for open-water disposal in accordance with DMMP. Each DMMU composite sample will be analyzed for DMMP/SMS conventional parameters, the full suite of DMMP/SMS COCs, SVOCs, PCBs, PCDD/F, pesticides, and bulk tributyltin, as shown on Table 2. Sediment reference material will also be submitted for analysis of PCBs and PCDD/F in accordance with DMMP guidance. DMMP SLs and SMS SQSs are shown in Table 2.

Puget Sound Sediment Reference Material will be requested from the DMMO and will be submitted for PCB and dioxins/furans analysis for data quality evaluation. Certified reference material, as identified in documentation provided by Analytical Resources, Inc. (ARI) and included as Appendix B, will be used for data evaluation purposes for the metals, SVOCs, and pesticides analyses. The material will be handled and analyzed in accordance with DMMP guidance (DMMP 2016b).

The chemical analytical data generated from the chemical analysis will also be used to evaluate general sediment quality in accordance with the SMS (also shown in Table 2). Information regarding the chemical characteristics of sediments that will be potentially suspended and/or dispersed during construction may be required for obtaining Ecology's Water Quality Certification permits for the project.

Results of the SMS evaluation will be used to determine the antidegradation status of the surface material exposed by dredging, potential suitability of the material for beneficial use.

8.2 Laboratory Analyses Protocols

Analytic protocols, including sample holding times and method detection limits, will be in accordance with Environmental Protection Agency, PSEP, and DMMP's User Manual protocols and requirements. Laboratory testing procedures will be conducted in accordance with the DMMP's User Manual recommended protocols. Several details of these procedures are discussed below. Laboratory testing procedures will be conducted in accordance with the DMMP recommended protocols. Details of these procedures are discussed below.

8.3 Chain-of-Custody

A chain-of-custody record for the samples will be maintained throughout all sampling activities and will accompany samples during shipment to the laboratory, as previously described. Information tracked by the chain-of-custody records in the laboratory include sample identification number, date and time of sample receipt, analytical parameters required, location and conditions of storage, date and time of removal from and return to storage, signature of person removing and returning the sample, reason for removing from storage, and final disposition of the sample.

8.4 Limits of Detection

The samples will be analyzed for all the parameters listed in Table 2. Detection limits of all chemicals of concern must be below DMMP SLs. Failure to achieve this may result in a requirement to reanalyze or to conduct bioassays. All reasonable means, including additional cleanup steps and method modifications, will be used to bring all limits-of-detection below DMMP SLs.

All conventional parameters, including grain size, total organic carbon, total solids, total volatile solids, ammonia, and sulfides, will be analyzed. Particle grain-size distribution for each composite sample will be determined in accordance with American Society for Testing and Materials (ASTM) D 422 (modified). Wet sieve analysis will be used for the sieve sizes U.S. No. 4, 10, 20, 40, 60, 140, 200, and 230. Hydrogen peroxide will not be used in preparations for grain-size analysis. Hydrometer analysis will be used for particle sizes finer than the 230 sieve.[4] Water content will be determined using ASTM D 2216. Sediment classification designation will be made in accordance with U.S. Soil Classification System, ASTM D 2487.

8.5 Quality Assurance/Quality Control

The chemistry QA/QC procedures will follow PSEP and the QA/QC criteria established for the Puget Sound Dredged Disposal Analysis (PSDDA) Program/DMMP. The bioassay procedures will follow PSEP protocols and Puget Sound Water Quality Authority "Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments" and the QA/QC criteria established for the SMS/DMMP programs. Bioassay performance standards and evaluation guidelines are included in Appendix A (Table A-3).

8.6 Bioassay Archives

The need to submit samples for bioassay testing will be evaluated after the dredged material characterization data results are reviewed. The exceedance of one or more SLs for DMMP COCs in the DMMU samples will trigger bioassay testing. All reporting limits must also be less than DMMP SLs in order to avoid the need for bioassay testing. All possible means, including re-extraction, additional cleanup steps, or different analysis methods, may be used to lower reporting limits.

A reference sediment sample will be collected from one of the recommended stations for Grays Harbor, depending on the grain size and TOC percent of the DMMU/subunit samples. The sediment reference sampling location will be confirmed with the DMMO prior to collecting the sediment.

The reference sample will be collected using a Van Veen or power-grab sampler operated from a vessel outfitted for that purpose. Reference samples will be collected in laboratory-supplied containers and kept in iced coolers until they are delivered to the analytical laboratory. The grain size of the reference sediment will be determined in the field by using the wet sieving method with a #230 sieve and a graduated cylinder. The reference sample will be used if the fines content is within 10 percent of the fines content of the test sediment. If the fines content of the reference sediment is outside of that range, a different reference sample will be collected and used.

Samples selected for bioassay testing will be submitted for both acute and chronic tests to characterize toxicity. Bioassay testing will include the following tests.

- 10-day amphipod (*Eohaustorius estuarius*) mortality testing (acute toxicity)
- 20-day juvenile infaunal (*Neanthes arenaceodentata*) growth test (chronic toxicity)

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[4] No. 10 should be used to distinguish between gravel and sand and No. 230 to distinguish between fines and other.

- Sediment larval (Mytilus galloprovincialis or Dendraster excentricus) test (acute toxicity)

PSEP protocols will be followed with DMMP Sediment Management Annual Review Meeting (SMARM) updates (if any). Interstitial salinity will be measured prior to the initiation of the bioassay tests. Un-ionized ammonia and hydrogen sulfide will be measured prior to test initiation to determine the need for purging based on the parameters in the following table. The mediums of exposure needed for the measurement of ammonia and hydrogen sulfide are

- Porewater, for bedded sediment tests using *Neanthes*, *Eohaustorius*, and *Rhepoxynius*.
- Overlying water, for amphipod bioassay tests using *Ampelisca* and for the species used in the larval development test.

Purging, if needed, will be accomplished by replacing overlying water twice a day in the sample and surrogate sediment test beakers in conjunction with continuous aeration. The ammonia and hydrogen sulfide concentrations will be monitored in surrogate samples after each purge until the ammonia and hydrogen sulfide concentrations are less than the triggers in the table below. Each batch of test sediments will have associated and similarly purged control and reference sediments. Bioassay testing will then be initiated.

Reference Toxicant and Purging Triggers for the Various Bioassays (DMMP, 2015)

Trigger	**Bedded Sediment Test Triggers (mg/L)**				**Larval Test Triggers (mg/L)**	
	Neanthes	***Ampelisca***	***Eohaustorius***	***Rhepoxynius***	**Bivalve**	**Echinoderm**
Reference Toxicant						
Un-ionized Ammonia	0.23	0.118	0.4	0.2	0.02	0.007
Purging						
Un-ionized Ammonia	0.46	0.236	0.8	0.4	0.04	0.014
Hydrogen Sulfide	3.4	0.0094	0.122	0.099	0.0025	0.01

- Water quality measurements of the following parameters will be collected daily during amphipod and sediment larval bioassay testing.
- Temperature
- Aqueous salinity
- pH
- Dissolved oxygen (DO)

A negative control test will be run by the bioassay-testing laboratory. Sediment samples collected from lower Yaquina Bay near Newport, Oregon, will be used for the negative control.

8.7 Laboratory Written Report

A written report will be prepared by the analytical laboratory documenting all the activities associated with sample analyses. At a minimum, the following will be included in the report.

- Results of the laboratory analyses and QA/QC results, including case narrative
- All protocols used during analyses
- Chain-of-custody procedures, including explanation of any deviation from those identified herein
- Any protocol deviations from the approved sampling plan
- Location and availability of data
- QA2 data required by Ecology
- Electronic data deliverable in EIM format

As appropriate, this sampling plan may be referenced in describing protocols.

9.0 REPORTING

9.1 Quality Assurance/Quality Control Report

The project QA/QC representative will prepare a QA/QC report based upon activities involved with the field sampling and review of the laboratory analytical data. The laboratory QA/QC reports will be incorporated by reference. This report will identify any field and laboratory activities that deviated from the approved sampling plan and the referenced protocols and will make a statement regarding the overall validity of the data collected. The QA/QC report will be incorporated into the final report.

9.2 Final Report

BergerABAM shall prepare a written report documenting all activities associated with collection, compositing, transportation of samples, and chemical and biological analysis. The chemical analytical report will be included as an appendix. The following will be included in the Dredged Material Characterization Report.

- Type of sampling equipment used.
- Protocols used during sampling and testing and an explanation of any deviations from the sampling plan protocols.
- Logs of the samples showing descriptions of each sample and identifying horizons etc. along the full length of the core.
- Methods used to locate the sampling positions within an accuracy of 3 meters.
- Locations where the samples were collected. Locations will be reported in latitude and longitude to the nearest tenth of a second.

- A plan view of the project showing the actual sampling locations and DMMU boundaries.
- Chain-of-custody procedures used and explanation of any deviations from the sampling plan procedures.
- Description of sampling and compositing procedures.
- Final QA/QC report and validation report.
- Data results relative to DMMP and SMS criteria in a table.
- Depth of recovery for each sample.
- Measured water depth and tide information for each sampling location confirmation of sediment elevation.
- A table showing the compositing scheme for each DMMU.
- Bioassay results, including bioassay laboratory report, if applicable.
- Data in EIM format submitted to DMMO.
- QA2 data required by Ecology for data validation prior to entering data in their Sediment Quality database and to the USACE in electronic format.
- Project cost data will be forwarded to the DMMO separately.

10.0 PROJECT TEAM AND RESPONSIBILITIES

10.1 General

The SAP includes (1) project planning and coordination; (2) field sample collection; (3) laboratory preparation and analyses; (4) QA/QC management; and (5) final data report. The program will use the following team members and responsibilities.

10.1.1 Project Planning and Coordination

Ms. Sally Fisher, of BergerABAM, is the senior project manager with overall responsibility for characterization activities and project permitting coordination.

10.1.2 Field Sample Collection

Ms. Victoria England will provide overall direction to the field and laboratory programs, and Ms. April Ryckman will coordinate field activities. Ms. England will be responsible for assuring that all the required logistics elements and protocols are followed, including accurate sample positioning, sample handling and field decontamination procedures, physical evaluation and logging of samples, and chain of custody of the samples until delivered to the analytical laboratory. Samples will be collected using equipment owned and operated by a subcontractor licensed to work in the state of Washington.

10.1.3 Laboratory Analysis

ARI will perform chemical analysis for this project. ARI will be provided with this SAP and will be required to follow the procedures described herein. Bioassays will be performed by Northwestern Aquatic Sciences. The laboratory reports will include any deviations from the SAP in their analytical data packages for the project.

10.1.4 Quality Assurance/Quality Control Management

Ms. Fisher will provide general QA/QC oversight and senior review for the field sampling and laboratory programs. Ms. England will review laboratory QA/QC data to assure validity of data and conformance to QA/QC requirements, and will provide a written QA/QC report.

10.1.5 Final Data Report

Ms. England will be responsible for preparation of the final sampling data report identifying sample locations, field and laboratory methods, QA/QC, lessons learned, and data results.

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Table 1. Compositing Scheme and DMMU Volumes

DMMU ID	Core Station	DMMU Subunit ID	Sample Depth (ft below mudline)	Assumed Sample Elevation (MLLW)	Z-layer Elevation (MLLW)	Total Core Length with Z layer	Z-layer Sample ID	Ranking	Frequency Requirement Based on Ranking	Approximate Total DMMU Volume (cy)
DMMU-1 (surface)	SS-1	SS-1A	0-4	-32 to -36	--	--	--	low-moderate	1 subsample/ 8,000 cy and 1 DMMU/32,000 cy (surface)	23,217
	SS-2	SS-2A	0-4	-25 to -29	--	--	--			
	SS-5	SS-5A	0-4	-27 to -31	--	--	--			
	SS-6	SS-6A	0-4	-0 to -4	--	--	--			
DMMU-2 (surface)	SS-3	SS-3A	0-4	-36 to -40	--	--	--			23,268
	SS-4	SS-4A	0-4	-35 to -39	--	--	--			
	SS-7	SS-7A	0-4	-37 to -41	--	--	--			
	SS-8 (corrected)	SS-8A	0-4	--37 to --41	--	--	--			
DMMU-3 (subsurface)	SS-1	SS-1B	4-8	-36 to -38	-38 to -40	8	Z1	low-moderate	1 subsample/ 8,000 cy and 1 DMMU/48,000 cy (subsurface)	31,768
	SS-2	SS-2B	4-15	-29 to -33	-33 to -35	10	Z2			
	SS-5	SS-5B	4-9	-31 to -45*	-45 to -47	20	Z5			
	SS-6 (corrected)	SS-6B	4-10	-4 to -10	-10 to -12	12	Z6			
DMMU-4 (subsurface)	SS-3	SS-3B	4-12	-40 to -45	-45 to -47	11	Z3			31,747
	SS-4	SS-4B	4-9	-39 to -45	-45 to -47	12	Z4			
	SS-7	SS-7B	4-10	-41 to -45	-45 to -47	10	Z7			
	SS-8 (corrected)	SS-8B	4 to 11	-41 to -45	-45 to -47	10	Z8			
										110,000

*Total core lengths may be unachievable due to limitations of site conditions and equipment. We propose to take the bottom 2 feet and archive this material as representative of the post-dredge Z-layer. NOTE: Archive material will be collected every 2 feet from each core.

Table 2. DMMP and SMS Chemical Evaluation Criteria[1]

	DMMP Guidance			SMS Criteria	
Chemical	SL	BT	ML	SQS	CSL
Conventionals					
Total Solids (%)	--	--	--	--	--
Total Volatile Solids (%)	--	--	--	--	--
Total Organic Carbon (%)	--	--	--	--	--
Ammonia (mg/kg)	--	--	--	--	--
Total Sulfides (mg/kg)	--	--	--	--	--
Metals[2] mg/kg dry wt.					
Antimony	150	--	200	--	--
Arsenic	57	507.1	700	57	93
Cadmium	5.1	--	14	5.1	6.7
Chromium	260	--	--	260	270
Copper	390	--	1,300	390	390
Lead	450	975	1,200	450	530
Mercury	0.41	1.5	2.3	0.41	0.59
Selenium	--	3	--	--	--
Silver	6.1	--	8.4	6.1	6.1
Zinc	410	--	3,800	410	960
Organometallic Compounds					
Tributyltin – bulk (ug/kg)	--	73	--	--	--
PAHs					
LPAH[3] µg/kg dry wt.					
Total LPAH[4]	5,200	--	29,000	370	780
Acenaphthylene	560	--	1,300	66	66
Acenaphthene	500	--	2,000	16	57
Anthracene	960	--	13,000	220	1,200
Fluorene	540	--	3,600	23	79
Naphthalene	2,100	--	2,400	99	170
Phenanthrene	1,500	--	21,000	100	480
2-Methylnaphthalene	670	--	1,900	38	64
HPAH[3] µg/kg dry wt,					
Total HPAH[5]	12,000	--	69,000	960	5,300
Benzo(a)anthracene	1,300	--	5,100	110	270
Benzo(a)pyrene	1,600	--	3,600	99	210
Total Benzofluoranthenes[6]	3,200	--	9,900	230	450
Benzo(g,h,i)perylene	670	--	3,200	31	78
Chrysene	1,400	--	21,000	110	460
Dibenzo(a,h)anthracene	230	--	1,900	12	33
Fluoranthene	1,700	4,600	30,000	160	1,200
Indeno(1,2,3-c,d)pyrene	600	--	4,400	34	88
Pyrene	2,600	11,980	16,000	1,000	1,400

Table 2. DMMP and SMS Chemical Evaluation Criteria[1] (continued)

Chemical	DMMP Criteria			SMS Criteria	
	SL	BT	ML	SQS	CSL
Miscellaneous Extractables[3] µg/kg dry wt.					
Dibenzofuran	540	--	1,700	15	58
Hexachlorobutadiene	11	--	270	3.9	6.2
N-Nitrosodiphenylamine	28	--	130	11	11
Benzoic Acid	650	--	760	650	650
Benzyl Alcohol	57	--	870	57	73
Chlorinated Hydrocarbons[3] µg/kg dry wt.					
Hexachlorobenzene	22	168	230	0.38	2.3
1,2-Dichlorobenzene	35	--	110	2.3	2.3
1,4-Dichlorobenzene	110	--	120	3.1	9
1,2,4-Trichlorobenzene	31	--	64	0.81	1.8
Phthalates[3] µg/kg dry wt.					
Bis(2-ethylhexyl)phthalate	1,300	--	8,300	47	78
Butyl benzyl phthalate	63	--	970	4.9	64
Diethyl phthalate	200	--	1,200	61	110
Dimethyl phthalate	71	--	1,400	53	53
Di-n-butyl phthalate	1,400	--	5,100	220	1,700
Di-n-octyl phthalate	6,200	--	6,200	58	4,500
PCBs[3] µg/kg dry wt.					
Total PCBs	130	38[7]	3,100	12	65
Pesticides[3] µg/kg dry wt.					
4,4 DDD	16	--	--	--	--
4,4 DDE	9	--	--	--	--
4,4 DDT	12	--	--	--	--
Total DDT	--	50	69	--	--
Aldrin	9.5	--	--	--	--
Dieldrin	1.9	--	1700	--	--
Total Chlordane[8]	2.8	37	--	--	--
Heptachlor	1.5	--	270	--	--
Phenols[3] µg/kg dry wt.					
Pentachlorophenol	400	504	690	360	690
Phenol	420	--	1,200	420	1,200
2 Methylphenol	63	--	77	63	63
4 Methylphenol	670	--	3,600	670	670
2,4-Dimethylphenol	29	--	210	29	29
Dioxins/Furans					
Total TEQ (ppt dry wt)[9] (see DMMO Dioxin page)	-	15	-	-	-

Table 2. DMMP and SMS Chemical Evaluation Criteria[1] (continued)

Notes:

1. DMMP = Dredged Material Management Program (August 2016), SMS = Sediment Management Standards (April 2017).
2. Dry weight results are reported as milligrams per kilogram (mg/kg).
3. Dry weight results are micrograms per kilogram (µg/kg).
4. Total LPAH = The sum of acenaphthylene, acenaphthene, anthracene, fluorene, naphthalene and phenanthrene.
5. Total HPAH = The sum of benzo(a)anthracene, benzo(a)pyrene, total benzofluoanthenes, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, indeno(1,2,3,-c,d)pyrene and pyrene.
6. Total benzofluoranthenes = the sum of the "b", "j" and "k" isomers. The "j" isomer co-elutes with the "k" isomer, thus the concentration of the "j" isomer is included in the "k" isomer concentration.
7. This value is normalized to total organic carbon and is expressed in mg/kg carbon.
8. Total Chlordanes = The sum of cis-chlordane, trans-chlordane, cis-nonachlor, trans-nonachlor, and oxychlordane.
9. For the dispersive sites in Grays Harbor, each disposed DMMU must have a 2,3,7,8-TCDD concentration less than or equal to 5 ng/kg and a TEQ of less than or equal to 15 ng/kg. DMMUs with concentrations above these levels would be required to undergo bioaccumulation testing in order to qualify for open-water disposal.

SL = Screening Level
SQS = Sediment Quality Standards
CSL = Cleanup Screening Levels
BT = Bioaccumulation Trigger
ML = Maximum Level
LPAH = low molecular weight polynuclear aromatic hydrocarbon compounds
HPAH = high molecular weight polynuclear aromatic hydrocarbon compounds
TOC = Total organic carbon

Shading indicates that the criteria and results are TOC normalized. To normalize to total organic carbon, the dry weight concentration for each parameter is divided by the decimal fraction representing the percent total organic carbon content of the sediment.

Table 3. Proposed Sample Coordinates

Sample Point	WA State Plane		Decimal Degrees		UTM Zone 10 N	
	Northing	Easting	Longitude	Latitude	Northing	Easting
SS-1	615437.1732	787468.2999	-123.9176518	46.97030739	5202273.124	430196.556
SS-2	615423.9202	787741.4566	-123.9165563	46.97030352	5202271.719	430279.8773
SS-3	615255.9113	787583.4283	-123.9171596	46.96982453	5202219.027	430233.3661
SS-4	615301.0642	787882.1636	-123.9159719	46.96998368	5202235.657	430323.9151
SS-5	615368.5402	788095.3073	-123.9151307	46.97019382	5202258.261	430388.1808
SS-6	615397.4067	788389.0691	-123.9139601	46.97030776	5202269.883	430477.3719
SS-7	615243.4884	787991.8488	-123.915523	46.96983898	5202219.178	430357.8759
SS-8	615237.011	788367.2874	-123.9140195	46.96986579	5202220.822	430472.2832

Datum: NAD 1983 State Plane Washington South FIPS 4602 Feet

SS-6 and SS-8 coordinates will be verified/modified prior to sampling based on corrections to plan by SLF on 2/24/18

Sampling and Analysis Plan
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

Figures 1 through 11

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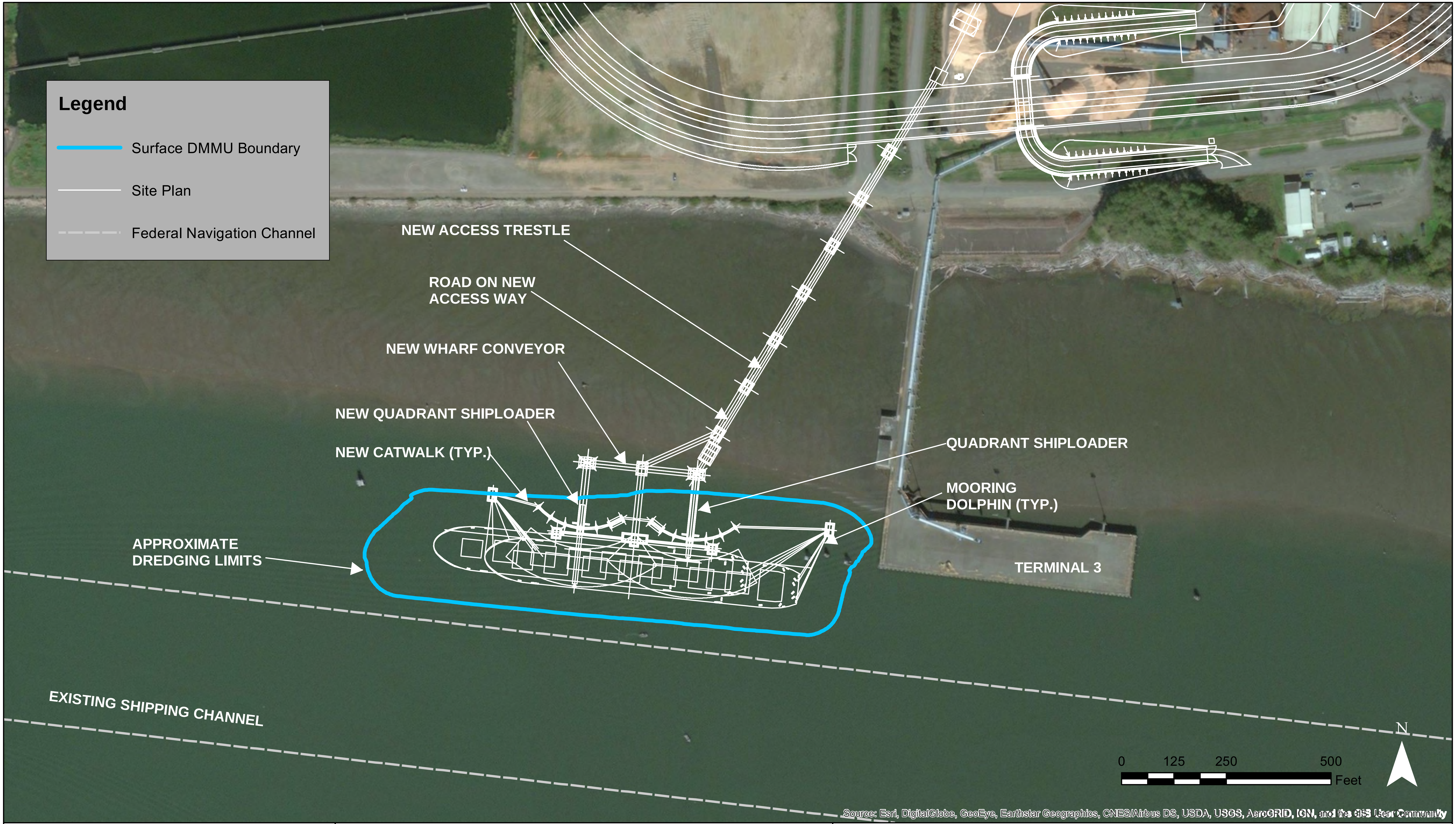
Proposed Grays Harbor Potash Export Facility-

Dredged Material Characterization Sampling and Analysis Plan

Figure 1- Vicinity Map and Project Site Location

February 2018



Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community

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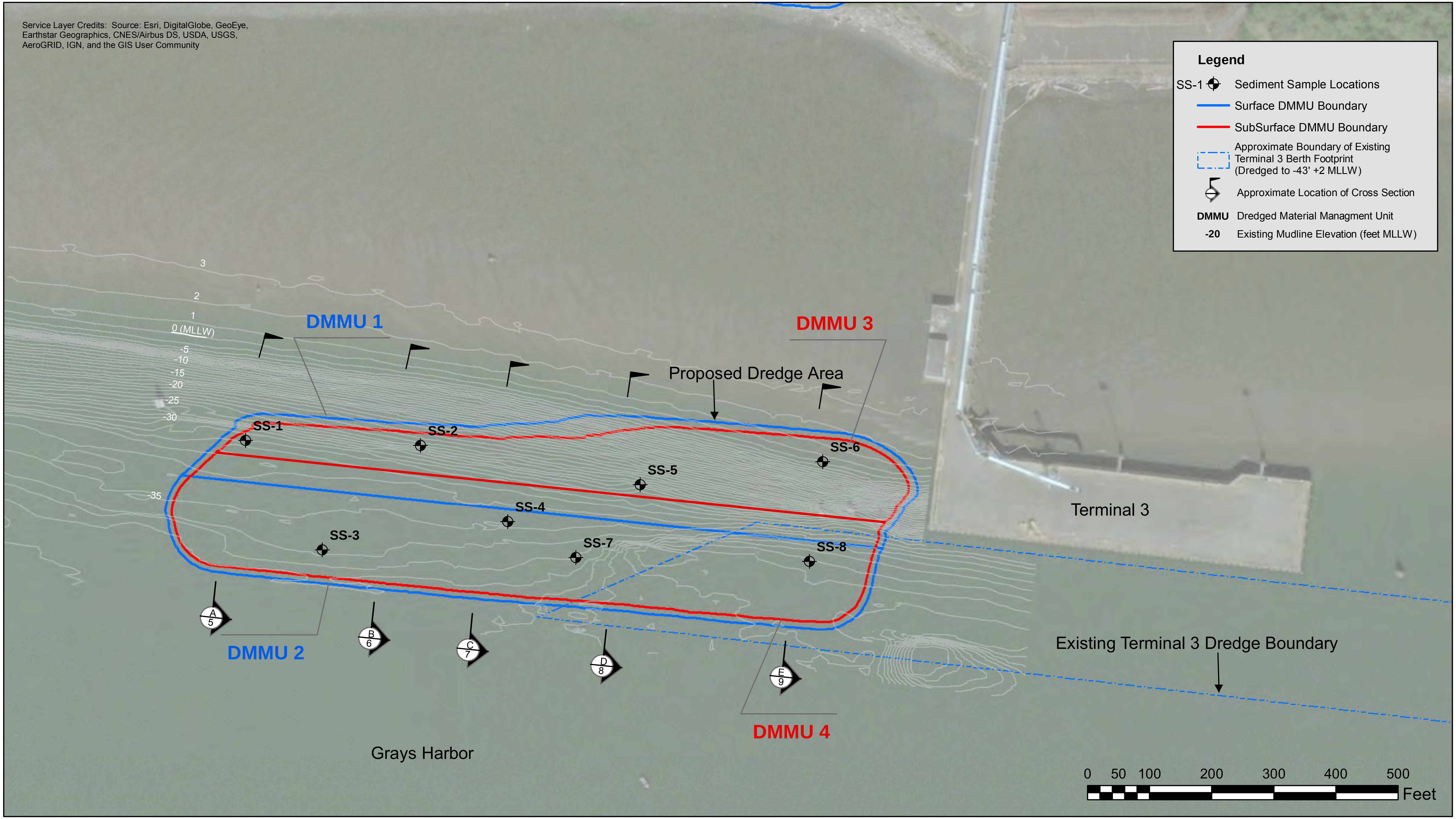
Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 2- Proposed Project Features
February 2018

General Notes
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM; WASHINGTON STATE PLANE SOUTH (WSPS), NAV83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING 2017.



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Proposed Grays Harbor Potash Export Facility - Dredged Material Characterization Sampling and Analysis Plan

Figure 3 - Site Plan-DMMUs and Proposed Sample Locations

February 2018

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83,US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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210 East 13th Street, Suite 300
Vancouver, Washington 98660-3231
(360) 823-6100 FAX: (360) 823-6101

Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 4 - Proposed Dredge Area and Bathymetry

February 2018

1" = 120'
60 0 60 120
scale feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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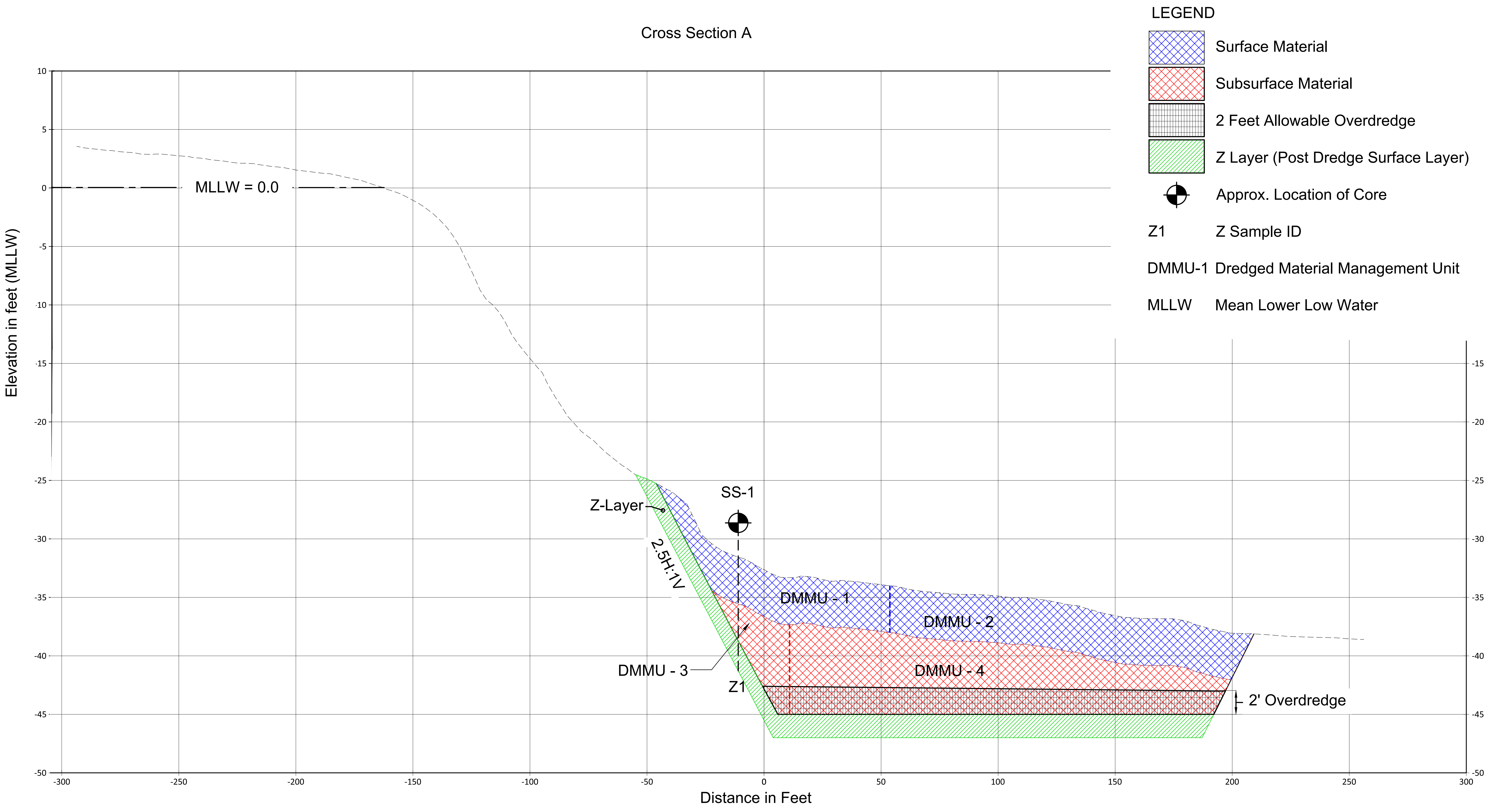

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Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 5 - Cross Section A: STA 3+00

February 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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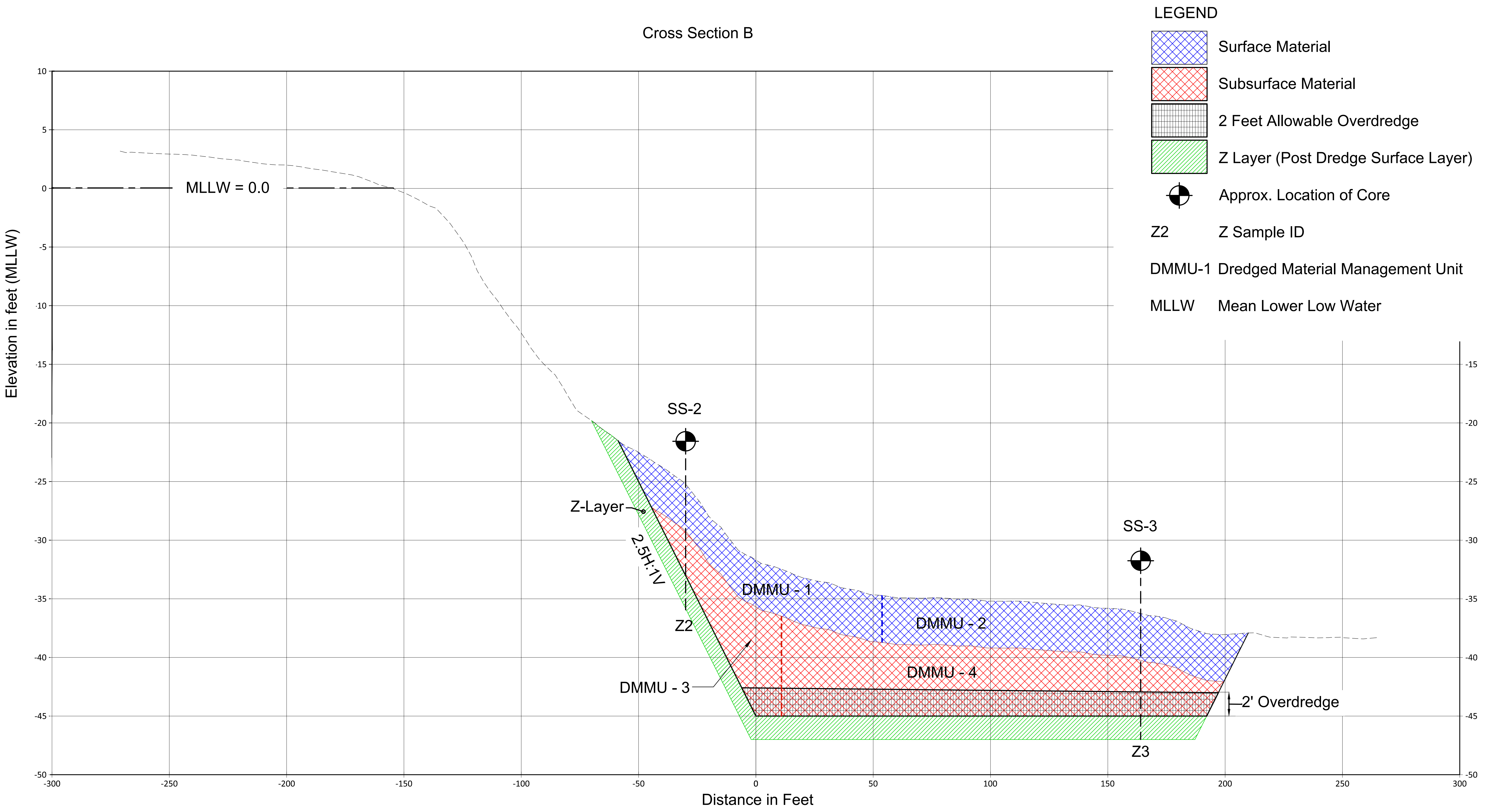

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Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 6 - Cross Section B: STA 5+00

February 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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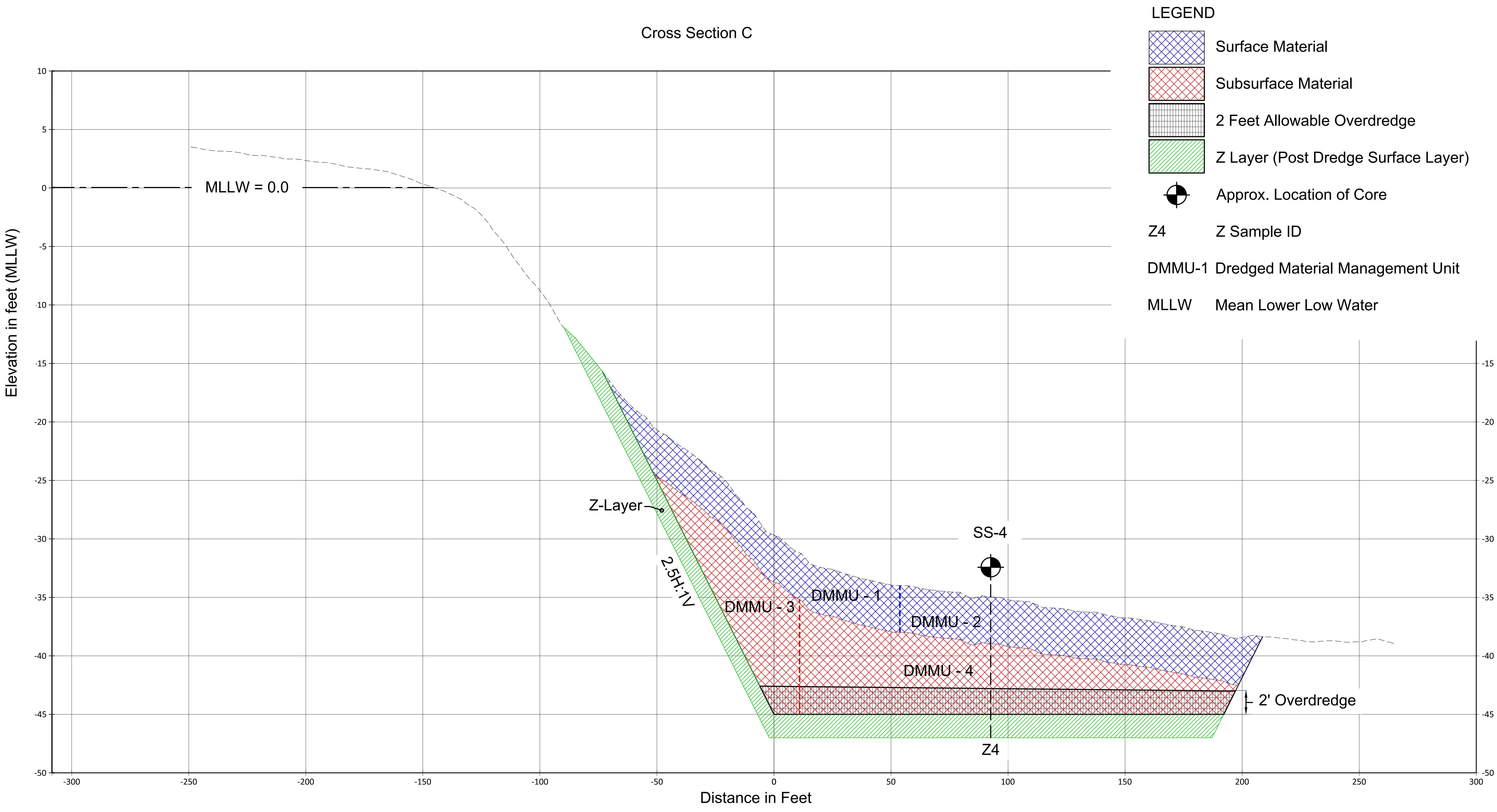

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Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 7 - Cross Section C: STA 7+00

February 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet
Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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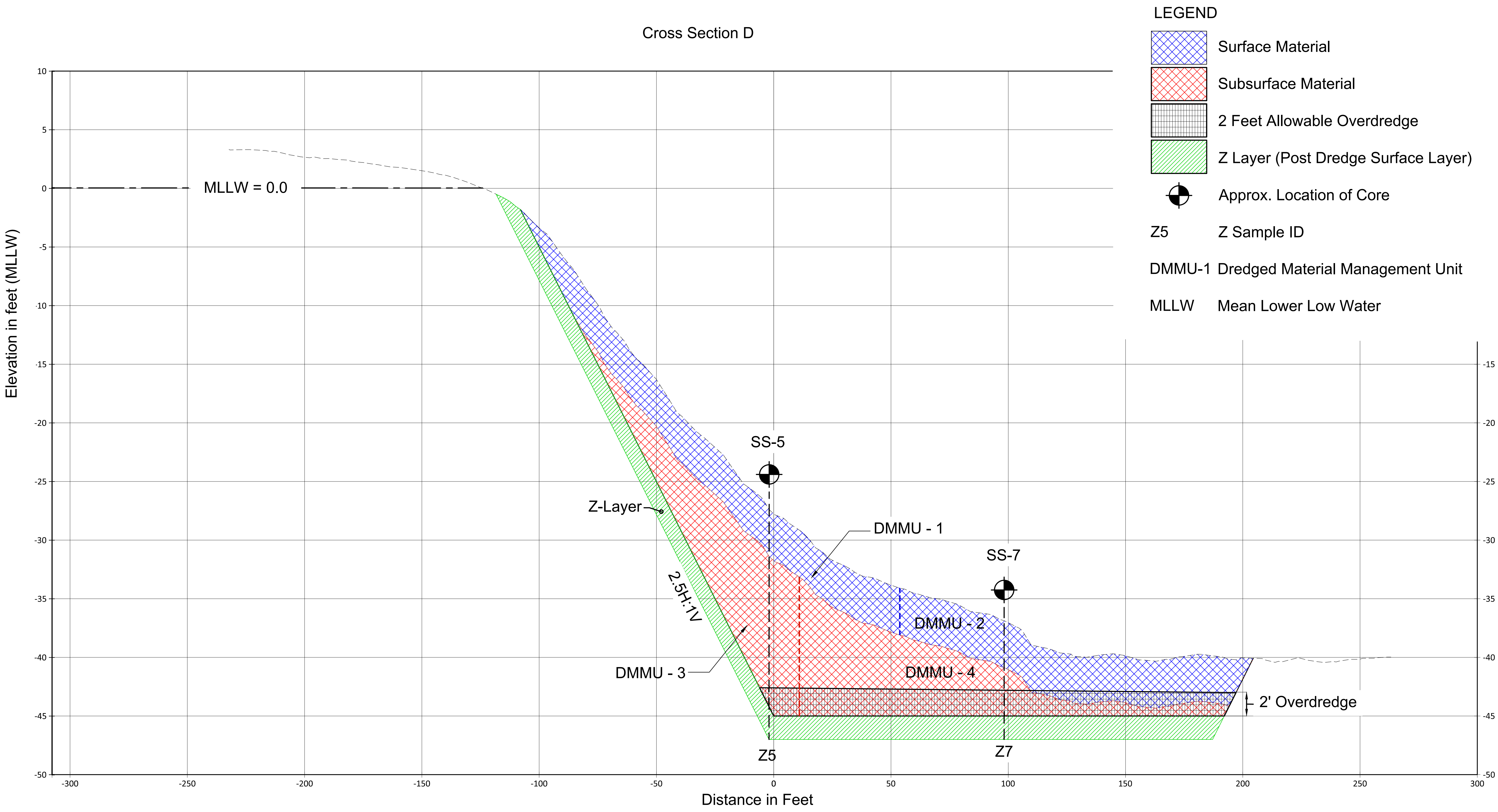

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Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 8 - Cross Section D: STA 9+00

February 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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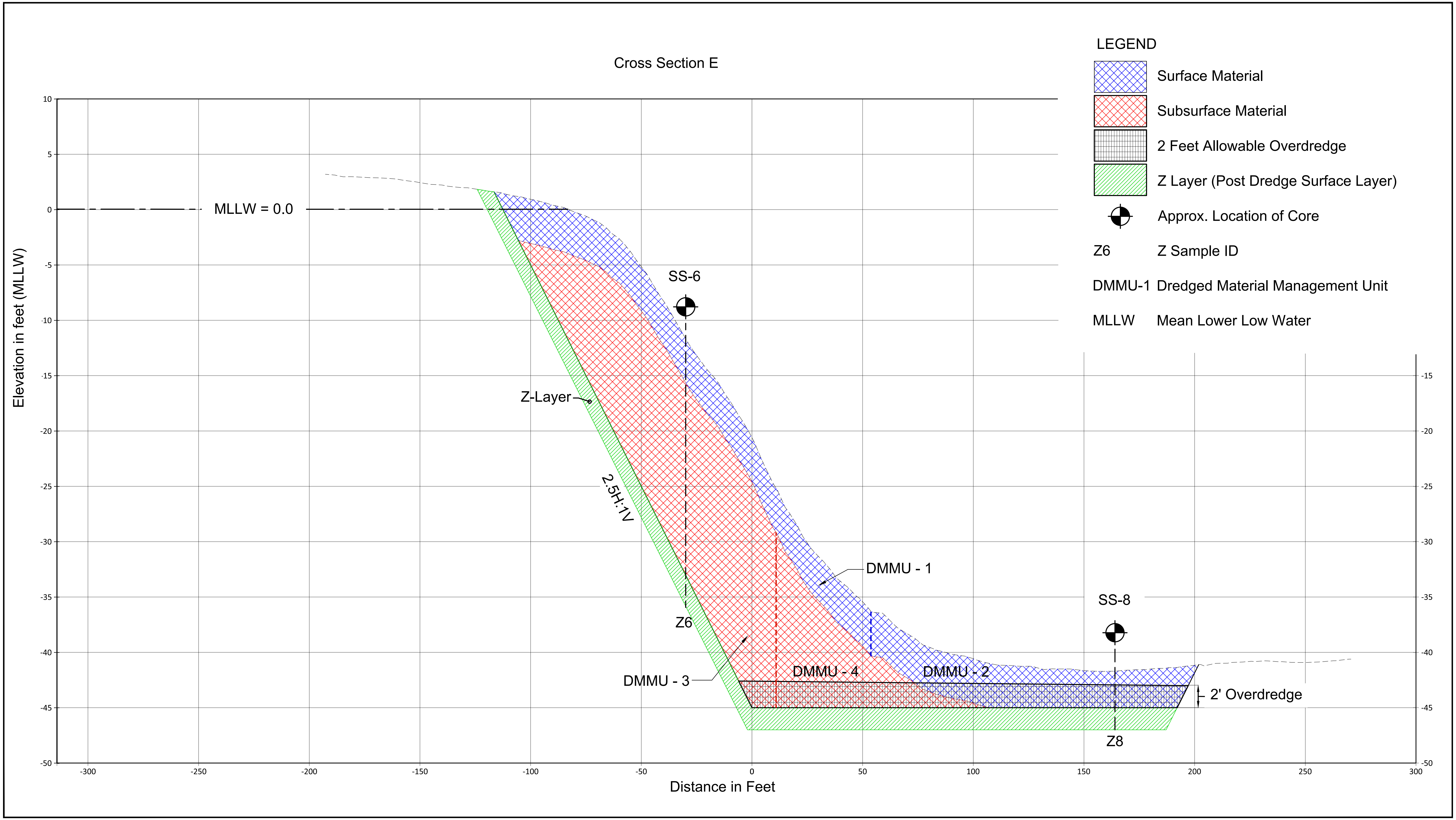

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Proposed Grays Harbor Potash Export Facility -
Dredged Material Characterization Sampling and Analysis Plan

Figure 9 - Cross Section E: STA 12+00

February 2018

Horizontal Scale 1" = 40' — 20 0 20 40 feet

Vertical Scale 1" = 8' — 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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Project: **Station:**

Project No:

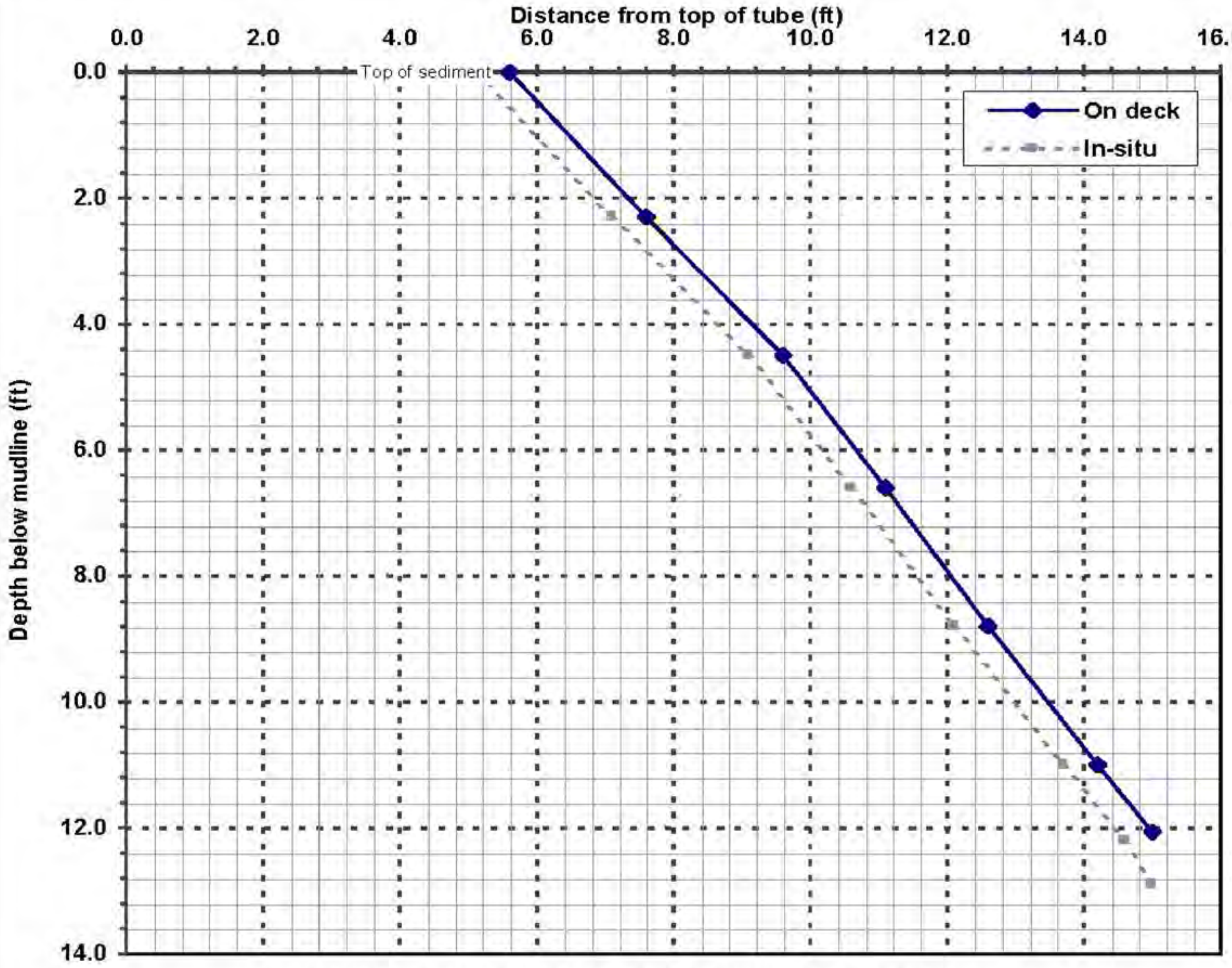
Collected by: Gary Maxwell

Date: 5/16/01 **Time:** 10:35

Water depth: 14.0 ft **Mudline:** -6.0 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: Sunny Driven to refusal



Penetration 12.9 ft/ On deck recovery 9.4 ft = 73% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-2.3	2	87%
2.3-4.5	2	91%
4.5-6.6	1.5	71%
6.6-8.8	1.5	68%
8.8-11	1.6	73%
11-12.2	0.9	75%
12.2-12.9	0.4	57%

Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	5.6
1	6.47
2	7.34
3	8.24
4	9.15
5	9.96
6	10.67
7	11.37
8	12.05
9	12.75
10	13.47
11	14.20
12	14.95
13	No sample
14	No sample
15	No sample

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Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization Sampling and Analysis Plan

Figure 10 - Example Bore Graph
February 2018

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Project: Boeing DSOA Vertical Characterization
Project No: 12301-13

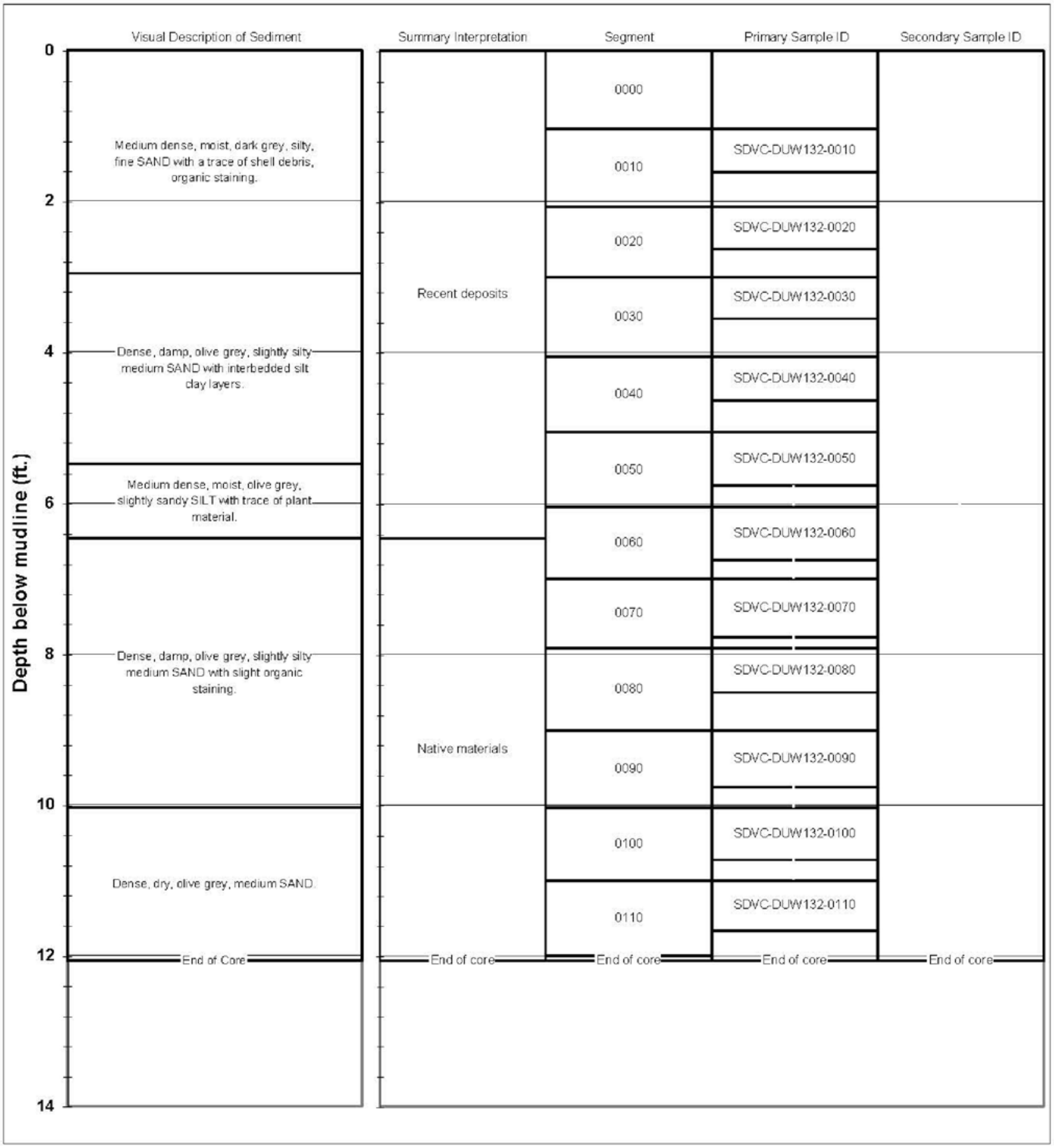
Station: SD-DUW132

Maximum depth of retained sediment: 12.1 ft
Percent recovery (on-deck): 73%

Mudline elevation: -6.0 **ft (MLLW)**

	Core collection	Laboratory processing
Date:	5/16/01	5/16/01
Time:	10:35	13:30

Field Log: Robert Gilmour
Summary Log: Robert Gilmour



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Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization Sampling and Analysis Plan

Figure 11 - Example Bore Log February 2018

Sampling and Analysis Plan
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

Appendix A

Sample Containers, Holding Times, Volume, and Chemical Analytical Methods and QA/QC Criteria

Table A-1. Sample Analytical Methods and Storage Criteria

Sample Type	Analytical Methods	Holding Time	Sample Size (1)	Temperature (2)	Container	Archive (3)
Particle Size	**PSEP (1986)/ASTM D-422 (modified)**	6 Months	100-200 g (75-150 ml)	4 degrees C	16 oz. glass jar	X
Total Solids	**PSEP (1986) / SM2540G**	14 Days	125 g (100 ml)	4 degrees C	8 oz. glass jar	
Total Volatile Solids	**PSEP (1986) / SM2540G**	14 Days	125 g (100 ml)	4 degrees C		
Total Organic Carbon	**SM 5310B/EPA 9060 (modified for sediments)**	14 Days	125 g (100 ml)	4 degrees C		
Ammonia	**Plumb (1981)**	7 Days	25 g (20 ml)	4 degrees C	4 oz. glass jar	
Metals (except Mercury)	**6010/6020/ 7000 & 200 Series**	6 Months	50 g (40 ml)	4 degrees C	4 oz. glass jar	
		2 years		-18 degrees C		
Semi-volatiles, Pesticides and PCBs	**8082/8270**	14 Days until extraction	150 g (120 ml)	4 degrees C	SVOC: 8 oz. glass jar Pesticides/PCBs: 8 oz. glass jar	
		1 Year until extraction		-18 degrees C		
		40 Days after extraction		4 degrees C		
Total Sulfides	**PSEP (1986) / Plumb (1981)**	7 Days	50 g (40 ml)	4 degrees C (4)	4 oz. glass jar	
Mercury	**7470/7471**	28 Days	50 g (40 ml)	-18 degrees C	4 oz. glass jar	

Sample Type	Analytical Methods	Holding Time	Sample Size (1)	Temperature (2)	Container	Archive (3)
Tributyltin (bulk)	**Krone (1989) / Unger (1986) / (PSEP 1997)**	6 months	50 g (40 ml)	-18 degrees C (5)	4 oz. glass jar	X
Dioxins/Furans	**EPA 1613B**	14 days until extraction	8 oz	4 degrees C	8 oz glass (amber)	
		1 year until extraction;		-18 +/- degrees C		
Bioassay	**10-day amphipod mortality test (acute toxicity)** **20-day juvenile infaunal growth test (chronic toxicity)** **Sediment larval test (acute toxicity)**	8 Weeks	4 liters	4 degrees C (5)	4-1-liter glass or polyethylene	X; bioassay archives are not frozen

(1) Recommended minimum field sample sizes for one laboratory analysis. Actual volumes to be collected have been increased to provide a margin of error and allow for retests.
(2) During transport to the lab, samples will be stored on ice. All temperatures are +/- 2 degrees Celsius. The mercury and archived samples will be frozen immediately upon receipt at the lab.

(3) For every DMMU, a 250-ml container is filled and frozen to run any or all of the analyses indicated.

(4) The sulfides sample will be preserved with 5 ml of 2 Normal zinc acetate for every 30 g of sediment.

(5) Headspace purged with nitrogen.

Table A-2. Laboratory QA/QC Requirements for Conventionals and COCs

Analysis Type	Method Blanks[1]	Replicates[1]	Triplicates[1]	CRM/RM	MS/MSD[1]	Surrogates[2]
Semivolatiles[3,4]	X[5]	X[6]		X	X	X
Pesticides[3,4]	X[5]	X[6]		X	X	X
PCBs[3,4]	X[5]	X[6]		X[7]	X	X
Dioxins	X[5]	X[6]		X[7]	X	X
Metals	X	X		X	X	
Ammonia	X		X			
Total Sulfides	X		X			
Total Organic Carbon	X		X	X		
Total Solids			X			
Total Volatile Solids			X			
Grain Size			X			
Tributyltin	X	X[6]			X	X

Notes:

CRM = Certified Reference Material; RM = Reference Material; MS/MSD = matrix spike/matrix spike duplicate

[1] Frequency of Analysis (FOA) = 5 percent or one per batch, whichever is more frequent.

[2] Surrogate spikes required for every sample, including matrix spiked samples, blanks, and reference materials.

[3] Initial calibrations required before any samples are analyzed, after each major disruption of equipment, and when ongoing calibration fails to meet criteria.

[4] Ongoing calibration required at the beginning of each work shift, every 10–12 samples or every 12 hours (whichever is more frequent), and at the end of each shift.

[5] FOA = one per extraction batch.

[6] Matrix spike duplicates may be used.

[7] The Puget Sound Sediment Reference Material must be used for projects in Puget Sound.

Table A-3. Solid Phase Bioassay Performance Standards and Evaluation Guidelines

Bioassay	Negative Control Performance Standard	Reference Sediment Performance Standard	Dispersive Disposal Site Interpretation Guidelines		Nondispersive Disposal Site Interpretation Guidelines	
			1-hit rule	2-hit rule	1-hit rule	2-hit rule
Amphipod	$M_C \leq 10\%$	$M_R - M_C \leq 20\%$	$M_T - M_C > 20\%$ and M_T vs. M_R SS ($p=.05$) and		$M_T - M_C > 20\%$ and M_T vs. M_R SS ($p=.05$) and	
			$M_T - M_R > 10\%$	NOCN	$M_T - M_R > 30\%$	NOCN
Larval	$N_C \div I \geq 0.70$	$N_R \div N_C \geq 0.65$	$N_T \div N_C < 0.80$ and N_T / N_C vs. NR/ N_C SS ($p=.10$) and		$N_T \div N_C < 0.80$ and N_T / N_C vs. N_R / N_C SS ($p=.10$) and	
			$N_R / N_C - N_T / N_C > 0.15$	NOCN	$N_R / N_C - N_T / N_C > 0.30$	NOCN
Neanthes growth	$M_C \leq 10\%$ and $MIG_C > 0.38$	$M_R \leq 20\%$ and $MIG_R \div MIG_C \geq 0.80$	$MIG_T \div MIG_C < 0.80$ and MIG_T vs. MIG_R SS ($p=.05$) and		$MIG_T \div MIG_C < 0.80$ and MIG_T vs. MIG_R SS ($p=.05$) and	
			$MIG_T / MIG_R < 0.70$	NOCN	$MIG_T / MIG_R < 0.50$	$MIG_T / MIG_R < 0.70$

M = mortality
N = normal larvae
I = initial count
MIG = mean individual growth rate (mg/individual/day)
SS = statistically significant
NOCN = no other conditions necessary
NA = not applicable
Subscripts: R = reference sediment, C = negative control, T = test sediment

Sampling and Analysis Plan
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

Appendix B

Analytical Resources, Inc.
Sediment Reference Certificates

ERA
A Waters Company

Certificate of Analysis

Lot No. D079-540

Metals in Soil

Catalog No. 540
Issue Date: September 28, 2012
Revision Date: November 27, 2012

Certification

Parameter	Total Concentration [1] (mg/kg)	Certified Value [2] (mg/kg)	Uncertainty [3]	QC PALs™ [4] (mg/kg)	PT PALs™ [5] (mg/kg)
aluminum	68100*	9060	5.2%	4180 - 13900	3860 - 14200
antimony	246	106	7.6%	DL - 232	24.5 - 271
arsenic	215	182	1.9%	151 - 214	129 - 236
barium	561	143	3..5%	119 - 168	104 - 183
beryllium	111	98.3	2.1%	82.5 - 114	73 3 - 123
boron	134	106	2.5%	77.2 - 135	63.6 - 152
cadmium	71.2	60.4	8.4%	50.2 - 70.6	44.2 - 78.1
calcium	24800*	6040	2.8%	5020 - 7050	4450 - 7620
chromium	366	125	1.2%	102 - 147	87 2 - 162
cobalt	195	163	1.9%	138 - 188	121 - 204
copper	101	80.1	7.6%	67.1 - 93.0	59.0 - 104
iron	42900*	12900	4.6%	6550 - 19300	4170 - 21700
lead	148	136	4.2%	114 - 159	99.4 - 173
magnesium	8740	2640	0.2%	1990 - 3280	1690 - 3580
manganese	864	279	2.1%	231 - 328	207 - 352
mercury	9.80	9.25	1.7%	6.63 - 11.9	4.75 - 13.8
molybdenum	60.0	42.3	4.0%	31.8 - 52.7	27.6 - 63.7
nickel	172	128	2.3%	108 - 148	93.6 - 166
potassium	19700*	2820	0.8%	2050 - 3590	1750 - 3890
selenium	102	85.9	4.1%	68.7 - 103	54.9 - 117
silver	70.2	61.3	2.6%	40.6 - 82.0	41.0 - 81.6
sodium	17200*	439	2.9%	324 - 554	212 - 666
strontium	238	110	6.0%	89.4 - 131	77.6 - 143
thallium	171	144	6.9%	117 - 171	98.3 - 190
tin	186	157	3.9%	124 - 189	94.2 - 219
titanium	3280*	241	2.6%	29.4 - 453	0.00 - 559
vanadium	193	104	4.6%	78 5 - 129	68.6 - 139
zinc	232	204	5.6%	167 - 240	142 - 265

Please see footnotes on back

DL = Detection Limit

ISO/IEC GUIDE 34:2009
ACCREDITED
REFERENCE MATERIAL PRODUCER
CERTIFICATE NO. 1539.03

ISO/IEC 17025:2005
ACCREDITED
CHEMICAL TESTING LABORATORY
CERTIFICATE NO. 1539.02

16341 Table Mtn Pkwy, Golden, CO 80403 800-372-0122 fax: 303-421-0159 www.eraqc.com

ERA

A Waters Company

Instructions for Catalog # 540
Metals in Soil
Revision 030512

Description:
- This standard is packaged in a 2-ounce glass jar containing approximately 40 grams of soil.
- This standard is not preserved.
- The standard can be stored at room temperature.
- This product is intended to be used as a quality control check of the entire analytical process for the analytes/matrix included in the standard.
- ERA suggests that when subsampling this product prior to analysis you use a minimum sample size of at least 0.2 g. Using a smaller sample size may invalidate the assigned value and/or uncertainty shown on the certificate of analysis.

Helpful Hints:
- The Mercury in this standard should be determined using the digestion and analytical procedures in the current version of EPA method 7471, or equivalent.
- The other metals in this standard should be determined using EPA digestion methods 3050 or 3051 followed by your normal analysis procedures. Digesting the sample using another method may yield significantly different results.
- This standard should not be analyzed for Hexavalent Chromium. A separate standard, ERA catalog number 921, is available for Hexavalent Chromium.
- Although all ERA soil standards have been thoroughly blended prior to shipping, the standards should be homogenized prior to taking an aliquot for analysis due to settling which may occur during shipping.
- The percent moisture of this standard should be determined and your analytical results adjusted accordingly and reported on a dry weight basis.
- High recoveries of the metals native to the soil matrix (e.g. aluminum, calcium, iron, magnesium, potassium and titanium) indicate a digestion procedure which may be too vigorous. In general, it should be noted that the methods used to digest metals in soil samples are not extremely rugged and close attention should be paid to the procedure to ensure analyte recoveries which are consistent and within the PALs™.

Instructions:
1. Open the Metals in Soil standard in a fume hood to avoid inhalation of dust.
2. Mix the sample well prior to removing aliquots for analysis.
3. Digest and analyze the standard using your normal procedures.
4. Determine the percent moisture of an aliquot of the Metals in Soil standard.
5. Report your results as mg/kg on a dry weight basis.

Safety:
ERA products may be hazardous and are intended for use by professional laboratory personnel trained in the competent handling of such materials. Responsibility for the safe use of these products rests entirely with the buyer and/or user. Material Safety Data Sheets (MSDS) for all ERA products are available by calling 1-800-372-0122.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected polybrominated diphenyl ether (PBDE) congeners, selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle size characteristics. Information values are provided for selected polychlorinated naphthalenes (PCNs) and hexabromocyclododecanes (HBCDs). All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment.

Certified Mass Fraction Values: Certified values for mass fractions of PAHs, PCB congeners, chlorinated pesticides, and trace elements are provided in Tables 1 through 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST using two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Mass Fraction Values: Reference values are provided for mass fractions of additional PAHs (some in combination) in Tables 5 and 6, additional PCB congeners and chlorinated pesticides in Table 7, PBDE congeners in Table 8, and additional inorganic constituents in Tables 9 and 10. Reference values are provided in Table 11 for the 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle size characteristics are provided in Table 12 and 13. Reference values for total organic carbon and percent extractable mass are provided in Table 14. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

Information Mass Fraction Values: Information values are provided in Table 15 for mass fractions of additional trace elements, in Table 16 for PCN congeners (some in combination), and in Table 17 for HBCD isomers. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1].

Expiration of Certification: The certification of **SRM 1944** is valid, within the measurement uncertainties specified, until **31 March 2017**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 27 September 2011
Certificate Revision History on Page 20

Robert L. Watters, Jr., Chief
Measurement Services Division

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification was performed by M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh, M.G. Vangel, and M.S. Levenson of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The sediment was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD), and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R.R. Greenberg, J.M. Keller, J.R. Kucklick, M. Lopez de Alda, K.E. Murphy, R. Olfaz, B.J. Porter, D.L. Poster, L.C. Sander, P. Schubert, M.M. Schantz, S.S. Vander Pol, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX, USA). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL, USA). Additional results for PBDE congeners were used from ten laboratories (see Appendix A) that participated in an interlaboratory study specifically for PBDEs in Marine Sediment coordinated by H.M. Stapleton of the NIST Analytical Chemistry Division. M. LaGuardia of Virginia Institute of Marine Science (Gloucester Point, VA, USA) provided one set of measurements for the HBCDs.

Values for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among fourteen laboratories (see Appendix B) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, ON, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix C) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC; Ottawa, ON, Canada).

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of test portions for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains a mass fraction of approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis or, if the constituents of interest are volatile, a separate test portion of the sediment should be removed from the bottle at the time of analysis and dried to determine the mass fraction on a dry-mass basis.

PREPARATION AND ANALYSIS⁽¹⁾

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250 µm to 61 µm), homogenized in a cone blender, radiation sterilized at an estimated minimum dose of 32 kilograys (^{60}Co), and then packaged in screw-capped amber glass bottles.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material as received contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze drying test portions of 1.6 g to 2.5 g for five days at 1 Pa with a –10 °C shelf temperature and a –50 °C condenser temperature. The mass fraction of moisture in SRM 1944 at the time of the certification analyses was 1.25 % ± 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. Techniques and solvents involved were Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, clean up of the extracts using solid-phase extraction (SPE), or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on four stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, a proprietary non-polar polysiloxane phase, and a smectic liquid crystalline stationary phase.

Seven sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), GC/MS (VI), and GC/MS (Sm), were obtained using four columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate test portions of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. (All extraction and LC solvent compositions are expressed as volume fractions unless otherwise noted.) The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g test portions from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These test portions were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the test portions were extracted, processed, and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g test portions from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone, and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (VI) analyses, three test portions of 0.7 g from one bottle of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 20 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary non-polar polysiloxane phase (0.25 µm film thickness) (DB-XLB, J&W Scientific). For GC/MS (Sm) 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 24 h with 250 mL of DCM. The extracts were processed as described above for

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(1) Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Test portions of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through two aminopropylsilane SPE cartridges connected in series to obtain the total PAH fraction. A second 1 g test portion from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength-programmed fluorescence detection. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1944 was assessed by analyzing duplicate test portions of 1 g from eight bottles selected by stratified random sampling. Test portions were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g test portion size.

PAH Isomers of Molecular Mass 300 and 302: For the determination of the molecular mass 300 and 302 PAH isomers, three test portions of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substitued methylpolysiloxane phase (0.25 µm film thickness; DB-17MS, J&W Scientific, Folsom, CA). Perdeuterated dibenzo[*a,i*]pyrene was added to the sediment prior to extraction for use as an internal standard.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, clean up/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g test portions from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C18 phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Test portions of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the test portions were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g test portions from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six test portions were extracted using PFE. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [3]), and 4,4'-DDT-d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [4]. Results from nineteen laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Polybrominated Diphenyl Ethers: Value assignment of the concentrations of eight PBDE congeners was based on the means of results from two interlaboratory studies [5,6] and two sets of data from NIST. The laboratories participating in the interlaboratory exercises (see Appendix A) employed the analytical procedures routinely used in their laboratories to measure PBDEs. For the two methods used at NIST, six test portions (between 1 g and 2 g) were extracted using PFE at 100 °C with DCM. The extracts were cleaned up using an alumina column (5 % deactivated) SPE column. Size exclusion chromatography (SEC) on a divinylbenzene-polystyrene column (10 µm particle size, 10 nm (100 angstrom) pore size, 7.5 mm i.d. × 300 mm, PL-Gel, Polymer Labs, Inc.) was then used to remove the sulfur. The PBDEs, as well as PCBs and pesticides, were quantified using GC/MS in the electron impact mode on a 0.18 mm i.d. × 30 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). The PBDEs were also quantified using GC/MS in the negative chemical ionization mode on a 0.18 mm i.d. × 10 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). Selected Carbon-13 labeled PBDE and PCB congeners were added to the sediment prior to extraction for use as internal standards for quantification purposes.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta- substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by fourteen laboratories that participated in an interlaboratory comparison study (see Appendix B). Each laboratory analyzed three test portions (typically 1 g) of SRM 1944 using their routine analytical procedures and high resolution gas chromatography with high resolution mass spectrometry detection (GC-HRMS). The analytical procedures used by all of the laboratories included spiking with ^{13}C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225, J&W Scientific, Folsom, CA) capillary column.

Analytical Approach for Inorganic Constituents: Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven laboratories that participated in an interlaboratory comparison exercise coordinated by NRCC [7] (see Appendix C). The analytical methods used for the determination of each element are summarized in Table 18. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma optical emission spectroscopy (ICPOES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven laboratories that participated in the NRCC interlaboratory comparison exercise. The reference mass fraction values in Table 9 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPOES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 15 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [8]. Test portions (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb, ^{111}Cd, and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same test portions; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the $^{112}Cd/^{111}Cd$ and $^{62}Ni/^{60}Ni$ ratios, respectively. The $^{208}Pb/^{206}Pb$ ratios were measured directly because interferences at these masses are negligible.

NIST Analyses using INAA: Analyses were performed in two steps [9]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg test portions from each of ten bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 MW, which corresponds to a neutron fluence rate of about 8×10^{13} $cm^{-2} \cdot s^{-1}$. After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample-to-detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg test portion from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 MW. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

Homogeneity Assessment for Inorganic Constitutents: For some of the trace elements, most notably Cd, Fe, Pb, Rb, Sb, Sc, and Th, the variations among the test portions measured at NIST (between 0.3 g and 0.5 g) were larger than expected from the measurement process. Based on experience, it was concluded that there is some material inhomogeneity for trace elements in the test portions used. Sample variations among the NIST measurements are used as slightly conservative estimates of the sample inhomogeneities.

Particle Size Information: Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle-size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle size distribution are calculated. The system has a working range from 0.7 µm to 700 µm.

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for total organic carbon (TOC) using similar procedures. Briefly, test portions of approximately 200 mg were reacted with 6 mol/L hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed test portions from six bottles of SRM 1944. For the determination of percent extractable mass, six test portions of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

Polychlorinated Naphthalenes: Value assignment of PCN congener concentrations was accomplished by combining results from the analysis of SRM 1944 by six laboratories that participated in an interlaboratory comparison study (see Appendix D). Each laboratory analyzed three test portions (typically 1 g to 2 g) of SRM 1944 using their routine analytical procedures that included high-resolution gas chromatography with either high-resolution mass spectrometry detection (GC-HRMS) or low-resolution MS in the negative chemical ionization mode. Calibration mixtures included either Halowax mixtures with known volume fractions of individual congeners or individual PCN congeners.

HBCDs: Value assignment of the concentrations of three HBCD isomers was accomplished by combining results from the analysis of SRM 1944 in two sets from NIST and one set from Virginia Institute of Marine Science. For the two sets analyzed at NIST, the second fraction from an acidified silica SPE clean-up was analyzed by LC/MS/MS for the HBCDs using both electrospray ionization (ESI) and atmospheric pressurized photoionization (APPI). A C18 column (3.0 mm × 150 mm × 3.5 µm column, Eclipse Plus, Agilent Technologies) and YMC Carotenoid S5 C30 column (4.6 mm × 250 mm × 5 µm column) were used with a solvent gradient using 2.5 mmol/L ammonium acetate in 12.5 % water in methanol and acetonitrile at a flow rate of 0.3 mL/min. Carbon-13 labeled HBCDs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Table 1. Certified Mass Fraction Values for Selected PAHs in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (mg/kg)		
Phenanthrene(c,d,e,f,g)	5.27	±	0.22
Fluoranthene(c,d,e,f,g)	8.92	±	0.32
Pyrene(c,d,e,f,g)	9.70	±	0.42
Benzo[*c*]phenathrene(c,d,e,f,h)	0.76	±	0.10
Benz[*a*]anthracene(c,d,e,f,g,h)	4.72	±	0.11
Chrysene,(h,k)	4.86	±	0.10(i)
Triphenylene(h,k)	1.04	±	0.27
Benzo[*b*]fluoranthene(g,h,j)	3.87	±	0.42
Benzo[*j*]fluoranthene(h,j)	2.09	±	0.44
Benzo[*k*]fluoranthene(c,d,e,f,g,h,j)	2.30	±	0.20
Benzo[*a*]fluoranthene(c,d,e,f,h,,j)	0.78	±	0.12
Benzo[*e*]pyrene(c,d,e,f,h,j)	3.28	±	0.11
Benzo[*a*]pyrene(c,d,e,f,g,h,j)	4.30	±	0.13
Perylene(c,d,e,f,g,h,j)	1.17	±	0.24
Benzo[*ghi*]perylene(c,d,e,f,,j,k)	2.84	±	0.10
Indeno[1,2,3-*cd*]pyrene(c,d,e,f,j,k)	2.78	±	0.10
Dibenz[*a,j*]anthracene(c,d,e,f,,j,k)	0.500	±	0.044
Dibenz[*a,c*]anthracene(,j,k)	0.335	±	0.013
Dibenz[*a,h*]anthracene(j,k)	0.424	±	0.069
Pentaphene(c,d,e,f,,j,k)	0.288	±	0.026
Benzo[*b*]chrysene(c,d,e,f,j,k,h)	0.63	±	0.10
Picene(c,d,e,f,j,k)	0.518	±	0.093

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the Comité International des Poids et Mesures (CIPM) approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) Gas chromatography/mass spectrometry (GC/MS) (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(f) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

(g) LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(h) GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

(i) The uncertainty interval for chrysene was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(j) GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

(k) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 2. Certified Mass Fraction Values for Selected PCB Congeners(a) in SRM 1944 (Dry-Mass Basis)

		Mass Fraction(b,c) (µg/kg)		
PCB 8	(2,4'-Dichlorobiphenyl)(d,e,f,g,h,i,j,k)	22.3	±	2.3
PCB 18	(2,2',5-Trichlorobiphenyl)(d,e,f,g,h,i,j,k)	51.0	±	2.6
PCB 28	(2,4,4'-Trichlorobiphenyl)(d,e,f,g,j,k)	80.8	±	2.7
PCB 31	(2,4',5-Trichlorobiphenyl)(d,e,f,g,j)	78.7	±	1.6(l)
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	60.2	±	2.0
PCB 49	(2,2'4,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	53.0	±	1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	79.4	±	2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)(e,g,h,i,j)	71.9	±	4.3
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)(e,g,h,i,j)	65.0	±	8.9
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)(d,e,f,g,h,i,j)	29.9	±	4.3
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	37.5	±	2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	73.4	±	2.5
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)(e,f,g,h,i,j,k)	24.5	±	1.1
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)(g,h,i,j)	63.5	±	4.7
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	58.0	±	4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	8.47	±	0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	62.1	±	3.0
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	49.7	±	1.2
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	16.93	±	0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	74.0	±	2.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)(d,e,f,g,h,i,j)	6.52	±	0.66
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	22.6	±	1.4
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	44.3	±	1.2
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)(d,e,f,g,h,i,j)	12.19	±	0.57
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	25.1	±	1.0
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)(d,e,f,g,h,i,j)	11.2	±	1.4
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)(d,e,f,g,h,i,j,k)	3.75	±	0.39
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)(d,e,f,g,h,i,j,k)	9.21	±	0.51
PCB 209	Decachlorobiphenyl(d,e,f,g,h,i,j,k)	6.81	±	0.33

(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(d) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(f). GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(g) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(h) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(i) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(j) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(k) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

(l) The uncertainty interval for PCB 31 was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (µg/kg)
Hexachlorobenzene(e,f,g,h,i,j)	6.03 ± 0.35
cis-Chlordane (α-Chlordane)(c,d,e,f,g,h,i,j)	16.51 ± 0.83
trans-Nonachlor (c,d,e,f,g,h,i,j)	8.20 ± 0.51

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(e) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(g) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(j) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fractions(a,b) (%)
Aluminum(c,d,e)	4	5.33 ± 0.49
Iron(c,d,e)	6	3.53 ± 0.16
		Mass Fractions(a,b) (mg/kg)
Arsenic(c,d,e,f,g)	10	18.9 ± 2.8
Cadmium(c,f,,h,i)	6	8.8 ± 1.4
Chromium(c,d,f,g,i)	9	266 ± 24
Lead(c,h,i)	5	330 ± 48
Manganese(c,d,e)	8	505 ± 25
Nickel(c,g,h,i)	6	76.1 ± 5.6
Zinc(c,d,e,g,i)	9	656 ± 75

(a) The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NIST using INAA.

(e) Measured at NRCC using ICPOES.

(f) Measured at NRCC using GFAAS.

(g) Measured at IAEA using INAA.

(h) Measured at NIST using ID-ICPMS.

(i) Measured at NRCC using ID-ICPMS.

Table 5. Reference Mass Fraction Values for Selected PAHs in SRM 1944

	Mass Fractions(a) (mg/kg)		
Naphthalene(b)	1.28	±	0.04(c)
1-Methylnaphthalene(b)	0.47	±	0.02(c)
2-Methylnaphthalene(b)	0.74	±	0.06(c)
Biphenyl(b)	0.25	±	0.02(c)
Acenaphthene(b)	0.39	±	0.03(c)
Fluorene(b)	0.48	±	0.04(c)
Dibenzothiophene(b)	0.50	±	0.03(c)
Anthracene(b)	1.13	±	0.07(c)
1-Methylphenanthrene(d,e,f,g)	1.7	±	0.1(h)
2-Methylphenanthrene(d,e,f,g)	1.90	±	0.06(h)
3-Methylphenanthrene(d,e,f,g)	2.1	±	0.1(h)
4-Methylphenanthrene and 9-Methylphenanthrene(d,e,f,g)	1.6	±	0.2(h)
2-Methylanthracene(d,e,f,g)	0.58	±	0.04(h)
3,5-Dimethylphenanthrene(d)	1.31	±	0.04(h)
2,6-Dimethylphenanthrene(d)	0.79	±	0.02(h,i)
2,7-Dimethylphenanthrene(d)	0.67	±	0.02(h,i)
3,9-Dimethylphenanthrene(d)	2.42	±	0.05(h,i)
1,6-, 2,9-, and 2,5-Dimethylphenanthrene(d)	1.67	±	0.03(h,i)
1,7-Dimethylphenanthrene(d)	0.62	±	0.02(h,i)
1,9- and 4,9-Dimethylphenanthrene(d)	1.20	±	0.03(h,i)
1,8-Dimethylphenanthrene(d)	0.24	±	0.01(h,i)
1,2-Dimethylphenanthrene(d)	0.28	±	0.01(h,i)
8-Methylfluoranthene(d)	0.86	±	0.02(h,i)
7-Methylfluoranthene(d)	0.69	±	0.02(h)
1-Methylfluoranthene(b)	0.39	±	0.01(c)
3-Methylfluoranthene(b)	0.56	±	0.02(c)
2-Methylpyrene(d)	1.81	±	0.04(h,i)
4-Methylpyrene(d)	1.44	±	0.03(h,i)
1-Methylpyrene(d)	1.29	±	0.03(h)
Anthanthrene(j)	0.9	±	0.1(h)

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) GC/MS (VI) on proprietary non-polar methylpolysiloxane phase after Soxhlet extraction with DCM.

(c) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, *U*, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, *k*, is determined from the Student’s *t*-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(d) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(g) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

(h) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(i) The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(j) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 6. Reference Mass Fractions for Selected PAHs of Relative Molecular Mass 300 and 302 in SRM 1944 (Dry-Mass Basis)

	Mass Fraction[(a,b,c)] (mg/kg)		
Coronene	0.53	±	0.04
Dibenzo[*b*,*e*]fluoranthene	0.076	±	0.008
Naphtho[1,2-*b*]fluoranthene	0.70	±	0.06
Naphtho[1,2-*k*]fluoranthene and Naphtho[2,3-*j*]fluoranthene	0.66	±	0.05
Naphtho[2,3-*b*]fluoranthene	0.21	±	0.01
Dibenzo[*b*,*k*]fluoranthene	0.75	±	0.06
Dibenzo[*a*,*k*]fluoranthene	0.22	±	0.02
Dibenzo[*j*,*l*]fluoranthene	0.56	±	0.03
Dibenzo[*a*,*l*]pyrene	0.12	±	0.02
Naphtho[2,3-*k*]fluoranthene	0.11	±	0.01
Naphtho[2,3-*e*]pyrene	0.33	±	0.02
Dibenzo[*a*,*e*]pyrene	0.67	±	0.05
Naphtho[2,1-*a*]pyrene	0.76	±	0.05
Dibenzo[*e*,*l*]pyrene	0.28	±	0.02
Naphtho[2,3-*a*]pyrene	0.23	±	0.01
Benzo[*b*]perylene	0.43	±	0.04
Dibenzo[*a*,*i*]pyrene	0.30	±	0.03
Dibenzo[*a*,*h*]pyrene	0.11	±	0.01

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's *t*-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(c) GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

Table 7. Reference Mass Fractions for Selected PCB Congeners(a) and Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(b) (µg/kg)		
PCB 45 (2,2’,3,6-Tetrachlorobiphenyl)(c)	10.8	±	1.4(d)
PCB 146 (2,2',3,4’,5,5'-Hexachlorobiphenyl)(c)	10.1	±	1.9(d)
PCB 163 (2,3,3’,4’,5,6-Hexachlorobiphenyl)(c)	14.4	±	2.0(d)
PCB 174 (2,2’,3,3’,4,5,6’-Heptachlorobiphenyl)(c)	16.0	±	0.6(d)
α-HCH(f,g,h,i)	2.0	±	0.3(e)
trans-Chlordane (γ-Chlordane)(c)	19.0	±	1.7(d)
cis-Nonachlor(g,h,i,l,m)	3.7	±	0.7(e)
2,4'-DDE(f,g,h,i,j,k,l,m)	19	±	3(e)
2,4'-DDD(h,j,k,l,m)	38	±	8(e)
4,4'-DDE(f,g,h,ihj,k,l,m)	86	±	12(e)
4,4'-DDD (f,g,h,I,j,k,l,m)	108	±	16(e)
4,4’-DDT(c)	170	±	32(d)

(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) NIST participation in the 2007 interlaboratory study using GC/MS.

(d) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student’s t-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(e) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or he mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(f) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(g) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(h) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(i) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(j) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(k) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(l) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts anlayzed as in GC-ECD (IIA).

(m) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 8. Reference Mass Fraction Values for Selected PBDEs in SRM 1944 (Dry-Mass Basis)

	Mass Fractions(a) (µg/kg)		
PBDE 47 (2,2’,4,4’-Tetrabromodiphenyl ether)(c,d,e,f)	1.72	±	0.28(b)
PBDE 99 (2,2',4,4’,5-Pentabromodiphenyl ether)(c,d,f)	1.98	±	0.26(b)
PBDE 100 (2,2',4,4’,6-Pentabromodiphenyl ether)(c,d)	0.447	±	0.027(b)
PBDE 153 (2,2',4,4’,5,5'-Hexabromodiphenyl ether)(c,d,e,f)	6.44	±	0.37(b)
PBDE 154 (2,2',4,4',5,6’-Hexabromodiphenyl ether)(c,d,f)	1.06	±	0.08(b)
PBDE 183 (2,2',3,4,4’,5',6-Heptabromodiphenyl ether)(c,d,e,f)	31.8	±	0.1(b)
PBDE 206 (2,2',3,3’,4,4',5,5',6-Nonabromodiphenyl ether)(d,e)	6.2	±	1.0(b)
PBDE 209 (Decabromodiphenyl ether) (c,d,e,f)	93.5	±	4.4(b)

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Reference values are weighted means of the results from two to four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [11,12].

(c) Results from ten laboratories participating in an interlaboratory study for PBDEs in sediment [12].

(d) Results from four laboratories participating in the 2007 interlaboratory study [13].

(e) NIST participation in the 2007 interlaboratory study using GC/MS.

(f) Data set from NIST for PBDEs using GC/MS following PFE with alumina SPE and SEC clean-up.

Table 9. Reference Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fraction(a,b) (%)		
Silicon[c,d]	81	31	±	3
		Mass Fraction(a,b) (mg/kg)		
Antimony(c,e,f,g)	18	4.6	±	0.9
Beryllium(c,h)	17	1.6	±	0.3
Copper(c,d,f)	101	380	±	40
Mercury(c,i)	18	3.4	±	0.5
Selenium(c,e,f)	24	1.4	±	0.2
Silver(c,d,e,g)	8	6.4	±	1.7
Thallium(c,f)	12	0.59	±	0.1
Tin(c,f)	22	42	±	6

(a) The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the uncertainty in the value. Here u_c accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NRCC using GFAAS.

(e) Measured at NIST using INAA.

(f) Measured at NRCC using ID-ICPMS.

(g) Measured at IAEA using INAA.

(h) Measured at NRCC using ICPOES.

(i) Measured at NRCC using cold vapor atomic absorption spectroscopy (CVAAS).

Table 10. Reference Mass Fraction Values for Elements in SRM 1944 as Determined by INAA (Dry-Mass Basis)

	Effective Degrees of Freedom	Mass Fraction(a,b) (%)		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1
		Mass Fraction(a,b) (mg/kg)		
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

(a) The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, the results should be used with caution. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the uncertainty in the value. Here u_c accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 11. Reference Mass Fraction Values for Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (µg/kg)		
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	0.80	±	0.07
Octachlorodibenzo-*p*-dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran(c)	0.039	±	0.015(d)
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006(e)
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006(e)
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ)(f)	0.25	±	0.01
Total Tetrachlorodibenzo-*p*-dioxins	0.25	±	0.05(e)
Total Pentachlorodibenzo-*p*-dioxins	0.19	±	0.06
Total Hexachlorodibenzo-*p*-dioxins	0.63	±	0.09
Total Heptachlorodibenzo-*p*-dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo-*p*-dioxins(g)	8.7	±	0.9
Total Dibenzofurans(g)	5.0	±	0.5

(a) Each reference value is the mean of the results from up to fourteen laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [11,12] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor, k, is the value from a Student's t-distribution for a 95 % confidence interval.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

(d) Degrees of freedom = 7 for this compound.

(e) Degrees of freedom = 12 for this compound.

(f) TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [15]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

(g) Total of tetra- through octachlorinated congeners.

Table 12. Reference Values for Particle Size Characteristics for SRM 1944

Particle Measurement	Value[(a)]
Mean diameter (volume distribution, MV, μm)[(b)]	151.2 ± 0.4
Mean diameter (area distribution, μm)[(c)]	120.4 ± 0.1
Mean diameter (number distribution, μm)[(d)]	75.7 ± 0.3
Surface Area (m^2/cm^3)[(e)]	0.050 ± 0.013

[(a)] The reference value is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[(b)] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

[(c)] The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

[(d)] The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

[(e)] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

Table 13. Percentage of the Volume That is Smaller Than the Indicated Size

Percentile	Particle Diameter[(a)] (μm)
95	296 ± 5
90	247 ± 2
80	201 ± 1
70	174 ± 1
60	152 ± 1
50[(b)]	135 ± 1
40	120 ± 1
30	106 ± 1
20	91 ± 1
10	74 ± 1

[(a)] The reference value for particle diameter is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[(b)] Median diameter (50 % of the volume is less than 135 μm).

Table 14. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

	Mass Fraction (%)		
Total Organic Carbon (TOC)[a,b]	4.4	±	0.3
Extractable Mass[c,d]	1.15	±	0.04

[a] Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[c] Extractable mass as determined from Soxhlet extraction using DCM.

[d] The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 15. Information Mass Fraction Values for Selected Elements in SRM 1944 as Determined by INAA (Dry-Mass Basis)

	Mass Fraction[a] (%)
Magnesium[b]	1.0

	Mass Fraction[a] (mg/kg)
Cerium[b]	65
Europium[b]	1.3
Gold[b]	0.10
Lanthanum[b]	39
Thorium[b]	13
Uranium[b]	3.1

[a] Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] Measured at IAEA using INAA

Table 16. Information Mass Fraction Values for Selected Polychlorinated Naphthalenes in SRM 1944 (Dry-Mass Basis)

			Mass Fraction[(a)] (μg/kg)
PCN	19	(1,3,5-Trichloronaphthalene)	1.4
PCN	23	(1,4,5-Trichloronaphthalene)	2.4
PCN	42	(1,3,5,7-Tetrachloronaphthalene)	2.7
PCN	47	(1,4,6,7-Tetrachloronaphthalene)	3.5
PCN	52	(1,2,3,5,7-Pentachloronaphthalene)	2.5
	60	(1,2,4,6,7-Pentachloronaphthalene)	
PCN	50	(1,2,3,4,6-Pentachloronaphthalene)	1.0
PCN	66	(1,2,3,4,6,7-Hexachloronaphthalene)	0.63
	67	(1,2,3,5,6,7-Hexachloronaphthalene)	
PCN	69	(1,2,3,5,7,8-Hexachloronaphthalene)	1.6
PCN	73	(1,2,3,4,5,6,7-Heptachloronaphthalene)	0.51
PCN	75	(Octachloronaphthalene)	0.20

(a) Mass fractions reported on dry-mass basis; material as received contains approximately 1.3 % moisture. Information values are the median of the results from six laboratories participating in an interlaboratory comparison exercise (Appendix D).

Table 17. Information Mass Fraction Values for Three HBCD Isomers in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (μg/kg)
alpha-HBCD(b)	2.2
beta-HBCD(b)	1.0
gamma-HBCD(b)	18

(a) The information value is the median of the results from three analytical methods.
(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 18. Analytical Methods Used for the Measurement of Elements in SRM 1944

Elements	**Analytical Methods**
Aluminum	FAAS, ICPOES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPOES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPOES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPOES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPOES, ICPMS, ID-ICPMS,
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPOES, ICPMS, ID-ICPMS, XRF, INAA

Methods

CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPOES	Inductively coupled plasma optical emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

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Certificate Revision History: 27 September 2011 (Addition of mass fraction values for PBDE and PCN congeners; change of mass fraction reference values; editorial changes); **22 December 2008** (Extension of certification period); **14 May 1999** (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of PBDEs in SRM 1944 [4].

D. Hoover and C. Hamilton, AXYS Analytical, Sidney, BC, Canada
S. Klosterhaus and J. Baker, Chesapeake Biological Laboratory, Solomons, MD, USA
S. Backus, Environment Canada, Ecosystem Health Division, Burlington, ON, Canada
E. Sverko, Environment Canada, Canada Centre for Inland Waters, Burlington, ON, Canada
P. Lepom, Federal Environmental Agency, Berlin, Germany
R. Hites and L. Zhu, Indiana University, Bloomington, IN, USA
G. Jiang, Research Center for Eco-Environmental Sciences, Beijing, China
H. Takada, Tokyo University of Agriculture and Technology, Tokyo, Japan
A. Covaci and S. Vorspoels, University of Antwerp, Antwerp, Belgium
A. Li, Universtiy of Illinois at Chicago, Chicago, IL, USA

APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA, USA
L. Phillips, AXYS Analytical Services Ltd., Sidney, British Columbia, Canada
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH, USA
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada
C. Brochu, Environment Québec, Laval, Québec, Canada
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden
C. Lastoria, Maxxam Analytics Inc., Mississauga, Ontario, Canada
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
T.L. Wade, Texas A&M University, College Station, TX, USA
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH, USA

APPENDIX C

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abbgy, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA, USA
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia
H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA, USA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA, USA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX, USA
K. Elrick, U.S. Geological Survey, Atlanta, GA, USA

APPENDIX D

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated naphthalenes in SRM 1944.

J. Kucklick, National Institute of Standards and Technology, Charleston, SC, USA
E. Sverko, Environment Canada, Canada Centre for Inland Waters, Burlington, ON, Canada
P. Helm, Ontario Ministry of the Environment, Etobicoke, ON, Canada
N. Yamashita, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan
T. Harner, Environment Canada, Meteorological Service of Canada, Toronto, ON, Canada
R. Lega, Ontario Ministry of the Environment, Etobicoke, ON, Canada

Certificate of Analysis

Certified Reference Material

Fluka Analytical

BNAs - Sandy Loam

Number CRM143-50G
Lot LRAA1235
Solvent (Matrix) Sandy Loam Soil
Hazard Irritant
Storage &Handling Store at 4ºC.
Expiration Date See Sample Label
Certification Date: April 02, 2013
Certified By: Christopher Rucinski - QA Director

Analyte	*Units*	*Certified Value* [1,4]	*k*[5]	*Standard Deviation* [2]	*Confidence Interval*	*Prediction Interval*
1,2-Dichlorobenzene	µg/Kg	5,410 ± 578	1.96	1,570	4,810 - 6,010	2,270 - 8,550
1,4-Dichlorobenzene	µg/Kg	1,460 ± 181	1.96	477	1,280 - 1,640	506 - 2,410
Hexachlorobutadiene	µg/Kg	8,340 ± 833	1.96	2,340	7,570 - 9,110	3,680 - 13,000
Naphthalene	µg/Kg	3,250 ± 327	1.96	922	2,930 - 3,560	1,410 - 5,080
Acenaphthene	µg/Kg	5,360 ± 404	1.96	1,150	4,980 - 5,740	3,060 - 7,650
Acenaphthylene	µg/Kg	3,790 ± 326	1.96	932	3,470 - 4,110	1,930 - 5,640
Anthracene	µg/Kg	3,160 ± 205	1.96	579	2,950 - 3,370	2,000 - 4,310
Benzo(a)anthracene	µg/Kg	1,190 ± 82.6	1.96	231	1,110 - 1,270	729 - 1,650
Benzo(a)pyrene	µg/Kg	2,950 ± 190	1.96	538	2,770 - 3,130	1,880 - 4,020
Benzo(b)fluoranthene	µg/Kg	5,600 ± 410	1.96	1,150	5,200 - 6,010	3,310 - 7,890
Benzo(g,h,i)perylene	µg/Kg	994 ± 97.9	1.96	272	900 - 1,090	453 - 1,530
Benzo(k)fluoranthene	µg/Kg	4,400 ± 347	1.96	991	4,050 - 4,740	2,430 - 6,370
Benzoic acid	µg/Kg	2,230 ± 1160	2.18	1,500	1,150 - 3,300	0.00 - 5,670
4-Bromophenyl phenyl ether	µg/Kg	5,840 ± 456	1.96	1,220	5,410 - 6,280	3,420 - 8,270
Butyl benzyl phthalate	µg/Kg	8,370 ± 577	1.96	1,540	7,800 - 8,930	5,300 - 11,400
4-Chloro-3-methylphenol	µg/Kg	5,920 ± 491	1.96	1,360	5,440 - 6,400	3,210 - 8,630
bis(2-Chloroisopropyl) ether	µg/Kg	1,800 ± 221	1.96	561	1,580 - 2,020	674 - 2,920
2-Chlorophenol	µg/Kg	5,960 ± 590	1.96	1,640	5,390 - 6,520	2,700 - 9,210
4-Chlorophenyl phenylether	µg/Kg	3,940 ± 321	1.96	857	3,620 - 4,250	2,230 - 5,640
Chrysene	µg/Kg	6,730 ± 448	1.96	1,270	6,300 - 7,170	4,200 - 9,270
Dibenzo(a,h)anthracene	µg/Kg	1,350 ± 112	1.96	305	1,230 - 1,460	738 - 1,950
Dibenzofuran	µg/Kg	7,610 ± 535	1.96	1,440	7,060 - 8,160	4,730 - 10,500
2,4-Dichlorophenol	µg/Kg	9,210 ± 821	1.96	2,270	8,390 - 10,000	4,680 - 13,700
bis(2-Ethylhexyl) phthalate (DEHP)	µg/Kg	7,650 ± 500	1.96	1,380	7,160 - 8,140	4,910 - 10,400

Analyte	*Units*	*Certified Value*[1,4]	*k*[5]	*Standard Deviation*[2]	*Confidence Interval*	*Prediction Interval*
Diethyl phthalate	µg/Kg	8,010 ± 569	1.96	1,540	7,410 - 8,610	4,940 - 11,100
2,4-Dimethylphenol	µg/Kg	5,800 ± 459	1.96	1,240	5,340 - 6,260	3,330 - 8,270
2,4-Dinitrophenol	µg/Kg	6,180 ± 1520	2.05	3,550	4,630 - 7,730	0.00 - 13,600
2,6-Dinitrotoluene (2,6-DNT)	µg/Kg	4,400 ± 376	1.96	986	4,020 - 4,780	2,430 - 6,370
Di-n-octyl phthalate	µg/Kg	7,910 ± 673	1.96	1,830	7,250 - 8,570	4,260 - 11,600
Fluoranthene	µg/Kg	4,990 ± 338	1.96	949	4,660 - 5,320	3,100 - 6,880
Fluorene	µg/Kg	4,610 ± 344	1.96	981	4,270 - 4,950	2,660 - 6,570
Indeno(1,2,3-cd) pyrene	µg/Kg	4,600 ± 357	1.96	1,010	4,260 - 4,950	2,590 - 6,620
Isophorone	µg/Kg	9,870 ± 993	1.96	2,710	8,880 - 10,900	4,470 - 15,300
2-Methyl-4,6-dinitrophenol	µg/Kg	8,340 ± 1210	1.96	3,080	7,180 - 9,500	2,190 - 14,500
2-Methylnaphthalene	µg/Kg	2,320 ± 221	1.96	632	2,110 - 2,540	1,060 - 3,580
4-Methylphenol (p-Cresol)	µg/Kg	5,150 ± 580	2.09	2,010	4,060 - 6,230	815 - 9,480
3+4-Methylphenol (m+p-Cresol)	µg/Kg	5,030 ± 579	2.07	1,140	4,470 - 5,600	2,590 - 7,480
2-Nitrophenol	µg/Kg	6,860 ± 618	1.96	1,680	6,260 - 7,450	3,510 - 10,200
4-Nitrophenol	µg/Kg	6,800 ± 889	1.96	2,390	5,930 - 7,660	2,030 - 11,600
n-Nitrosodiphenylamine	µg/Kg	6,420 ± 885	2.05	2,060	5,630 - 7,210	2,120 - 10,700
Pentachlorophenol	µg/Kg	4,210 ± 372	1.96	995	3,840 - 4,570	2,220 - 6,190
Phenanthrene	µg/Kg	1,910 ± 140	1.96	400	1,780 - 2,050	1,120 - 2,710
Phenol	µg/Kg	4,230 ± 385	1.96	1,100	3,850 - 4,610	2,040 - 6,420
Pyrene	µg/Kg	8,360 ± 543	1.96	1,540	7,820 - 8,890	5,290 - 11,400
2,4,5-Trichlorophenol	µg/Kg	7,430 ± 591	1.96	1,610	6,890 - 7,960	4,230 - 10,600
2,4,6-Trichlorophenol	µg/Kg	9,010 ± 628	1.96	1,730	8,400 - 9,620	5,570 - 12,400

Additional Information

Sample Description

The sample size provided as a pack of 5 x 10g units of soil.

The soil has been sterilized to minimize degradation of the sample.

The sample has been sized to 100 mesh.

The sample has been intentionally prepared with an apparent headspace.

USEPA Method 8270C was the primary method for certification (GC-MS). Contact RTC for further method details.

Storage

Certificate of Analysis

Certified Reference Material

BNAs - Sandy Loam

Number **CRM143-50G**
Lot LRAA1235
Solvent (Matrix) Sandy Loam Soil
Hazard Irritant
Storage &Handling Store at 4ºC.
Expiration Date See Sample Label
Certification Date: April 02, 2013
Certified By: Christopher Rucinski - QA Director

Storage

The sample should be stored at 4ºC. It has been determined to be stable for the duration of the expiration date.

After sub-sampling replace cap securely and store remaining sample at 4ºC.

The shelf life of the product was determined by historic stability of similar CRM's. The expiration date may be extended based on stock and popularity upon successful stability testing by a 17025 accredited laboratory.

Stability and shelf life after opening must be determined by the user, taking into account sampling frequency/volume and all local conditions.

Recommended Preparation

Extract an accurately weighed portion (recommended minimum sample is 10 grams) using SW846 Method 3540C, Soxhlet Extraction; 3541, Automated Soxhlet Extraction; 3550, Ultrasonic Extraction or other technique identified by the method to be acceptable for the analytes of interest.

In addition to the solvent systems listed in Method 3540C,the methylene chloride/acetone (1:1 v/v) system is acceptable.

Note: Sample extracts and calibration solutions should be in the same solvent.

Transfer the entire amount of one vial to your extraction system. Rinse the vial with a 2-5 mL your extraction solvent. Assume 10g for the sampling size. Smaller amounts may be sampled but RTC does not maintain homogeneity for sample sizes less than 10g.

Results based on as provided basis assume each vial contains 10g of dry soil.

Scope and Application

The Base Neutral Acid (BNA) Compounds in Soil Certified Reference Material (CRM) consists of four amber glass sample jar, with a Teflon lined closure containing approximately 10 grams of soil, fortified with 49 semi-volatile organics. Being a natural matrix waste sample the analyst is challenged by the same preparation problems, analytical interferences, etc. as is typical for similar matrices received by the laboratory for analysis. Rigorous analysis identified, quantified, and certified various aliphatic and aromatic banding which are listed on the enclosed Certificate of Analysis. The sample has been analyzed by 41 independent laboratories in a round-robin to meet the requirements specified by the ISO Guides 34 and 35, and ISO 17025.

Evaluation of Results

The Reference Value, 95% confidence interval(C.I.) for the Reference Value and 95% Prediction Interval (P.I.) around the Reference Value were obtained by the methods identified in the 'Scope and Application' section of this Certificate of Analysis. Samples were selected in a random fashion from the beginning to the end of the bottling sequence and sent for analysis by an independent laboratory round-robin. The data produced in the round-robin was used to calculate reference values by the USEPA EMSL-CINN's computer program “BIWEIGHT”.

The generated BIWEIGHT mean, BIWEIGHT standard deviation and BIWEIGHT standard deviation of the mean are used to calculate the 95% Confidence Interval (CI) for the mean and the 95% Prediction Interval (PI). For normally distributed data, the BIWEIGHT 95% CI compares well to the classical calculation method used to generate a 95% CI. For non-Gaussian data sets, the BIWEIGHT method is more robust in data treatment.

BIWEIGHT data are also used to calculate a 95% PI. The 95% PI compares well to a 95% tolerance limit calculated using classical methods. For normally distributed data, the BIWEIGHT 95% PI typically represents approximately a ±2 BIWEIGHT standard deviation window around the BIWEIGHT mean. Again, the BIWEIGHT method is more robust than classical methods when handling non-Gaussian data sets.

Laboratories performing the same analytical procedures on a sample whose values have been determined by the BIWEIGHT method can assume that the true mean, as determined by the method, is within the 95% CI window. Laboratories analyzing the sample should have results within the 95% PI window 19 out of 20 analyses. Laboratories should use the PI as guidance for laboratory performance.

Additional information on the program may be obtained by referring to the reference or by downloading the program from the EMSL-CINN web site. Additionally contact RTC for additional guidance - 1(307)742-5452 - support@rt-corp.com - www.rt-corp.com

Health and Safety Information

All RTC Certified Reference Materials are intended only for professional use by properly trained laboratory personnel. This CRM has been reviewed for both health and safety and shipping risks. It is classified as non hazardous and is not classified as hazardous goods for shipping by road, sea or air transport.

Health and Safety Information

A full international MSDS as a downloadable pdf file is available at www.rt-corp.com

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1 Certified values are the robust statisitical mean when prepared according to instructions from an Interlaboratory Study and internal rigorous testing.
2 The standard deviation is the robust statistical standard deviation from the round robin interlaboratory study.
4 Expanded Uncertainty (Ucrm) - All uncertainty values in this document expressed as ± *value* are expanded uncertainties.
5 ***k*:** Coverage factor derived from a t-distribution table, based on the degrees of freedom of the data set. **Confidence interval = 95%**

TRACEABILITY: The standard was manufactured under an ISO 17025 certified quality system. The balance used to weigh raw materials is accurate to +/- 0.0001g and calibrated regularly using mass standards traceable to NIST. All dilutions were preformed gravimetrically. Additionally, individual analytes are traceable to NIST SRMs where available and specified above.

HOMOGENEITY ASSESSMENT: Between-bottle homogeneity was assessed in accordance with ISO Guide 35. Completed units were sampled over the course of the bottling operation. Samples were taken in the following manner: the units produced in the bottling operation were divided into three chronological groups, those from the Early third, the Middle third, and the Late third (Groups). A pre-determined number of sample units were then randomly selected from each group. A subset of each group was then randomly selected for chemical analysis. The results of the chemical analysis were then compared by Single Factor Analysis of Variance (ANOVA).

UNCERTAINTY STATEMENT: Uncertainty values in this document are expressed as Expanded Uncertainty (Ucrm) corresponding to the 95% confidence interval. Ucrm is derived from the combined standard uncertainty multiplied by the coverage factor k, which is obtained from a *t*-distribution and degrees of freedom. The components of combined standard uncertainty include the uncertainties due to characterization, homogeneity, long term stability, and short term stability (transport). The components due to stability are generally considered to be negligible unless otherwise indicated by stability studies.

THIS PRODUCT WAS DESIGNED, PRODUCED AND VERIFIED FOR ACCURACY AND STABILITY IN ACCORDANCE WITH ISO 17025 (AClass Cert AT-1467) and ISO GUIDE 34 (AClass Cert AR-1470).

MSDS reports for components comprising greater than 1.0% of the solution or 0.1% for components known to be carcinogens are available upon request.

Manufactured and certified by Sigma-Aldrich RTC, Inc.

305 - 61

5410	2270	8550	1,2-Dichlorobenzene	42.0%	158.0%
1460	506	2410	1,4-Dichlorobenzene	34.7%	165.1%
8340	3680	13000	Hexachlorobutadiene	44.1%	155.9%
3250	1410	5080	Naphthalene	43.4%	156.3%
5360	3060	7650	Acenaphthene	57.1%	142.7%
3790	1930	5640	Acenaphthylene	50.9%	148.8%
3160	2000	4310	Anthracene	63.3%	136.4%
1190	729	1650	Benzo(a)anthracene	61.3%	138.7%
2950	1880	4020	Benzo(a)pyrene	63.7%	136.3%
5900	3310	7890	Benzo(b)fluoranthene	56.1%	133.7%
994	453	1530	Benzo(g,h,i)perylene	45.6%	153.9%
4400	2430	6370	Benzo(k)fluoranthene	55.2%	144.8%
2230	0	5670	Benzoic acid	0.0%	254.3%
5840	3420	8270	4-Bromophenyl phenyl ether	58.6%	141.6%
8370	5300	11400	Butyl benzyl phthalate	63.3%	136.2%
5920	3210	8630	4-Chloro-3-methylphenol	54.2%	145.8%
1800	674	2920	bis(2-Chloroisopropyl) ether	37.4%	162.2%
5960	2700	9210	2-Chlorophenol	45.3%	154.5%
3940	2230	5640	4-Chlorophenyl phenylether	56.6%	143.1%
6730	4200	9270	Chrysene	62.4%	137.7%
1350	738	1950	Dibenzo(a,h)anthracene	54.7%	144.4%
7610	4730	10500	Dibenzofuran	62.2%	138.0%
9210	4680	13700	2,4-Dichlorophenol	50.8%	148.8%
7650	4910	10400	bis(2-Ethylhexyl) phthalate (DEHP)	64.2%	135.9%
8010	4940	11100	Diethyl phthalate	61.7%	138.6%
5800	3330	8270	2,4-Dimethylphenol	57.4%	142.6%
6180	0	13600	2,4-Dinitrophenol	0.0%	220.1%
4400	2430	6370	2,6-Dinitrotoluene (2,6-DNT)	55.2%	144.8%
7910	4260	11600	Di-n-octyl phthalate	53.9%	146.6%
4990	3100	6880	Fluoranthene	62.1%	137.9%
4610	2660	6570	Fluorene	57.7%	142.5%
4600	2590	6620	Indeno(1,2,3-cd) pyrene	56.3%	143.9%
9870	4470	15300	Isophorone	45.3%	155.0%
8340	2190	14500	2-Methyl-4,6-dinitrophenol	26.3%	173.9%
2320	1060	3580	2-Methylnaphthalene	45.7%	154.3%
5150	815	9480	4-Methylphenol (p-Cresol)	15.8%	184.1%
5030	2590	7480	3+4-Methylphenol (m+p-Cresol)	51.5%	148.7%
6860	3510	10200	2-Nitrophenol	51.2%	148.7%
6800	2030	11600	4-Nitrophenol	29.9%	170.6%
6420	2120	10700	n-Nitrosodiphenylamine	33.0%	166.7%
4210	2220	6190	Pentachlorophenol	52.7%	147.0%
1910	1120	2710	Phenanthrene	58.6%	141.9%
4230	2040	6420	Phenol	48.2%	151.8%
8360	5290	11400	Pyrene	63.3%	136.4%
7430	4230	10600	2,4,5-Trichlorophenol	56.9%	142.7%
9010	5570	12400	2,4,6-Trichlorophenol	61.8%	137.6%

NATIONAL RESEARCH COUNCIL CANADA

Certificate of Analysis

ARI
I-7937
I-7938
I-7939

Certified Reference Material

HISS-1, MESS-3 and PACS-2

Marine sediment reference materials for trace metals and other constituents

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The following tables show those constituents for which certified and information values have been established. Certified values and their uncertainties are reported as mass fractions (based on dry mass). The uncertainties represent 95% confidence limits for an individual sub-sample of 250 mg or greater.

Table 1: Certified quantity values – Mass fraction

Element	HISS-1 mg/kg	MESS-3 mg/kg	PACS-2 mg/kg
Antimony (b,g,i,n,q)	(0.13) *	1.02 ± 0.09	11.3 ± 2.6
Arsenic (b,g,h,i,n,x)	0.801 ± 0.099	21.2 ± 1.1	26.2 ± 1.5
Beryllium (b,g,i)	0.129 ± 0.023	2.30 ± 0.12	1.0 ± 0.2
Cadmium (b,g,i,q)	0.024 ± 0.009	0.24 ± 0.01	2.11 ± 0.15
Chromium (b,g,i,n,q,x)	30.0 ± 6.8†	105 ± 4	90.7 ± 4.6
Cobalt (b,g,i,n,x)	(0.65) *	14.4 ± 2.0	11.5 ± 0.3
Copper (b,g,i,n,q,x)	2.29 ± 0.37	33.9 ± 1.6	310 ± 12
Lead (b,g,i,q,x)	3.13 ± 0.40	21.1 ± 0.7	183 ± 8
Lithium (b,g,i,q)	2.83 ± 0.54	73.6 ± 5.2	32.2 ± 2.0
Manganese (b,g,i,n,x)	66.1 ± 4.2	324 ± 12	440 ± 19
Mercury (a,c,q)	(0.01) *	0.091 ± 0.009	3.04 ± 0.20
Molybdenum (g,i,q)	(0.13) *	2.78 ± 0.07	5.43 ± 0.28
Nickel (b,g,i,q,x)	2.16 ± 0.29	46.9 ± 2.2	39.5 ± 2.3
Selenium (b,g,h)	0.050 ± 0.007	0.72 ± 0.05	0.92 ± 0.22
Silver (b,g,i,q)	0.016 ± 0.002	0.18 ± 0.02	1.22 ± 0.14
Strontium (f,i,n,q,x)	96.9 ± 11.2	129 ± 11	276 ± 30
Thallium (b,q)	(0.06) *	0.90 ± 0.06	(0.6) *
Tin (b,g,i,q)	(0.11) *	2.50 ± 0.52	19.8 ± 2.5
Uranium (q)	(0.26) *	(4) *	(3) *
Vanadium (b,g,i,n,x)	6.80 ± 0.78	243 ± 10	133 ± 5
Zinc (b,g,i,n,q,x)	4.94 ± 0.79	159 ± 8	364 ± 23

* for information only

† see page 4

National Research Council Canada
Conseil national de recherches Canada

Canada

Table 2: Matrix and minor constituents - Mass fraction

Element	HISS-1 g/100g	MESS-3 g/100g	PACS-2 g/100g
Aluminum (f,i,n,x)	0.73 ± 0.05	8.59 ± 0.23	6.62 ± 0.32
Carbon (e)	—	(2) *	(3.3) *
Calcium (f,i,n,x)	1.14 ± 0.10	1.47 ± 0.06	1.96 ± 0.18
Clorine (n,x)	(0.35) *	—	(3) *
Iron (f,i,n,x)	0.246 ± 0.009	4.34 ± 0.11	4.09 ± 0.06
Potassium (f,n,x)	0.332 ± 0.013	(2.6) *	1.24 ± 0.05
Magnesium (f,i,x)	0.075 ± 0.016	(1.6) *	1.47 ± 0.13
Sodium (f,i,n)	0.373 ± 0.026	(1.6) *	3.45 ± 0.17
Phosphorus (i,x)	—	(0.12) *	0.096± 0.004
Sulfur (i,x)	—	(0.19) *	1.29 ± 0.13
Silicon (i,x)	(44) *	(27) *	(28) *
Titanium (f,i,n,x)	0.076 ± 0.004	0.44 ± 0.06	0.443± 0.032

* for information only

Coding

a - Atomic fluorescence spectrometry
b - Inductively coupled plasma mass spectrometry (ICPMS)
c - Cold vapour atomic absorption spectrometry
e - Coulometry
f - Flame atomic absorption spectrometry
g - Graphite furnace atomic absorption spectrometry
h - Hydride generation atomic absorption spectrometry
i - Inductively coupled plasma atomic emission spectrometry
l - High-performance liquid chromatography ICPMS
m - Gas chromatography microwave induced plasma atomic emission
n - Instrumental neutron activation analysis
q - Isotope dilution inductively coupled plasma mass spectrometry
r - Infrared spectrometry
x - X-ray fluorescence spectrometry

Not all the methods listed above were applied to all three certified reference materials.

Table 3: Butlytin mass fractions in PACS-2

Butyltin	PACS-2 µg/g (as Sn)
Tributyltin (l,m)	0.832 ± 0.095
Dibutyltin (l,m)	1.100 ± 0.135
Monobutyltin (l,m)	(0.7) *

* for information only

The uncertainties in the certified values of the butyltins reflect expanded uncertainties. A separate certificate for the butyltins is available upon request.

Intended use

These reference materials are primarily intended for use in the calibration of procedures and the development of methods for the analysis of marine sediments and materials with similar matrices.

Preparation of material

HISS-1 was collected from the Hibernia Shelf, off the coast of Newfoundland. MESS-3 is from the Beaufort Sea. PACS-2 was collected from Esquimalt harbour, B.C. All were freeze dried, screened to pass a No. 120 (125 µm) screen, blended and bottled by Institute staff using the facilities of the Canada Centre for Mineral and Energy Technology in Ottawa. After bottling, the samples were radiation sterilized with a minimum dose of 25 kGy by Nordion International Inc. to minimize any effects from biological activity.

Instructions for drying

Although initially free from moisture following the freeze drying, the materials, which contain sea salt, have adsorbed moisture during subsequent operations. They should be dried to a constant mass before use. Drying for several hours at 105 °C is recommended as a relatively simple method to achieve a dry mass for most purposes.

Storage and sampling

It is recommended that the material be stored in a cool, clean location. Each bottle is packaged in a trilaminate foil pouch which serves as an impermeable barrier to mercury vapour. Under conditions of high ambient levels of mercury vapour, mercury is able to penetrate the plastic cap of the bottle, thereby potentially contaminating the contents. The bottle contents should be well mixed by rotation and shaking prior to use, and tightly closed immediately thereafter.

Storage of PACS-2 for organotin stability

To ensure the stability of the organotin species in PACS-2, it is necessary to store the material at a temperature of 4 °C or lower.

Information values

Information values are considered less reliable than certified values because they are not based on the results of at least two independent methods, there were insufficient analyses performed or inhomogeneity is suspected. These values are given for information only and care should be excised not to attribute more reliability to these values than they warrant.

Homogeneity

Randomly selected bottles were used for the analytical determinations. Results from different bottles showed no significant differences compared to results from subsamples within bottles; nor was there any correlation between values obtained and bottle sequence. Thus, it is assumed that all bottles of each of these materials have essentially the same composition. An exception is Co in HISS-1, for which results indicated sample inhomogeneity.

† Chromium in HISS-1

It became apparent during the certification of HISS-1 that there is a significant fraction of Cr that is not easily solubilized. The certified value of 30 mg/kg was obtained using solid sampling techniques or prolonged digestion with hydrofluoric, sulphuric and perchloric acids. Less vigorous acid dissolution techniques (including microwave heating using closed vessels at high pressure) typically result in a Cr mass fraction between 10 and 13 mg/kg.

Metrological traceability

Results presented in this certificate are traceable to the SI through gravimetrically prepared standards of established purity and international measurement intercomparisons. As such, they serve as suitable reference materials for laboratory quality assurance programs, as outlined in ISO/IEC 17025. This CRM is registered at the Bureau International des Poids et Mesures (BIPM) in Appendix C of the Comité International des Poids et Mesures database listing Calibration and Measurement Capabilities accepted by signatories to the Mutual Recognition Arrangement of the Metre Convention.

Accreditation

The Chemical Metrology laboratory is compliant to ISO 17025 and ISO Guide 34, with approval by The Inter-American Metrology System (SIM). The certificate of approval is available upon request.

Updates

Users should ensure that the certificate they have is current. Our web site at http://www.nrc-cnrc.gc.ca will contain any new information.

References

1. Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM 100:2008.

Acknowledgements

The following staff members of the Measurement Science and Standards portfolio at the NRC contributed to the production and certification of HISS-3, MESS-3 or PACS-2: S. Berman, V.J. Boyko, V.P. Clancy, J. Lam, P. Maxwell, J.W. McLaren, B. Methven, C. Brophy, R. Sturgeon, S. Willie and L. Yang.

The cooperation of the following during the certification of one or more of these sediments is gratefully acknowledged:

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R. Parker, Department of Geology, University of Auckland, New Zealand.

C. Davies, Z. Huang and P. Moore, Brooks Rand Ltd., Seattle, Washington.

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R. Smith, Skidaway Inst. of Oceanography, Savannah, GA.

Issac Pereiro and Ryszard Lobinski, Laboratoire de Chimie Bio-Inorganique et Environnement, Pau, France.

HISS-1
Date of issue: February 1997
Date of expiry: December 2014

MESS-3
Date of issue: January 2000
Date of expiry: December 2014

PACS-2
Date of issue: August 1997
Date of expiry: December 2014
Butyltin values updated: August 2004
Date of expiry (butyltins): December 2014
Butyltin expiry dates revised: January 2012

Approved by: ____________________

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Email: CRM-MRCInorganic-Inorganiques@nrc-cnrc.gc.ca

Également disponible en français sur demande.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1941b

Organics in Marine Sediment

This Standard Reference Material (SRM) is marine sediment collected at the mouth of the Baltimore Harbor. SRM 1941b is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in marine sediment and similar matrices. Information is also provided for total organic carbon, carbon, hydrogen, and nitrogen. All of the constituents for which certified, reference, and information values are provided in SRM 1941b were naturally present in the sediment material before processing. A unit of SRM 1941b consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 29 PCB congeners, and 7 chlorinated pesticides are provided in Tables 1 through 3. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques along with results from an interlaboratory comparison study [1,2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 44 additional PAHs (some in combination), 13 additional PCB congeners, and 2 additional chlorinated pesticides in Tables 4 to 7. A reference value for total organic carbon is provided in Table 8. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 9 for carbon, hydrogen, and nitrogen. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

Expiration of Certification: The certification of **SRM 1941a** is valid, within the measurement uncertainties specified, until **01 October 2020**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this material was under the leadership of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 December 2011
Certificate Revision History on Page 13

Robert L. Watters, Jr. Chief
Measurement Services Division

Analytical measurements for the certification of SRM 1941b were performed at NIST by J.R. Kucklick, B.J. Porter, D.L. Poster, M.M. Schantz, P. Schubert, S. Tutschku, and L.L. Yu of the NIST Analytical Chemistry Division. Measurements for percent total organic carbon were provided by a commercial laboratory and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The carbon, hydrogen, and nitrogen data were provided by a commercial laboratory. Results were also used from 38 laboratories (see Appendix A) that participated in an interlaboratory comparison exercise coordinated by NIST.

Collection and preparation of SRM 1941b were performed by M.P. Cronise and C.N. Fales of the NIST Measurement Services Division and B.J. Porter and M.M. Schantz of the NIST Analytical Chemistry Division. The sediment material was collected with the assistance of G.G. Lauenstein, J. Collier, and J. Lewis (National Oceanic and Atmospheric Administration).

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh and J.H. Yen of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 1941b must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1941b are reported on a dry-mass basis. The SRM, as received, contains approximately 2.4 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis; or a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate subsample.

PREPARATION AND ANALYSIS(1)

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39^{o}12.3'N and 76^{o}31.4'W). This location is very near the site where SRM 1941 and SRM 1941a were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 150 µm (100 % passing), homogenized in a cone blender, radiation sterilized (^{60}Co), and then packaged in screw-capped amber glass bottles each containing approximately 50 g.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1941b are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1941b was determined by measuring the mass loss after freeze drying subsamples of 1.1 g to 1.3 g for four days at 1 Pa with a –10 °C shelf temperature and a –50 °C condenser temperature. The moisture content in SRM 1941b at the time of the certification analyses was 2.39 % ± 0.08 % (95 % confidence level). Analytical results for the organic constituents were determined on an as-received basis and then converted to a dry-mass basis by dividing by the conversion factor of 0.9761 (g dry mass/g as-received mass).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1941b was similar to that reported for the recent certification of several environmental matrix SRMs [3-6] and is described in detail elsewhere [7]. The approach consisted of combining results from analyses using various combinations of different extraction techniques and solvents, clean-up/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized-fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid-phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas

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(1) Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (all column compositions are given as % mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, and a relatively non-polar proprietary phase.

Three sets of GC/MS results, designated as GC/MS (I), GC/MS (II), and GC/MS (III) were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of approximately 1 g from ten bottles of SRM 1941b were extracted using PFE with DCM [8]. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 2 % DCM in hexane (all solvent concentrations are given as % volume fraction). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 5 g subsamples from six bottles of SRM 1941b. These samples were extracted using PFE with DCM. The high molecular mass compounds were removed from the extracts using size exclusion chromatography (SEC) with a preparative-scale divinylbenzene-polystyrene column (10 µm particle size with 100 Å diameter pores), and the sulfur was removed from the extracts by adding copper powder. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The analysis was by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17 MS, J&W Scientific, Folsom, CA). For the GC/MS (III), 9 g subsamples from six bottles of SRM 1941b were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific, Folsom, CA).

Two sets of LC-FL results, designated as LC-FL (total) and LC-FL (isomer), were used in the certification process. For the LC-FL (total), subsamples of approximately 1 g from six bottles of SRM 1941b were extracted using PFE with a mixture of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through an aminopropylsilane SPE cartridge using 2 % DCM in hexane to obtain the total PAH fraction. For the LC-FL (isomer), a 5 g subsample from the six bottles was extracted using PFE with DCM and processed through an aminopropylsilane SPE cartridge using 10 % DCM in hexane; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [9-12]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5 µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [10,11].

For the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [13]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PAHs in SRM 1941b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PAHs.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1941b was assessed by analyzing duplicate samples of approximately 1 g from ten bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at this sample size.

PAH Isomers of Molecular Mass 300 and 302: For the determination of the molecular mass 300 and 302 isomers, three subsamples of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substitued methylpolysiloxane phase (0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). Perdeuterated dibenzo[*a,i*]pyrene was added to the sediment prior to extraction for use as an internal standard [14].

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1941b was similar to that reported for the recent certification of several environmental matrix SRMs [4,5,15-17], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, clean-up/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity for the separation of PCBs and chlorinated pesticides. The analytical methods are described in detail elsewhere [7].

Six sets of results were obtained designated as GC-ECD (I) A and B, GC/MS (I) A and B, GC/MS (II), and Interlaboratory Comparison Exercise. For the GC-ECD (I) analyses, approximately 10 g subsamples from six bottles of SRM 1941b were extracted using PFE with DCM. Copper powder was added to the extract to remove elemental sulfur, and SEC, as described above, was used to remove the high molecular mass compounds. The concentrated extract was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase, (0.25 µm film thickness, DB-5, J&W Scientific, Folsom, CA), and a 0.25 mm × 60 m fused silica capillary column with a non-polar proprietary phase, (0.25 µm film thickness, DB-XLB, J&W Scientific, Folsom, CA). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the proprietary phase are designated as GC-ECD (IB). For the GC-ECD analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [18,19]), and endosulfan I-d_4, 4,4'-DDE-d_8, 4,4'-DD-d_8, and 4,4'-DDT-d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

Two sets of results were obtained by GC/MS. For GC/MS (I), approximately 9 g subsamples from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone for approximately 18 h. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS with two ionization modes, electron impact (EI) and negative ion chemical ionization (NICI). The GC/MS EI method, GC/MS (IA), used a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase, (0.25 µm film thickness, DB-XLB, J&W Scientific, Folsom, CA). The GC/MS NICI method, GC/MS (IB), used a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase, (0.25 µm film thickness, DB-5MS, J&W Scientific, Folsom, CA). The GC/MS (II) results were obtained in the same manner as the GC/MS (IA) analyses except that three subsamples were Soxhlet extracted with DCM for approximately 18 h. For the GC/MS analyses, selected carbon-13 labeled PCB congeners and chlorinated pesticides were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [13]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1941b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

The reference value for PCB 77 was determined from a separate fraction. The samples were extracted and processed as for GC-ECD (I) above. The first (PCB and lower polarity pesticide) fraction from the semi-preparative aminopropylsilane column was further fractionated using a Cosmosil PYE column (5 µm particle size, 4.6 mm i.d. × 25 cm, Phenomenex, Torrance, CA) [20]. Three fractions were collected: the first fraction contained the pesticides and multi-*ortho* PCBs, the second fraction contained the polychlorinated naphthalenes, non-*ortho* PCB congeners, and some mono-*ortho* PCB congeners, and the third fraction removed the residual planar compounds from the column. The second fraction was analyzed by GC/MS NICI using the same column as GC/MS (IB) above. Carbon-13 labeled PCB 77 was used as an internal standard for quantification purposes.

Total Organic Carbon: Two laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from three bottles of SRM 1941b. One of the laboratories also analyzed three subsamples from three bottles of SRM 1941b for carbon, hydrogen, and nitrogen.

Table 1. Certified Concentrations for Selected PAHs in SRM 1941b

PAHs	Mass Fractions (dry-mass basis)[a] µg/kg
Naphthalene[d,e,f,g,h,i]	848 ± 95[b]
Fluorene[d,e,f,g,h,i]	85 ± 15[b]
Phenanthrene[d,e,f,g,h,i]	406 ± 44[b]
Anthracene[d,e,f,g,h,i]	184 ± 18[b]
3-Methylphenanthrene[d,e,f]	105 ± 13[b]
2-Methylphenathrene[d,e,f]	128 ± 14[b]
1-Methylphenanthrene[d,e,f,i]	73.2 ± 5.9[b]
Fluoranthene[d,e,f,g,h,i]	651 ± 50[b]
Pyrene[d,e,f,g,h,i]	581 ± 39[b]
Benz[*a*]anthracene[d,e,f,g,h,i]	335 ± 25[b]
Chrysene[f,h]	291 ± 31[b]
Triphenylene[f,h]	108 ± 5[c]
Benzo[*b*]fluoranthene[e,g]	453 ± 21[b]
Benzo[*k*]fluoranthene[d,e,f,g]	225 ± 18[b]
Benzo[*e*]pyrene[d,e,f,i]	325 ± 25[b]
Benzo[*a*]pyrene[d,e,f,h,i]	358 ± 17[b]
Perylene[d,e,f,h,i]	397 ± 45[b]
Benzo[*ghi*]perylene[d,e,f,h,i]	307 ± 45[b]
Indeno[1,2,3-*cd*]pyrene[d,e,f,h,i]	341 ± 57[b]
Dibenz[*a*,*j*]anthracene[d,e,f,h]	48.9 ± 4.6[b]
Dibenz[*a*,*c*]anthracene[e,h]	36.7 ± 5.2[b]
Dibenz[*a*,*h*]anthracene[e,h]	53 ± 10[b]
Benzo[*b*]chrysene[d,e,f,h]	53 ± 12[b]
Picene[d,e,f]	46.6 ± 4.7[b]

[a] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[b] Certified values are weighted means of the results from two to six analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

[c] The certified value is an unweighted mean of the results from two analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

[d] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[e] GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[f] GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] LC-FL (total) of total PAH fraction after PFE with DCM.

[h] LC-FL (isomer) of isomeric PAH fractions after PFE with DCM.

[i] 1999 Interlaboratory Comparison Study [13] with between 21 and 29 laboratories submitting data for each PAH.

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 1941b

PCB Congeners		Mass Fractions (dry-mass basis)[b] µg/kg		
PCB 8	(2,4'-Dichlorobiphenyl)[e,f,g,h,i]	1.65	±	0.19[c]
PCB 18	(2,2',5-Trichlorobiphenyl)[e,f,g,h,i]	2.39	±	0.29[c]
PCB 28	(2,4,4'-Trichlorobiphenyl)[e,f,g,h,i]	4.52	±	0.57[c]
PCB 31	(2,4',5-Trichlorobiphenyl)[e,g,h]	3.18	±	0.41[c]
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl)[e,f,g,h,i]	3.85	±	0.20[d]
PCB 49	(2,2'4,5'-Tetrachlorobiphenyl)[e,f,g,h]	4.34	±	0.28[d]
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)[e,f,g,h,i]	5.24	±	0.28[d]
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)[e,g,h,i,j]	4.96	±	0.53[d]
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)[e,f,h,j]	1.14	±	0.16[c]
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)[e,g,h,i]	3.93	±	0.62[d]
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)[e,f,g,h,j]	2.90	±	0.36[d]
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)[e,g,h,i,j]	5.11	±	0.34[d]
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)[e,f,g,h,i,j]	1.43	±	0.10[d]
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)[e,g,h,j]	4.62	±	0.36[d]
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)[e,f,g,h,i,j]	4.23	±	0.19[d]
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)[e,f,g,h,i,j]	0.696	±	0.044[d]
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)[e,g,h,j]	3.60	±	0.28[d]
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)[e,f,g,j]	4.35	±	0.26[c]
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[e,f,g,h,i,j]	5.47	±	0.32[d]
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)[e,f,g,h,j]	0.507	±	0.090[c]
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)[e,f,g,h,i,j]	1.35	±	0.09[d]
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)[e,f,g,h,i,j]	3.24	±	0.51[d]
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)[e,f,g,j]	0.979	±	0.087[c]
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)[e,f,g,h,i,j]	2.17	±	0.22[d]
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)[e,f,g,j]	1.04	±	0.06[c]
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)[e,g,i,j]	0.645	±	0.060[d]
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)[e,g,j]	0.777	±	0.034[c]
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)[e,g,h,i,j]	2.42	±	0.19[d]
PCB 209	Decachlorobiphenyl[e,f,g,h,i,j]	4.86	±	0.45[d]

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [18] and later revised by Schulte and Malisch [19] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 201 and PCB 107 (see Table 5) are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200 and the IUPAC PCB 107 is listed as PCB 108.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[c] Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

[d] Certified values are weighted means of the results from three to six analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

[e] GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[f] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[g] GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

[h] GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

[i] 1999 Interlaboratory Comparison Study [13] with between 13 and 31 laboratories submitting data for each PCB congener.

[j] GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions (dry-mass basis)[a] µg/kg		
Hexachlorobenzene[d,e,f,g]	5.83	±	0.38[b]
cis-Chlordane[d,e,f,g,h]	0.85	±	0.11[c]
trans-Chlordane[d,e,g]	0.566	±	0.093[b]
cis-Nonachlor[d,g,h]	0.378	±	0.053[c]
trans-Nonachlor[d,e,f,g,h]	0.438	±	0.073[b]
4,4'-DDE[d,f,g,h]	3.22	±	0.28[c]
4,4'-DDD[d,f,g,h]	4.66	±	0.46[c]

[a] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[b] Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

[c] Certified values are weighted means of the results from three to five analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

[d] GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[e] GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

[f] GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

[g] 1999 Interlaboratory Comparison Study [13] with between 13 and 31 laboratories submitting data for each pesticide.

[h] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 4. Reference Concentrations for Selected PAHs in SRM 1941b

PAHs	Mass Fractions (dry-mass basis)[a] µg/kg		
1-Methylnaphthalene[d,e,f,i]	127	±	14[b]
2-Methylnaphthalene[d,e,f,i]	276	±	53[b]
2,6-Dimethylnaphthalene[d,e,f,i]	75.9	±	4.5[b]
2,3,5-Trimethylnaphthalene[d,e,f,i]	25.5	±	5.1[b]
Biphenyl[d,e,f,i]	74.0	±	8.0[b]
Acenaphthylene[d,e,f,i]	53.3	±	6.4[b]
Acenaphthene[d,e,f,i]	38.4	±	5.2[b]
9-Methylphenanthrene[e]	63.5	±	2.5[c]
4-Methylphenanthrene and 9-Methylphenanthrene[d,f]	80.1	±	4.8[b]
2-Methylanthracene[e,f]	36	±	15[b]
8-Methylfluoranthene[d]	49.5	±	2.7 [c]
7-Methylfluoranthene[d]	45.4	±	1.5 [c]
1-Methylfluoranthene[d]	42.4	±	2.1 [c]
3-Methylfluoranthene[d]	28.8	±	1.3 [c]
2-Methylpyrene[d]	78.7	±	4.0 [c]
4-Methylpyrene[d]	66.4	±	2.6 [c]
1-Methylpyrene[d]	52.5	±	2.3 [c]
Acephenanthrene[f]	30.5	±	1.9 [c]
Benzo[*c*]phenanthrene[d,e,f]	58	±	15[b]
Benzo[*a*]fluoranthene[d,e,f]	73	±	18[b]
Benzo[*j*]fluoranthene[e]	217	±	5[c]
Indeno[1,2,3-*cd*]fluoranthene[f]	9.63	±	0.34[c]
Pentaphene[f]	25.3	±	1.0[c]

[a] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[b] Reference values are weighted means of the results from two to four analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

[c] Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [2]. The coverage factor, k, is determined from the Student's t-distribution for the appropriate degrees of freedom and 95 % confidence for each analyte.

[d] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[e] GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[f] GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] LC-FL (total) of total PAH fraction after PFE with DCM.

[h] LC-FL (isomer) of isomeric PAH fractions after PFE with DCM.

[i] 1999 Interlaboratory Comparison Study [13] with between 14 and 26 laboratories submitting data for each PAH.

Table 5. Reference Concentrations for Selected PAHs of
Molecular Weight 300 and 302 in SRM 1941b

PAHs	Mass Fractions (dry-mass basis)[a,b,c] µg/kg
Coronene	72.6 ± 4.7
Dibenzo[*b*,*e*]fluoranthene	10.3 ± 0.3
Naphtho[1,2-*b*]fluoranthene	91.0 ± 3.1
Naphtho[1,2-*k*]fluoranthene and Naphtho[2,3-*j*]fluoranthene	79.8 ± 2.5
Naphtho[2,3-*b*]fluoranthene	23.5 ± 0.3
Dibenzo[*b*,*k*]fluoranthene	95.6 ± 3.1
Dibenzo[*a*,*k*]fluoranthene	26.6 ± 0.4
Dibenzo[*j*,*l*]fluoranthene	63.8 ± 1.8
Dibenzo[*a*,*l*]pyrene	11.1 ± 1.0
Naphtho[2,3-*k*]fluoranthene	10.7 ± 0.6
Naphtho[1,2-*a*]pyrene	16.7 ± 1.4
Naphtho[2,3-*e*]pyrene	33.2 ± 2.3
Dibenzo[*a*,*e*]pyrene	76.1 ± 3.6
Naphtho[2,1-*a*]pyrene	59.2 ± 1.8
Dibenzo[*e*,*i*]pyrene	35.0 ± 2.4
Naphtho[2,3-*a*]pyrene	16.5 ± 0.6
Benzo[*b*]perylene	38.2 ± 1.2
Dibenzo[*a*,*i*]pyrene	25.5 ± 1.0
Dibenzo[*a*,*h*]pyrene	6.94 ± 0.29

[a] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[b] Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, *U*, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, *k*, is determined from the Student's *t*-distribution for the associated degrees of freedom and 95 % confidence level for each analyte.

[c] GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM [14].

Table 6. Reference Concentrations for Selected PCB Congeners[a] in SRM 1941b

PCB Congeners	Mass Fractions (dry-mass basis)[b,c] µg/kg
PCB 45 (2,2',3,6-Tetrachlorobiphenyl)[d,e]	0.73 ± 0.12
PCB 56 (2,3,3',4-Tetrachlorobiphenyl)[d,f,g]	1.21 ± 0.11
PCB 63 (2,3,4',5-Tetrachlorobiphenyl)[e,f,g]	0.213 ± 0.040
PCB 70 (2,3',4',5-Tetrachlorobiphenyl)[e,f,g]	4.99 ± 0.29
PCB 74 (2,4,4',5-Tetrachlorobiphenyl)[e,f,g]	2.04 ± 0.15
PCB 77 (3,3',4,4'-Tetrachlorobiphenyl)[h]	0.31 ± 0.03
PCB 107 (2,3,3',4,5'-Pentachlorobiphenyl)[d,e,f,g]	0.628 ± 0.028
PCB 132 (2,2',3,3',4,6'-Hexachlorobiphenyl)[d,f,g]	1.28 ± 0.27
PCB 146 (2,2',3,4',5,5'-Hexachlorobiphenyl)[e,f,g]	1.22 ± 0.12
PCB 158 (2,3,3',4,4',6-Hexachlorobiphenyl)[d,e,f,g]	0.65 ± 0.15
PCB 163 (2,3,3',4',5,6-Hexachlorobiphenyl)[e,f,g]	1.28 ± 0.06
PCB 174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)[d,e,f,g]	1.51 ± 0.39
PCB 193 (2,3,3',4',5,5',6-Heptachlorobiphenyl)[d,e,f,g]	0.292 ± 0.075

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [18] and later revised by Schulte and Malisch [19] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 201 (see Table 2) and PCB 107 are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200 and the IUPAC PCB 107 is listed as PCB 108.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

[c] For these PCB congeners except PCB 77, the reference values are unweighted means of the results from two to four analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled within-method variance following the ISO and NIST Guides [2]. For PCB 77, the reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's *t*-distribution corresponding to two degrees of freedom and 95 % confidence level for PCB 77.

[d] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[e] GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

[f] GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

[h] GC/MS NICI on a 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (I) fractionated using a PYE column.

Table 7. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions (dry-mass basis)[a,b] µg/kg
2,4'-DDE[c,d]	0.38 ± 0.12
4,4'-DDT[e,f]	1.12 ± 0.42

[a] Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.
[b] The reference values are unweighted means of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].
[c] GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).
[d] GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).
[e] GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.
[f] 1999 Interlaboratory Comparison Study [13] with 10 laboratories submitting data for 4,4’-DDT.

Table 8. Reference Value for Total Organic Carbon in SRM 1941b

Total Organic Carbon (TOC)	2.99 % ± 0.24 % mass fraction[a,b]

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.
[b] The reference value for total organic carbon is a weighted mean value from routine measurements made by two laboratories [21]. The uncertainty listed is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance. The reporting follows the ISO and NIST Guides [2].

Table 9. Information Values for Carbon, Hydrogen, and Nitrogen in SRM 1941b

Elements	Mass Fractions (dry-mass basis)[a] %
Carbon	3.3
Hydrogen	1.2
Nitrogen	< 0.5

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.

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Certificate Revision History: 01 December 2011 (Extension of certification period; editorial changes); 16 August 2004 (This revision removes the reference values for the butyl tins and makes editorial changes); 15 July 2002 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

The laboratories listed below performed measurements that contributed to the certification of SRM 1941b, Organics in Marine Sediment.

Arthur D. Little, Inc; Cambridge, MA, USA
Axys Analytical Services; Sidney, BC, Canada
B & B Laboratories; College Station, TX, USA
Battelle Ocean Sciences; Duxbury, MA, USA
Bedford Institute of Oceanography; Dartmouth, NS, Canada
California Department of Fish and Game; Rancho Cordova, CA, USA
Central Contra Costa Sanitary District; Martinez, CA, USA
Chesapeake Biological Laboratory; Solomons, MD, USA
Centro de Investigaciones Energeticas Medioambientales y Tecnologicas; Madrid, Spain
City of Los Angeles Environmental Monitoring Division; Playa del Rey, CA, USA
City of San Jose Environmental Services Department; San Jose, CA, USA
Columbia Analytical Services; Kelso, WA, USA
East Bay Municipal Utility District; Oakland, CA, USA
Florida Department of Environmental Protection; Tallahassee, FL, USA
Manchester Environmental Laboratory; Port Orchard, WA, USA
Murray State University; Murray, KY, USA
Massachusetts Water Resources Authority Central Lab; Winthrop, MA, USA
National Research Council of Canada; Ottawa, Ontario, Canada
National Oceanic and Atmospheric Association (NOAA), National Marine Fisheries Service (NMFS), Auke Bay Laboratory; Juneau, AK, USA
NOAA, National Ocean Service/Center for Coastal Environmental Health and Biomolecular Research; Charleston, SC, USA
NOAA, NMFS, Sandy Hook Marine Laboratory; Highlands, NJ, USA
NOAA, NMFS, Northwest Fisheries Science Center; Seattle, WA, USA
Orange County Sanitation District; Fountain Valley, CA, USA
Philip Analytical Services; Burlington, Ontario, Canada
Serv de Hidrografia Naval; Buenos Aires, Argentina
Skidaway Institute of Technology; Savannah, GA, USA
Southwest Laboratory of Oklahoma; Broken Arrow, OK, USA
Severn Trent Knoxville Laboratory; Knoxville, TN, USA
Texas A&M University, Geochemical and Environmental Research Group; College Station, TX, USA
Texas Parks and Wildlife Department; San Marcos, TX, USA
University of California at Los Angeles, Institute of Geophysics and Planetary Physics; Los Angeles, CA, USA
University of Connecticut, Environmental Research Institute; Storrs, CT, USA
University of Rhode Island, Graduate School of Oceanography; Narragansett, RI, USA
US Department of Agriculture, Environmental Chemistry Laboratory; Beltsville, MD, USA
US Environmental Protection Agency, Atlantic Ecology Division; Narragansett, RI, USA
US Geological Survey, National Water Quality Laboratory; Denver, CO, USA
Woods Hole Group Environmental Lab; Raynham, MA, USA
Wright State University; Dayton, OH, USA

Sampling and Analysis Plan
Proposed Grays Harbor Potash Export Facility
Dredged Material Characterization

Appendix C
Signature Page for Subcontractors

PROPOSED GRAYS HARBOR POTASH EXPORT FACILITY
DREDGED MATERIAL CHARACTERIZATION

SIGNATURE PAGE FOR SUBCONTRACTORS

Approval signatures indicate that subcontractors have reviewed this Sampling and Analysis Plan and agree to follow the methods and quality assurance procedures contained herein.

____ Date: ________________
Amanda Volgardsen
Analytical Resources, Inc.

____ Date: ________________
Gerald J. Irissarri
Northwest Aquatic Sciences

____ Date: ________________
Gary Maxwell
AMEC/WOOD

____ Date: ________________
Eric Parker
Research Support Services

From: Warner, Lauran C CIV USARMY CENWS (US)
To: Fisher, Sally; England, Victoria; Ryckman, April; Roberts, Grace
Cc: Celia Barton (celia.barton@dnr.wa.gov); Laura Inouye (lino461@ecy.wa.gov); Justine Barton (barton.justine@epa.gov)
Subject: SAP Approval: Proposed GH Potash Facility Dredged Material Characterization, 2/22/18 revision 3
Date: Friday, February 23, 2018 5:54:36 PM

This SAP is approved subject to the following:

1. Correct and update Table 1. Compositing Scheme and DMMU Volumes to reflect most recent sampling design and cross-sections.
2. Move SS-6 to a nearby location where the Z-layer can more likely be reached (shorter core).
3. Move SS-8 to a nearby location where at least 3 feet of sub-surface material can be sampled and so that DMMU 4 is represented by at least 4 sample locations.
4. Include name of proposed bioassay laboratory.

I am available by phone at 206-383-4143 at any time during sampling. I plan to be on-site to observe sample processing on Monday, 2/26 and will update you on my time of arrival on Monday morning.

- Lauran

Lauran Warner
Biologist - Dredged Material Management Office
lauran.c.warner@usace.army.mil
D: 206-764-6550
M: 206-383-4143

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix B
DMMO Memorandum:
Status of Sediment Characterization for
Proposed Grays Harbor Potash Export Facility
Grays Harbor, Washington – 3 July 2018

CENWS-ODS-ND

MEMORANDUM FOR: Sally Fisher, BergerABAM July 3, 2018

SUBJECT: Status of sediment characterization for Proposed Grays Harbor Potash Export Facility, Grays Harbor, Washington.

1. INTRODUCTION. This memorandum addresses the status of sediment characterization performed to date for BHP Billiton Canada Inc. (BHP) by BergerABAM. The purpose of the characterization is to evaluate proposed dredged material for inwater disposal, per Dredged Material Management Program (DMMP) guidelines. BHP proposes to dredge approximately 110,000 cubic yards (cy) of sediment to provide a new berth to support a dual-quadrant ship loader capable of servicing bulk material export vessels in Hoquiam, Washington.

   The DMMP agencies (U.S. Army Corps of Engineers, Environmental Protection Agency, and Washington Departments of Ecology and Natural Resources) reviewed the report *Dredged Material Characterization Report, Revision B, Proposed Grays Harbor Potash Export Facility, Hoquiam, WA*, dated 17 May 2018. That report cites deviations from the approved Sampling and Analysis Plan (SAP) in which two out of the planned 8 cores were not collected, as well as reduced sample recovery in a third core and other deviations (Table 1).

Table 1. Summary of planned and actual core locations and recovery.

DMMU ID	Core Station	Planned Sample Elevation (MLLW)	Actual Sample Elevation (MLLW)	Actual Location
DMMU-1 (surface)	SS-1	-32 to -36	-26 to -30	Moved 5.7 ft N
	SS-2	-25 to -29	-15 to -19	Moved 5.7 ft N
	SS-5	-27 to -31	-24 to -28	Moved 4.0 ft S
	SS-6	-12 to -16	-1 to -5	Moved 35.7 ft W
DMMU-2 (surface)	SS-3	-36 to -40	-33 to -37	Moved 29.2 ft E
	SS-4	-35 to -39	NS	NA
	SS-7	-37 to -41	NS	NA
	SS-8	--37 to --41	-35 to -39	Moved 13.7 ft N
DMMU-3 (subsurface)	SS-1	-36 to -42	-30 to -34	Moved 5.7 ft N
	SS-2	-29 to -35	-19 to -27	Moved 5.7 ft N
	SS-5	-31 to -47	-28 to -33	Moved 4.0 ft S
	SS-6	-16 to -33	-5 to -6	Moved 35.7 ft W
DMMU-4 (subsurface)	SS-3	-40 to -47	NS	Moved 29.2 ft E; no penetration or recovery
	SS-4	-39 to -47	NS	NA
	SS-7	-41 to -47	NS	NA
	SS-8	-41 to -47	-39 to -45	

Notes:
NS = Not sampled
NA – Not applicable
Orange shaded samples were not collected.

2. ANALYSIS. The agencies reviewed the submitted report for compliance with DMMP guidelines, in order to establish whether the agencies could determine suitability for open water disposal with the available information. The analysis considered number of samples, representativeness of sampled material, and results of laboratory analyses.

a. **Number of Samples.** This project was ranked “low-moderate,” based on DMMP guidelines used when there are few or no sources of chemicals of concern but insufficient data to support a “low” rank. Some of the BHP footprint overlaps the current Port of Grays Harbor T3, which is ranked “low.” But the BHP footprint also includes new dredging of bedded sediments—not entirely recently deposited sediments as it is for T3 maintenance dredging--and thus similar characteristics to material tested at T3 could not be assumed.

For low-moderate projects, DMMP requires a maximum of 32,000 cy for one surface (upper 4 feet of the dredge prism) DMMU. Each subsurface (deeper than 4 ft.) DMMU can represent up to 48,000 cy.

The approved SAP identified four DMMUs, per Table 2. All planned samples were collected for DMMU-1 and DMMU-3. DMMU-2 only had ½ the planned number of samples collected. However, the total number of surface samples fall within the range for required sampling density. Thus the DMMP was willing to look at further evidence for DMMU-2 to see if additional sampling would be required to determine whether DMMU-2 was suitable for open water disposal.

DMMU-4 had only one sample successfully collected. The DMMP does not consider this sufficient sampling density to evaluate this DMMU for open-water disposal.

Table 2. Summary of DMMUs and volume

DMMU ID	Core Station	Approximate *Planned* DMMU Volume (cy)	Approximate *Actual* DMMU Volume (cy)	Frequency Requirement Based on Ranking
DMMU-1 (surface)	SS-1	22,616	22, 616	1 sample/ 8,000 cy and 1 DMMU/32,000 cy (surface)
	SS-2			
	SS-5			
	SS-6			
DMMU-2 (surface)	SS-3	22,675	11,338	
	SS-4			
	SS-7			
	SS-8			
Total Surface DMMU Volume		45, 291	33,954	
Average Surface DMMU Volume		22,646	16,977	
DMMU-3 (subsurface)	SS-1	31,154	31,154	1 sample/ 8,000 cy and 1 DMMU/48,000 cy (subsurface)
	SS-2			
	SS-5			
	SS-6			
DMMU-4 (subsurface)	SS-3	31,161	7,790	
	SS-4			
	SS-7			
	SS-8			
Total Subsurface DMMU Volume		62,315	38,944	
Average Subsurface DMMU Volume		31,158	19,472	

Note:
Orange shaded samples were not collected.

b. Representativeness of Samples. The DMMP agencies looked at both horizontal and vertical representativeness of the samples collected. Samples from the most shoreward DMMUs (1 and 3) had successful samples collected from the horizontal length of the dredge prism. The shoreward area represents a steep slope, and samples were collected from both the bottom and top of that slope. Thus, for both DMMU-1 and DMMU-3, the DMMP considers horizontal representation of those samples sufficient. DMMU-2 had only two successful samples, with both the east and west ends of the prism covered. Though the mid-portion of the prism was not sampled, the DMMP continued to look at the weight of evidence for DMMU-2 to establish whether further sampling was required. The only sample for DMMU-4, from SS8, was from the east end of the dredge prism. This was not considered sufficient horizontal coverage for this DMMU.

   All sample cores from the surface DMMUs (1 and 2) recovered the full 4 ft portion of the planned vertical dredge prism, but the full prism was not recovered in most of the subsurface sample cores (see Table 1). At least 4 ft of vertical material was recovered in DMMU-3 samples SS1, SS2, and SS5. Only one ft of material was recovered from SS6, though that sample was from the shallowest portion of the dredge prism. SS8, from DMMU-4, obtained 6 ft of vertical recovery. Only core SS6 reached the bottom of the dredge prism. However, at this point there is no reason to believe that the deeper subsurface material is any different than the material recovered. The existing steep slope, adjacent to a navigation channel, does not show evidence of active erosion or accumulation. The adjacent federal navigation channel and port berth are frequently dredged of rapidly accumulating material from the Hoquiam and Chehalis Rivers, which both empty into Grays Harbor upstream of the project area.

c. Results of Laboratory Analyses. The main contamination concerns in this area are residual dioxins/furans from previous local operation of chlor-oxide bleach process paper mills. Thus the DMMP is particularly concerned about fine bedded sediments within the estuary that may have been exposed prior to plant closures, and not since removed as part of previous dredging projects. Grain size analysis (Table 3) showed >50% fines in all DMMUs but DMMU-2. It is the grain sizes from this DMMU—including much higher sand and lower fines content than the others—which tips the weight of evidence towards acceptance of current DMMU-2 data as sufficient for determining suitability. The DMMP does request that any material from DMMU-2 recovered during additional sampling be analyzed for physical characteristics (total organic carbon and grain size) to confirm the physical consistency with previous samples. Should physical characteristics show substantially different characteristics, the DMMP may request that archives from all DMMU-2 samples be composited for further chemical analysis.

   Chemical analysis of all current samples shows no detected or undetected exceedances of any DMMP chemical of concern. The one sample representing DMMU-4 showed slightly higher detections of some chemicals than in the other three DMMUs, including dioxins (Table 4.)

3. POST-DREDGE SURFACE. Z-samples (representing the top two feet of the proposed post-dredge surface) were not recovered in most samples. Future sampling should again attempt to collect Z-layer material. If Z-layer samples are not recovered, the bottom one foot of the sample core should be archived for potential further analysis, separate from any other subsample archive. Should DMMU-4 eventually be found unsuitable for open-water disposal, or if there is evidence of increasing dioxins with depth, additional sampling to evaluate the post-dredge surface may be required.

Table 3. Grain size results from existing samples.

	Surface		Subsurface	
	DMMU-1	DMMU-2	DMMU-3	DMMU-4
Gravel	0.0%	0.2%	0.0%	0.0%
Sand	46.9%	73.2%	37.2%	48.6%
Silt	45.4%	25.4%	53.0%	43.6%
Clay	7.7%	1.2%	9.9%	7.8%
Total Fines (Silt + Clay)	53.1%	26.6%	62.9%	51.5%

Note: DMMU 4 is represented by only one sample

Table 4. Results of dioxin/furan analyses for existing DMMU samples.

	DMMP Grays Harbor Guidelines			Surface		Subsurface	
	SL	BT	ML	DMMU-1	DMMU-2	DMMU-3	DMMU-4
2,3,7,8-TCDD	---	5	---	0.78 J	0.54 J	0.767 J	1.22
Total TEQ (pptr dry wt)	---	15	---	2.81	1.48	2.61	4.52

Note: DMMU 4 is represented by only one sample

4. CONCLUSION. Based on the preceding analysis, the DMMP concludes that suitability for open-water disposal can be determined with existing data for DMMU-1 and DMMU-3. For DMMU-2, one analysis of material collected from SS-4 and SS-7 should be analyzed for physical characteristics to confirm consistency with previously sampled material. A determination of suitability cannot be made for DMMU-4 with existing data. Should BHP pursue further characterization of DMMU-4, or if there are additional changes to this project, a Sampling and Analysis Plan Addendum should be prepared for approval prior to any additional sampling.

This memo has been coordinated with the DMMP agency representatives and reflects program consensus on the status of characterization for the subject project.

Appendix C

Sampling and Analysis Plan Addendum and DMMO Approval Email

BergerABAM

210 East 13th Street, Suite 300, Vancouver, Washington 98660-3231
360/823-6100 • 360/823-6101 Fax • www.abam.com

Memorandum

40600-HS-MEM-55026
Revision 1

Date: 18 July 2018

Subject: Revised Dredged Material Characterization Sampling and Analysis Plan Addendum
(Proposed Grays Harbor Potash Export Facility, A17.0202.00)

From: Grace Roberts, BergerABAM

To: Lauran Warner, Dredged Material Management Office (DMMO)

INTRODUCTION

This memorandum serves as an addendum to the Dredged Material Characterization Sampling and Analysis Plan (SAP) (BergerABAM 2018) for the proposed dredged material associated with the BHP Billiton Canada, Inc. (BHP) Proposed Grays Harbor Potash Export Facility located on the north shore of Grays Harbor in Hoquiam, Washington (Sheet 1).

Sediment characterization sampling of the proposed dredged material, dredged management units (DMMU's) 1 through 4 (Sheet 2) occurred in February 2018. Adverse weather conditions prohibited the collection of subsurface material from DMMU-4. DMMU's 1 and 3 were sampled according to the SAP and adequate surface and subsurface material was collected for analysis. Samples within DMMU-2 (surface material) were collected at SS-3 and SS-8 although weather prohibited the collection of samples at SS-4 and SS-7. The only material collected successfully from DMMU-4 (subsurface material) was collected from boring SS-8.

Sampling locations varied from planned locations from approximately 4 feet to 30 feet due to mudline surface conditions. Sample locations moved more than a few feet from the planned location were coordinated with the Dredged Material Management Office (DMMO). A summary of 'planned' versus 'actual' sample locations is depicted in Table 1 and Sheets 3 through 6.

Table 1. February 2018 Sampling: Planned vs. Actual Sample Locations in DMMU's 1 through 4

DMMU ID	Core Station	Planned Sample Elevation (MLLW)	Actual Sample Elevation (MLLW)	Actual Location	
DMMU-1 (surface)	SS-1	-32 to -36	-26 to -30	Moved 5.7 ft N	
	SS-2	-25 to -29	-15 to -19	Moved 5.7 ft N	
	SS-5	-27 to -31	-24 to -28	Moved 4.0 ft S	
	SS-6	-12 to -16	-1 to -5	Moved 35.7 ft W	
DMMU-2 (surface)	SS-3†	-36 to -40	-33 to -37	Moved 29.2 ft E	July 2018 sampling event
	SS-4†	-35 to -39	NS*	N/A	
	SS-7†	-37 to -41	NS	N/A	
	SS-8	--37 to --41	-35 to -39	Moved 13.7 ft N	
DMMU-3 (subsurface)	SS-1	-36 to -42	-30 to -34		
	SS-2	-29 to -35	-19 to -27		
	SS-5	-31 to -47	-28 to -33		
	SS-6	-16 to -33	-5 to -6		
DMMU-4 (subsurface)	SS-3†	-40 to -47	-37 to -38		July 2018 sampling event
	SS-4†	-39 to -47	NS		
	SS-7†	-41 to -47	NS		
	SS-8	-41 to -47	-39 to -45		

*NS=Not Sampled
† Proposed re-sample locations for July 2018

The DMMO determined that sampling within DMMUs 1 and 3 is representative of the conditions in those DMMUs and sufficient to provide a suitability determination for the material in those DMMUs.

The DMMO is concerned about contaminated fine bedded sediments in the project area from previous local operation of paper mills. Grain size analysis showed >50% fines in all DMMUs but DMMU-2. The DMMO has requested (per DMMO memorandum dated 3 July 2018) that the surface material from the portion of DMMU-2 that was not previously characterized (i.e. surface samples from SS-4, and SS-7) be composited (DMMU-2a) and analyzed for total organic carbon and grain size to confirm physical consistency with previous samples. The remaining surface material subsamples from SS-3, SS-4 and SS-7 will be archived separately.

Additional samples are required to adequately characterize the material in DMMU-4 for the purposes of suitability determination. Three sampling locations within DMMU-4 (SS-3, SS-4, and SS-7) will be resampled using a vibracorer. The February 2018 sampling locations and proposed re-sampling locations are depicted in the cross-sections on Sheets 3 through 6.

SAMPLE COLLECTION

Sampling and analysis for this project will be performed in accordance with Dredged Material Management Program (DMMP)/Puget Sound Estuary Program (PSEP) protocols. Samples will be collected using vibracore equipment. Cores will be processed and sampled as described in the DMMP-approved project SAP (BergerABAM 2018).

Subsamples collected from the surface material in SS-4 and SS-7 (DMMU-2a) will be composited and analyzed for a total organic carbon (TOC) and grain size analysis. The surface and subsurface material subsamples collected from SS-3, SS-4 and SS-7 will be archived separately.

DMMU-4 will be analyzed for DMMP/SMS conventional parameters, the full suite of marine DMMP/SMS contaminants of concern (COCs) including semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), polychlorinated dibenzodioxins/furans (PCDD/F), pesticides and bulk tributyltin, as shown on Table 2 in the SAP (BergerABAM 2018). Sediment reference material will also be submitted for analysis of PCBs and PCDD/F in accordance with DMMP guidance.

If the Z-layer is unachievable below DMMU-4, the bottom foot of material in each core will be collected and archived.

Vibracore Sampling

Three sampling locations within the subsurface material of DMMU-4 (SS-3, SS-4, and SS-7) will be resampled using vibracore sampling equipment mounted on a vessel operated by Research Support Services, Inc. (RSS) of Anacortes, Washington. Cores to capture surface, subsurface, and Z-layer samples will range from 10 to 12 feet below mudline. As noted in the attachment (Job Hazard Analysis and Operating Procedures – Sediment Core Sampling with Rossfelder P-3 Vibracore aboard the *Carolyn Dow*) the average success for vibracore sampling equipment ranges from 5 to 15 feet below mudline depending on material and conditions. We will make multiple attempts to collect material at each location to the fullest extent possible. We will extract and hold material from any cores where recovery is than 75% (per the DMMP User's Manual) , make additional attempts at that location, and contact the DMMO for guidance if recovery continues to be less than 75%. We have scheduled two full sampling days plus a contingency day to allow for resampling efforts.

We do not anticipate having issues achieving the penetration and recover of the surface material (upper four feet below mudline). We will consider the bottom one foot of each core as representative of the Z-layer if the target Z-layer depth is not achieved.

The vibracore system consists of a Rossfelder P-3 electric vibracorer, an optional buoyant frame, stainless steel core barrels with CAB (butyrate) liners, and a stainless steel core nose. The sample is retained in the core tube by a one-way valve at the top of the core that closes when penetration stops, and by a sheet metal "core catcher" affixed to the core nose.

The liners are clear, allowing visual evaluation of the core in the field. They are left intact and full-length for transport to the processing area. The sample remains undisturbed from the time it is extracted until it is processed.   The samples will be processed on board the vessel or onshore.

Positioning and navigation for sediment sample locations will be accomplished using a Trimble TSC-1, Pro XRS global positioning system (DGPS). The DGPS system consists of an antenna at the top of the vessels a-frame crane, and a GPS receiver and data logger in the wheelhouse. To confirm the accuracy/consistency of the readings, coordinates will be collected at a fixed point on site at the beginning and end of each day.

The subsurface elevation will be corrected for tidal fluctuations by using NOAA tide chart predictions, lead line, tide gauge on Terminal 3 pier, and noting how high or low the water level is compared to the Ordinary High Water Mark (OHWM) (Elevation = 0). Subsurface elevations are determined by noting the tidal elevation and subtracting or adding that number from/to the measured depth from the bottom of the vessel (water level) to mudline. For example, if the vessel's bottom finder reads 33 feet and the tide is at +3, the corrected subsurface elevation is -[33 feet - (+3)] = -30 feet.

Operations will be conducted from the *Carolyn Dow,* a 36-foot aluminum landing craft with a bow-mounted a-frame. On-board personnel will consist of an RSS vessel operator and technician/deckhand, and one BergerABAM field staff. One BergerABAM staff member will remain on-shore to accept the cores.

A pre-departure briefing will be conducted to familiarize personnel of site-specific hazards, vessel hazards, safety equipment, emergency procedures and sampler deployment.

Sediment sampling will be conducted from an anchored boat at the locations shown on Sheet 2. The sample location coordinates are shown in Table 2. The vibracore will be deployed over the bow of the vessel and brought aboard to remove the core liner containing the sample.

Table 2. July 2018 Sample Location Coordinates

Sample Point	WA State Plane Coordinates	
	Northing	Easting
SS-3†	615258	787616
SS-4†	615301.1	787882.2
SS-7†	615243.5	787991.8

† Proposed re-sample locations for July 2018

Further operating procedures can be found in the attached report *Job Hazard Analysis and Operating Procedures – Sediment Core Sampling with Rossfelder P-3 Vibracore aboard the Carolyn Dow* (Research Support Services for BergerABAM, June 2018).

Boring logs, sampling equipment decontamination, positioning, and chain-of-custody procedures, laboratory analysis, QA/QC will be carried out as described in the DMMP-approved SAP (BergerABAM 2018).

FINAL REPORT

As described in the 2018 SAP (BergerABAM), BergerABAM shall prepare a written report documenting all activities associated with collection, compositing, transportation of samples, and chemical and biological analysis. Additional data resulting from the resampling of DMMU-4 and DMMU-2 at sample locations SS-3, SS-4, and SS-7 will be included in the final report (Sheet 2).

REFERENCES

BergerABAM 2018. Dredged Material Management Program Sampling and Analysis Plan (SAP). 40600-HS-PLN-55007. Revision 3. Proposed Grays Harbor Potash Facility Dredged Material Characterization. Prepared for BHP Billiton Canada Inc. A17.0202.00. 22 February 2018.

ATTACHMENTS

Sheets 1-6

- Sheet 1 – Vicinity Map and Project Site Location
- Sheet 2 – Actual Dredge Area and Bathymetry
- Sheet 3 – Cross Section A
- Sheet 4 – Cross Section B
- Sheet 5 – Cross Section C
- Sheet 6 – Cross Section D

Job Hazard Analysis and Operating Procedures – Sediment Core Sampling with Rossfelder P-3 Vibracore aboard the *Carolyn Dow* (June 2018)

BHP BergerABAM

Proposed Grays Harbor Potash Export Facility-
Sampling and Analysis Plan Addendum

Sheet 1- Vicinity Map and Project Site Location

June 2018

LEGEND

11+00— —	Station
▤	Proposed Bathymetry
—	Surface DMMU
—	Sub Surface DMMU
SS-1 ⌖	Actual Sediment Sample Location
PSS-1 ⌖	Planned Sediment Sample Location
SS-4 ⌖	Unachievable Sediment Sample Location
DMMU-1	Dredged Material Management Unit

BHP

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Proposed Grays Harbor Potash Export Facility -
Sampling and Analysis Plan Addendum

Sheet 2 -Actual Dredge Area and Bathymetry June 2018

1" = 120' 60 0 60 120
scale feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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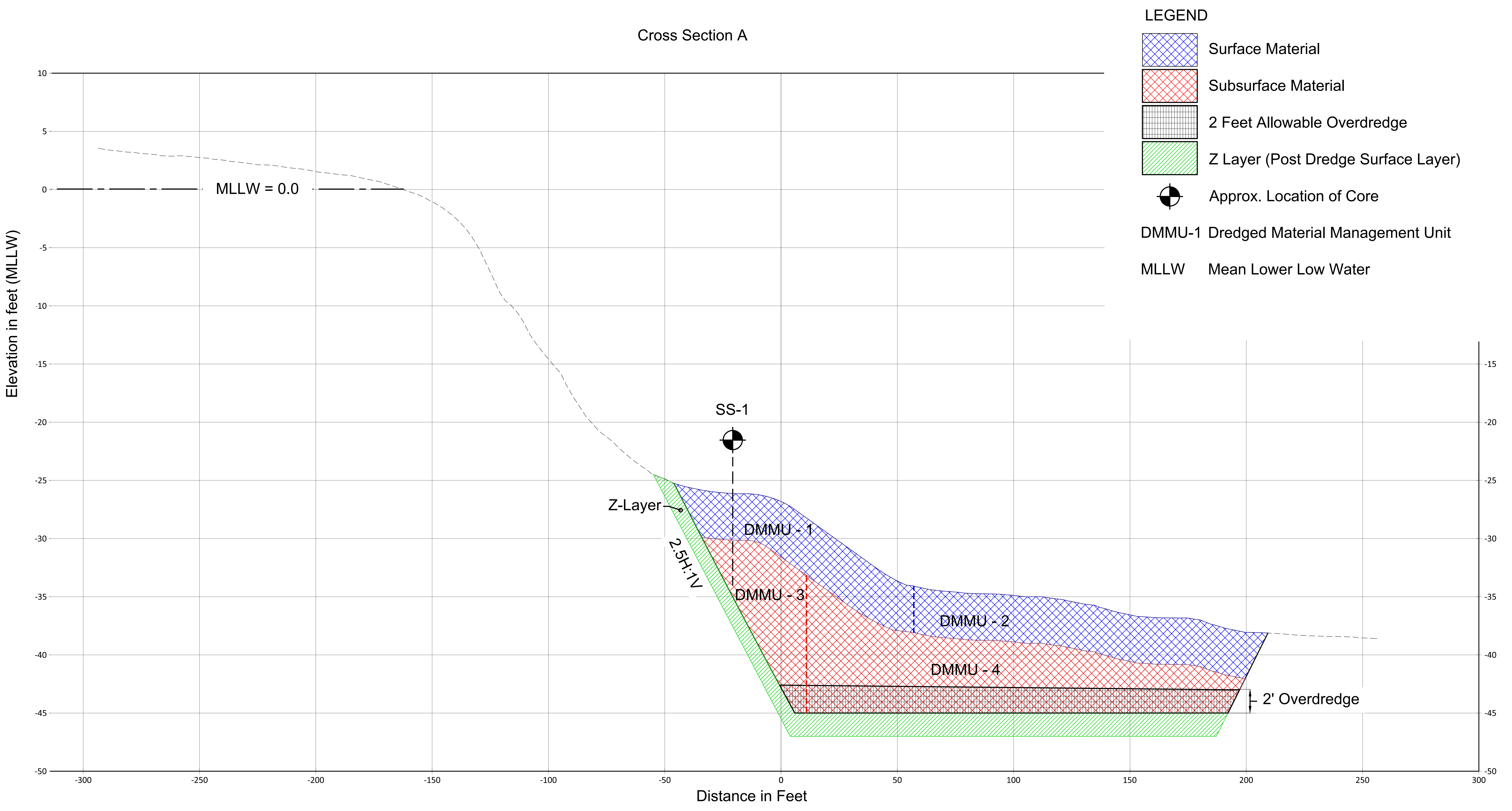

Proposed Grays Harbor Potash Export Facility -
Sampling and Analysis Plan Addendum

Sheet 3 - Cross Section A: STA 3+00

June 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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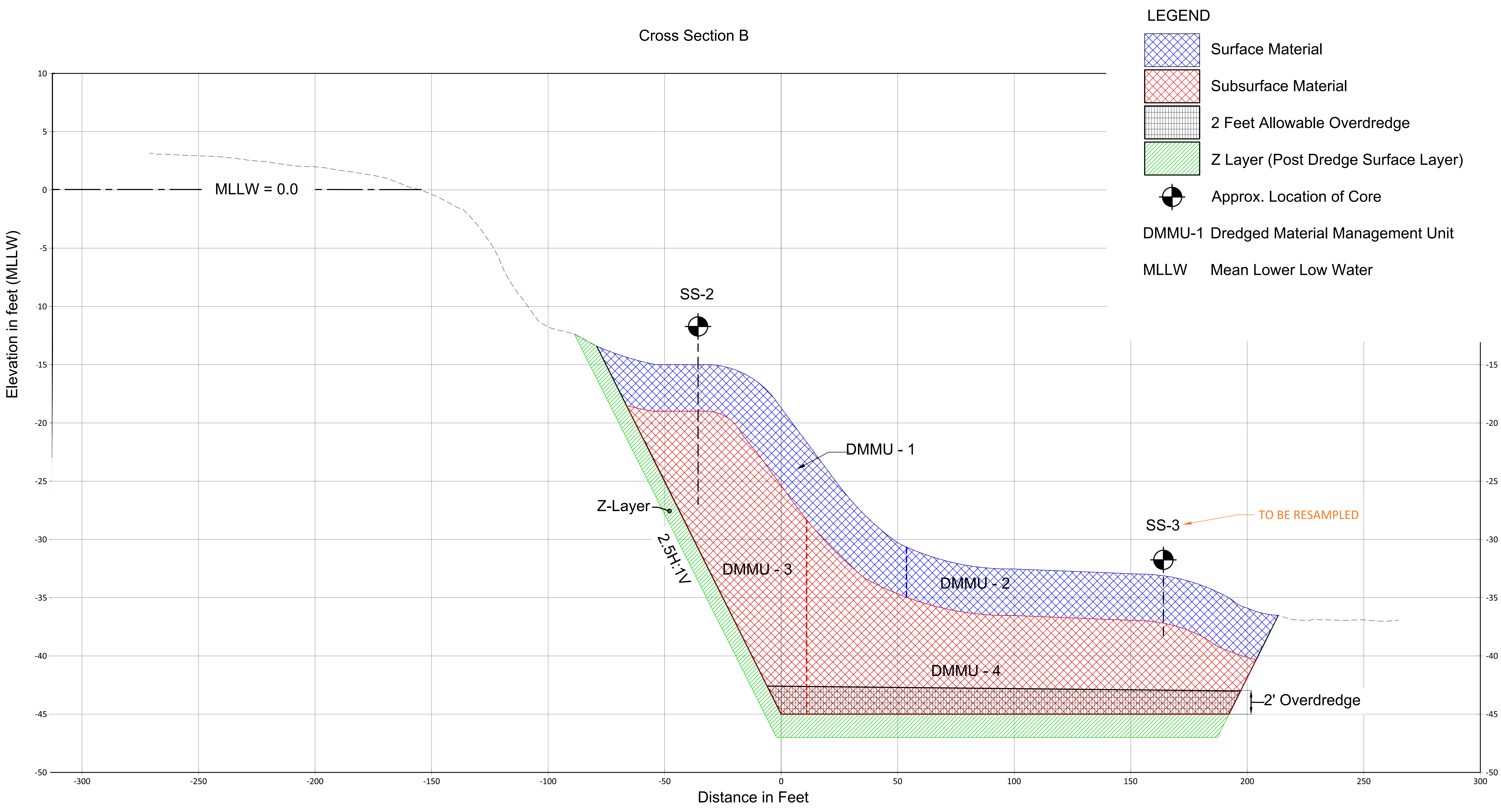
Actual Sample Locations Shown



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Proposed Grays Harbor Potash Export Facility -
Sampling and Analysis Plan Addendum

Sheet 4 - Cross Section B: STA 5+00

June 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:
1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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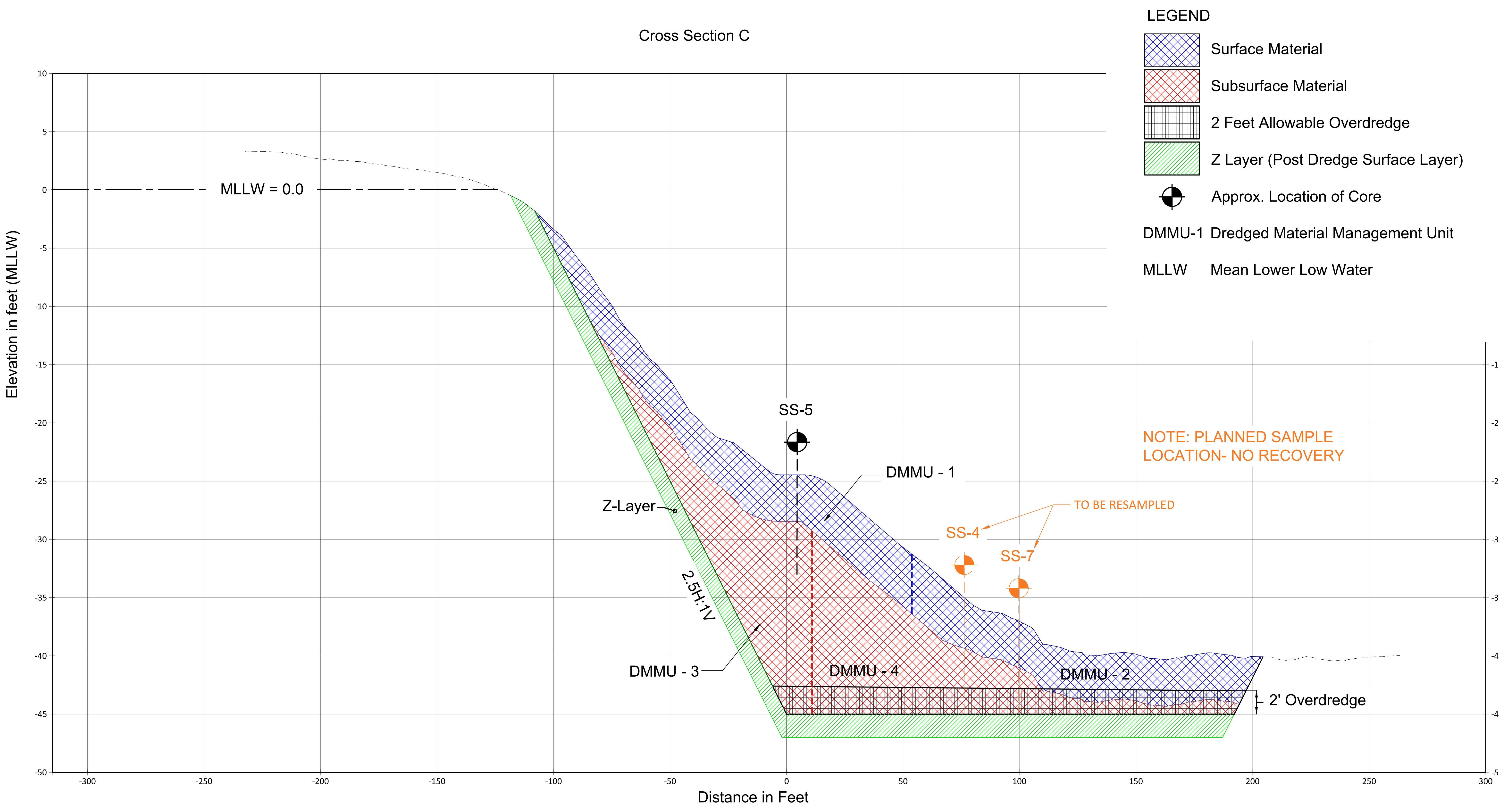
Actual Sample Locations Shown

Cross Section C



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Proposed Grays Harbor Potash Export Facility -
Sampling and Analysis Plan Addendum

Sheet 5 - Cross Section C: STA 9+00

June 2018

Horizontal Scale 1" = 40' (20 0 20 40 feet)
Vertical Scale 1" = 8' (4 0 4 8 feet)

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
3. HORIZONTAL DATUM: WASHINGTON STATE PLANE SOUTH (WSPS), NAD83, US FEET.
4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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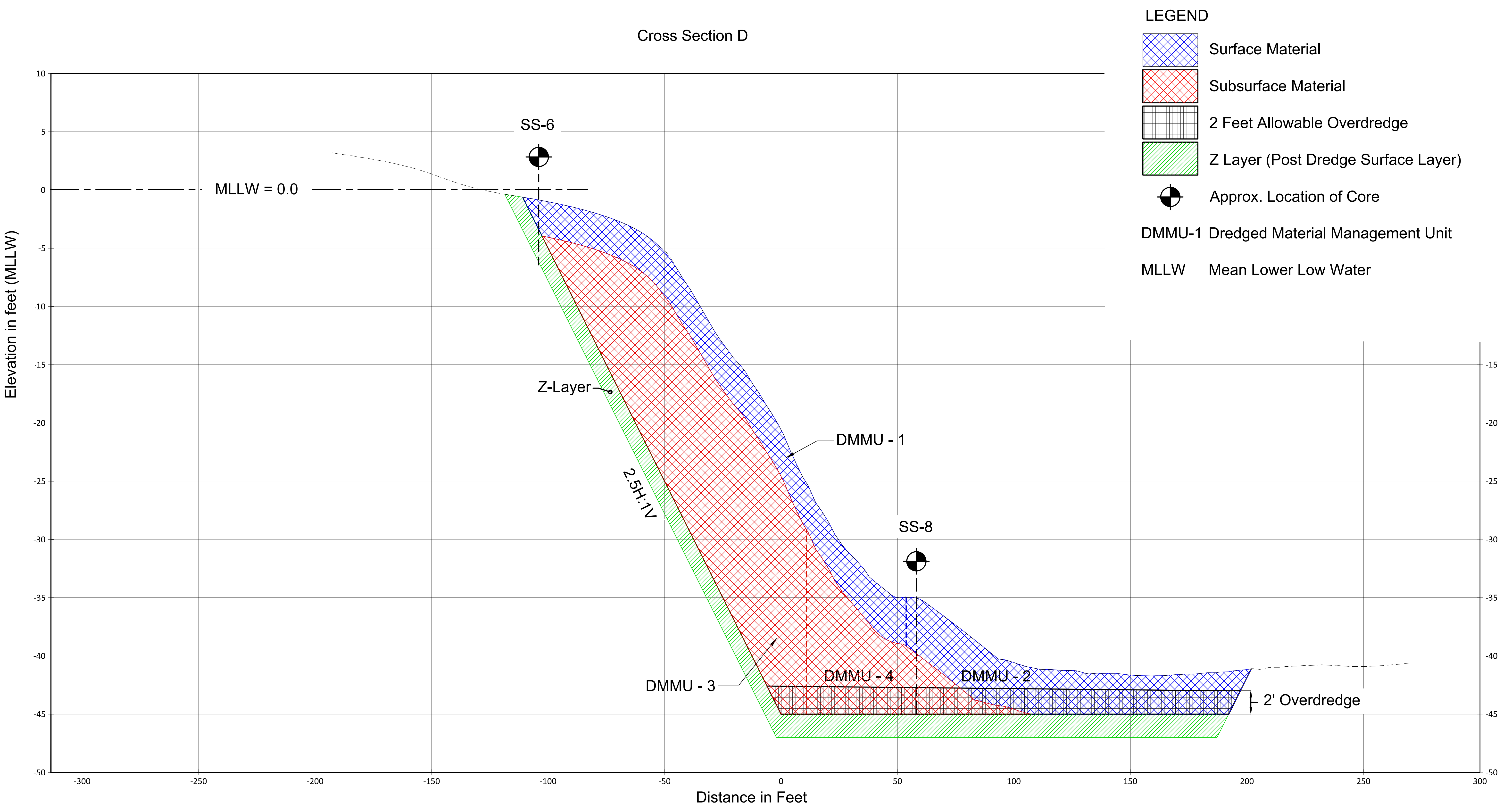
Actual Sample Locations Shown



Proposed Grays Harbor Potash Export Facility -
Sampling and Analysis Plan Addendum

Sheet 6 - Cross Section D: STA 12+00

June 2018

Horizontal Scale 1" = 40' 20 0 20 40 feet

Vertical Scale 1" = 8' 4 0 4 8 feet

GENERAL NOTES:

1. DREDGING UP TO 110,000 CY WILL BE REQUIRED TO ACCOMMODATE THE NEW FACILITY AND BERTH
2. HYDROGRAPHIC SURVEY DATA COLLECTED BY BERGLUND, SCHMIDT & ASSOCIATES DECEMBER 2017.
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4. VERTICAL DATUM: MEAN LOWER LOW WATER
5. BASE MAP WAS DEVELOPED BY AUSENCO ENGINEERING, 2017

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Actual Sample Locations Shown

Dredged Material Characterization Sampling and Analysis Plan Addendum

Proposed Grays Harbor Potash Export Facility
Hoquiam, Washington

Job Hazard Analysis and Operating Procedures – Sediment Core Sampling with Rossfelder P-3 Vibracore aboard the *Carolyn Dow* (June 2018)

Job Hazard Analysis and Operating Procedures

Sediment Core Sampling with Rossfelder P-3 Vibracore

Aboard the *Carolyn Dow*

June 2018

Prepared by
Research Support Services
for Berger ABAM

Contents

1. Introduction and Objectives

This Job Hazard Analysis addresses sediment core sampling using Research Support Services' Rossfelder P-3 Vibracore.

1.1 Overview of Activities

Vibracoring is an efficient technique for collecting cores in a range of marine sediments to moderate depths of penetration. Our vibracore system consists of a Rossfelder P-3 electric vibracorer, an optional buoyant frame, stainless steel core barrels with CAB (butyrate) liners, and a stainless steel core nose.

The sample is retained in the core tube by a one-way valve at the top of the core that closes when penetration stops, and by a sheet metal "core catcher" affixed to the core nose.

The liners are clear, allowing visual evaluation of the core in the field. They are left intact and full-length for transport to the processing area. The sample remains undisturbed from the time it is extracted until it is processed.

Positioning and navigation for sediment sample locations will be accomplished using a Trimble TSC-1, Pro XRS global positioning system (DGPS). The DGPS system consists of an antenna at the top of the vessels a-frame crane, and a GPS receiver and data logger in the wheelhouse. To confirm the accuracy/consistency of the readings, coordinates will be collected at a fixed point on site at the beginning and end of each day.

Operations will be conducted from the *Carolyn Dow,* a 36-foot aluminum landing craft with a bow-mounted a-frame. On-board personnel will consist of an RSS vessel operator and technician/deckhand, one Client field manager, and one Client technician.

A pre-departure briefing will be conducted to familiarize personnel of site-specific hazards, vessel hazards, safety equipment, emergency procedures and sampler deployment.

Sediment sampling will be conducted from an anchored boat at locations stipulated by the Client. The vibracore will be deployed over the bow of the vessel and brought aboard to remove the core liner containing the sample. If necessary, excess sediment will be released at the approximate location where it was collected.

1.2 Known Physical Site Hazards

Site-specific hazards include the possibility of current, surge and wind-driven waves. Working around structures in these conditions requires sustained attention to one's surroundings. Care must be taken when moving around the boat and working with suspended equipment.

The project area may be an active port facility, and vessel traffic can pose a hazard. It is important to maintain communication with the facility operators and with vessel operators in the area. Contact information and procedures for addressing vessel traffic issues will be addressed in the job-specific HASP provided by the Client.

1.3 Contaiminants of Concern

The primary routes of exposure in this marine environment are dermal exposure to and ingestion of sediment. Contact should be minimized through proper use of protective equipment and safety protocols as described below.

Potential contaminants in surface water at this site that may be encountered during dive operations include bacteria from stormwater runoff and oils on the surface of the water. Contact with and ingestion of river water is to be avoided.

Other potential contaminants may be present in sediment on the deck and sampling equipment. These chemicals will typically be bound in the wet solid matrix of the sediment.

Although sediments in the Duwamish River may contain contaminants, concentration levels in the water and sediment are expected to be below levels that will produce significant exposures when prescribed personal protective equipment (PPE) such as gloves and rubber boots are used. Limiting contact with sediments and water will further minimize any contact exposures.

The contaminants of concern in the material under the capped areas could include arsenic, copper, lead, mercury, zinc, dioxin/furans, PBDEs, TBT, PCBs, and PAHs.

Surface personnel will be working in an open-air environment and chemicals known or suspected to be present at the site pose a low risk for inhalation. Because the sediments will be wet, dust is not considered a significant issue. Care should be taken, however, to avoid breathing water mist associated with rinsing and decon procedures. The *Decontamination Zone* will be positioned downwind to reduce this risk.

2. Operating procedures

2.1 General Safety

1. Suspended equipment always presents a risk of injury from swinging and falling equipment.
2. Always make sure that personnel are clear of the equipment during deployment and retrieval.
3. Always make sure personnel are ready before lifting the sampler.
4. When the core head is lifted, never position yourself between it and other fixed objects to avoid crushing injuries.
5. When the core assembly is lifted, watch for wakes that might cause it to swing. If sea conditions are causing the boat to roll excessively, it is recommended that sampling be postponed until calmer conditions are present.

2.2 Deployment

1. The core barrel, liner and core nose are decontaminated according to protocols.
2. These are assembled, the core nose is riveted to the barrel, and the assembly clamped into the tool for deployment.
3. Operators lower the unit and drive the core into the sediment until the desired penetration or refusal are met. Measurements are taken before and after the drive to determine depth of penetration.

2.3 Retrieval

1. The core assembly is brought aboard and disassembled. Technicians remove the liner from the barrel and store it vertically until suspended sediments above the sample have settled.
2. An initial calculation of %-recovery is made by dividing the height of the recovered core by the penetration depth. In-field judgments of the acceptability of the core are often made based on this number. The calculation will be repeated before processing to account for further settling of the sample.
3. Head water is then carefully drained off by scoring the liner near the mudline. The excess liner is cut off and both ends are capped for transport to the processing facility.

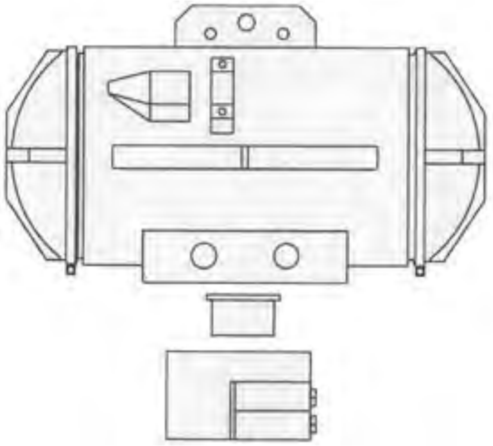
2.4 Processing

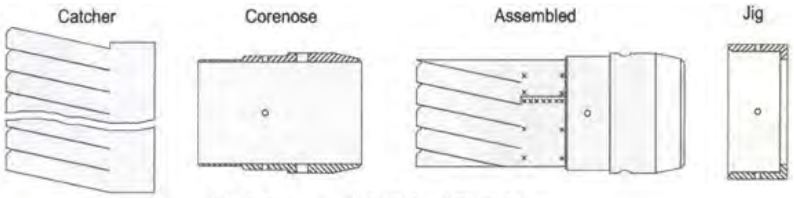
1. Once at the field lab, the core is placed in a trough and safely cut lengthwise with power shears.
2. Technicians make two cuts in the liner and remove a panel approximately a third of its diameter, revealing the sample. This exposure can be done incrementally to delay sub-sampling for volatiles lower in the core.

2.5 P-3 Specifications

WORKING DEPTH	To 600m (2,000 ft.)
POWER REQUIREMENTS	460v, 50-60 Hz, 3ph
AVERAGE RUNNING AMPS	4.5 Amps to 7 Amps
FORCE	16.0 KN to 24.0 KN on 60 Hz
VIBRATION FREQUENCY	3,450 vpm on 60 Hz

TYPICAL CORETUBE DIAMETER	4 in
TYPICAL CORETUBE LENGTH	5-15 ft
STANDARD CLAMP	102mm (4.0")
VIBROHEAD WEIGHT	68 kg (150 lbs)
TYPICAL SHIPPING WEIGHT	410 kg (900 lbs)
ADD WEIGHSTAND BALLAST	73 kg (160 lbs)
ADD GENERATOR IF REQUIRED	10.5 KVA, 272 kg (600 lbs)





4 Inch Corenose used with Hard Plastic Liner

3. Activity Hazard Analysis

The following table identifies potential hazards associated with this job and lists controls, or behaviors, that will be instituted to help prevent or manage safety incidents.

Job Steps	**Hazards**	**Controls**
Off-water transportation and mobilization	Vehicle collision	Seat belts shall be worn at all times by driver and passengers, and there shall be no hand-held cell phone use by the driver.
Boat launching and loading	Boat ramps and docks may be slippery The floating boat is heavy and can drift with wind and current	Move slowly and test the traction underfoot. Use dock cleats to control the boat once it has floated free of the trailer.
Loading equipment onto the boat at the ramp	Lifting injuries	Teamwork and proper lifting techniques are key to avoiding back strain and injuries from dropped equipment
	Slip, trips and falls	Docks and ramps may be slippery—move carefully. The boat will roll; the dock will not—take care stepping aboard. Load the boat evenly.
General boat safety	Falling overboard	All personnel shall wear personal flotation devices (PFD) when in boats or over water unless they are in a zipped drysuit. Hydrostatic PFDs will be checked daily to ensure they have a properly armed and functional kit, and that emergency pull tabs are easily accessible. A life ring and throw rope are on-hand to help retrieve someone who has fallen overboard
	Lifting injuries	Teamwork and proper lifting techniques are key to avoiding back strain and injuries from dropped equipment
	Slips, trips and falls	An orientation to the vessel will point out potential hazards such as slippery surfaces and protruding objects.

Job Steps	Hazards	Controls
	Boat wakes	All personnel shall keep watch for wakes from passing vessels and make others aware.
	Windlass injury	All personnel shall be instructed in windlass safety, and will remain clear whether or not they are in operation.
	Vessel traffic	Approaching vessels shall be contacted by VHF and flagged by the topside crew. In areas with vessel traffic systems, VTS should be made aware of the dive operations and may request participation in the traffic system or notification of diver entries and exits.
	Heat and cold stress	Workers shall be dressed appropriately for the conditions and communicate if they are too hot or cold. Workers shall monitor each other's comfort level throughout the day and drink plenty of fluids. Air conditioning is available in the wheelhouse to help avoid overheating. The wheelhouse can be heated if necessary.
	Fire	The fire extinguisher will be pointed out during the safety briefing. Avoid refueling a hot generator or power pack.
	Lightening	Discontinue work and find shelter. Confirm that the storm is moving away and no lightening is present before resuming work.
Conducting sampling operations in general	Injuries from the a-frame and hydraulics	Pinch points will be pointed out, and hardhats will be required when a-frame is in motion or suspended loads are present.
	Overhead equipment	Wear a hard hat on deck. Be aware and watch out for each other. Keep loads as low as possible.
	Falling equipment	Keep hands and feet from under loads. Keep heavy equipment on-deck when possible.
	Hot equipment	Keep hands away from power pack exhaust.

Job Steps	Hazards	Controls
		Be aware the power pack cabinet may be hot.
	Abrasive deck paint	Take care with knuckles when setting things down.
	Loss of concentration while focusing on sampling	Maintain awareness of the surroundings at all times. Point out hazards that other’s may not see.
	Tiredness at the end of the day can lead to mistakes and injury during end-of-day procedures	Anticipate the need for attention to safety even after the sampling is done.
Issues specific to Vibracore deployment	Accumulation of sediment on deck can make it slippery	Be aware that the deck may become more slippery during sampling, especially unpainted areas like hatch covers. Keep the deck rinsed of sediment between samples.
	The equipment could drop unexpectedly	In the unlikely event of a winch line failure, the coring assembly could drop abruptly into the water or onto the deck. Technicians must take care not to become entangled in cabling and to stay out from underneath suspended equipment.
	The coring assembly is heavy	When guiding the equipment onto the deck or into the water, be mindful of the possibility it could swing in response to waves or a boat wake. Keep hands on top, feet and legs clear when the equipment is suspended above the deck.

4. Equipment Considerations

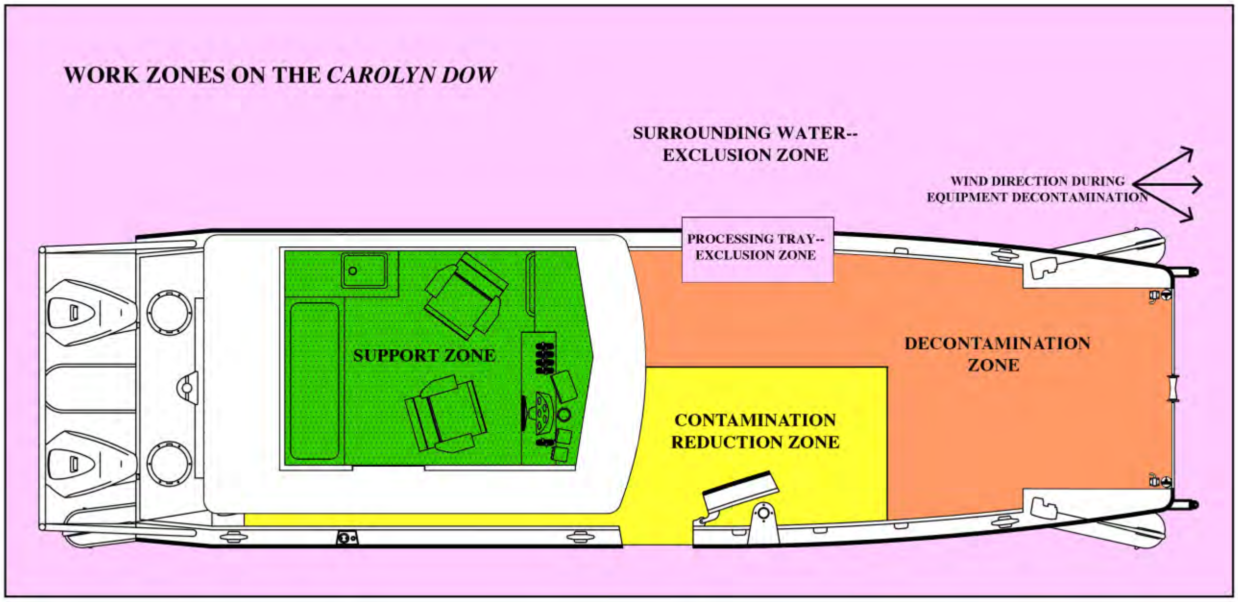
Equipment to be Used	**Training Requirements**	**Inspection Requirements**
Boats	Boaters safety training for smaller vessels, USCG Master for Carolyn Dow. First aid, CPR, AED, O2 admin for all crew	Daily inspection for functionality, worn hoses, leaks, etc. Check safety gear.
Navigation and survey electronics	Experienced and familiar with component function and operating procedures	Visual inspection, pre-operations test to ensure proper operation
Sediment samplers	Experienced and familiar with safe operating procedures. Appropriate OSHA training for the expected contaminants per HASP	Visual inspection, pre-operations test to ensure proper operation
Company vehicles and trailers	Licensed appropriately for the vehicle and familiar with towing procedures	Annual DOT inspection Pre-trip inspections and daily fluid checks

5. Work Zones

A small boat presents challenges to the textbook contamination management model because of the limited space available for transitioning from dirty to cleaner environments. Aboard the *Carolyn Dow* there will be four areas:

1. An *Exclusion Zone* represented by the surrounding water and the processing tray.
2. *A Decontamination Zone* including the bow door and part of the foredeck for rinsing sediment and site water from technicians and storing re-useable equipment.
3. A *Contamination Reduction Zone* that includes the rest of the deck, but not the wheelhouse, where care will be taken to keep the deck clean of sediment through frequent rinsing.
4. A *Support Zone* in the wheelhouse, where everyone entering will have been through the potable-water rinsing process on deck and rinsed their boots again before entering.

The diagram below shows the boat in plan view and the locations of these zones:



Ryckman, April

From: Warner, Lauran C CIV USARMY CENWS (US) <Lauran.C.Warner@usace.army.mil>
Sent: Monday, July 30, 2018 2:45 PM
To: Ryckman, April; Roberts, Grace; Fisher, Sally; England, Victoria
Cc: Valerie.Bond@bhpbilliton.com; Berzolla, Heather; Laura Inouye (lino461@ecy.wa.gov); Celia Barton (celia.barton@dnr.wa.gov); Justine Barton (barton.justine@epa.gov)
Subject: BHP Grays Harbor revised SAPA - approval

Grace, Victoria, Sally and Victoria: Thank you all for your prompt responses to our questions.

The SAP Addendum is approved as received on July 19, 2018, with the following caveats:

1. If core samples are unable to reach the Z-layer, we understand that the bottom foot of a core sample will be archived for use as a potential Z-sample, and the DMMP supports that approach. However, we will need to reserve judgment on the applicability of a given sample to serve as a Z-layer dependent on details such as the core depth achieved, core logs, grain size data, consideration of past sampling results, or other pertinent details.
2. Submittal of a signed signature page for subcontractors prior to field mobilization.

Please keep us updated on your sampling plans as one or more of us will likely join you in the field for at least one day.

Thanks - Lauran

Lauran Warner
Biologist - Dredged Material Management Office
lauran.c.warner@usace.army.mil
D: 206-764-6550
M: 206-383-4143

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix D

February 2018 - Diver Collected Bore Graphs

Mudmole™ Bore Log

Project: Grays Habor Dredge Chaaracterization **Station:** SS-1

Project No: BergerABAM **Position:** NAD83 WA S

Collected by: GSM 615440 Northing

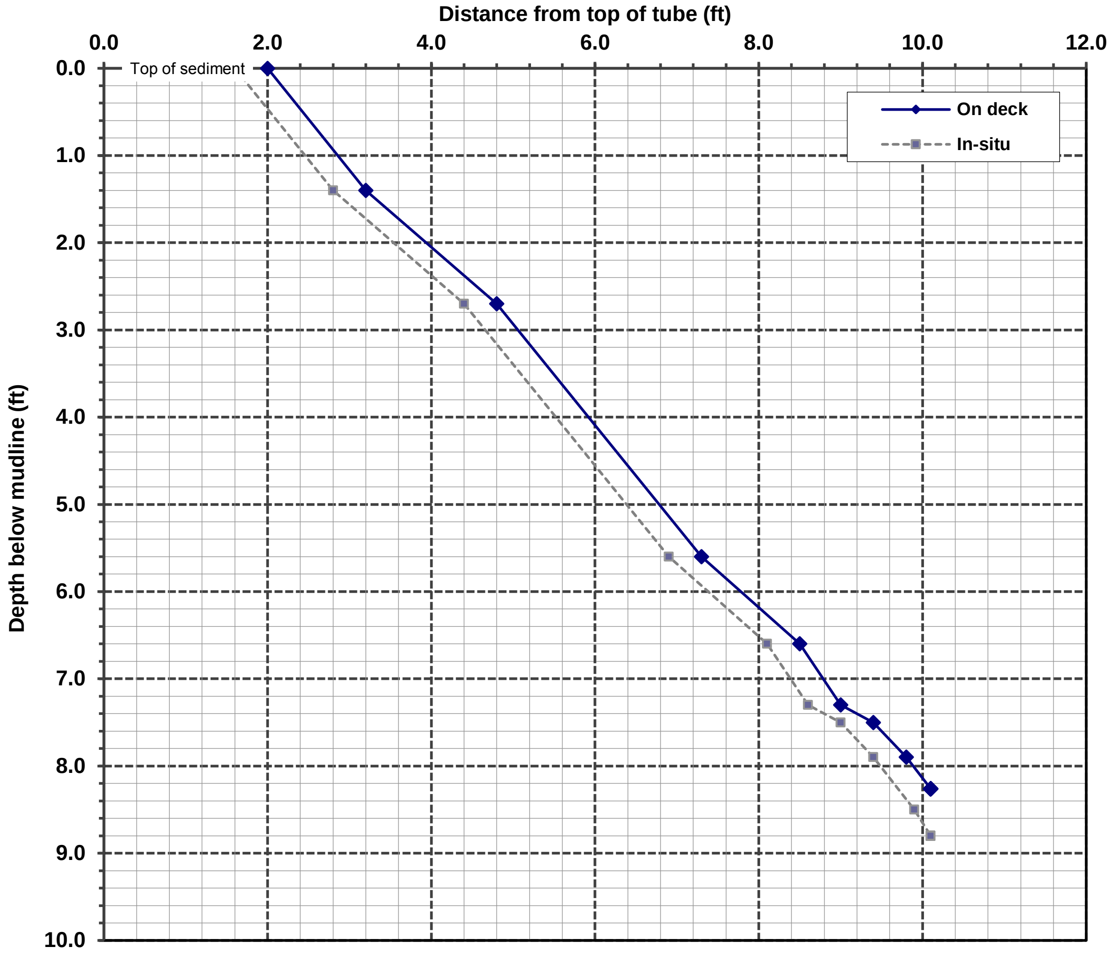
Date: 2/26/2018 **Time:** 16:50 787469 Easting

Water depth: 26.0 ft **Mudline:** -25.9 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: N/A

Driven to refusal



Penetration 8.8 ft/ On deck recovery 8.1 ft = 92% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-1.4	1.2	86%
1.4-2.7	1.6	123%
2.7-5.6	2.5	86%
5.6-6.6	1.2	120%
6.6-7.3	0.5	71%
7.3-7.5	0.4	200%
7.5-7.9	0.4	100%
7.9-8.5	0.5	83%
8.5-8.8	0.2	67%

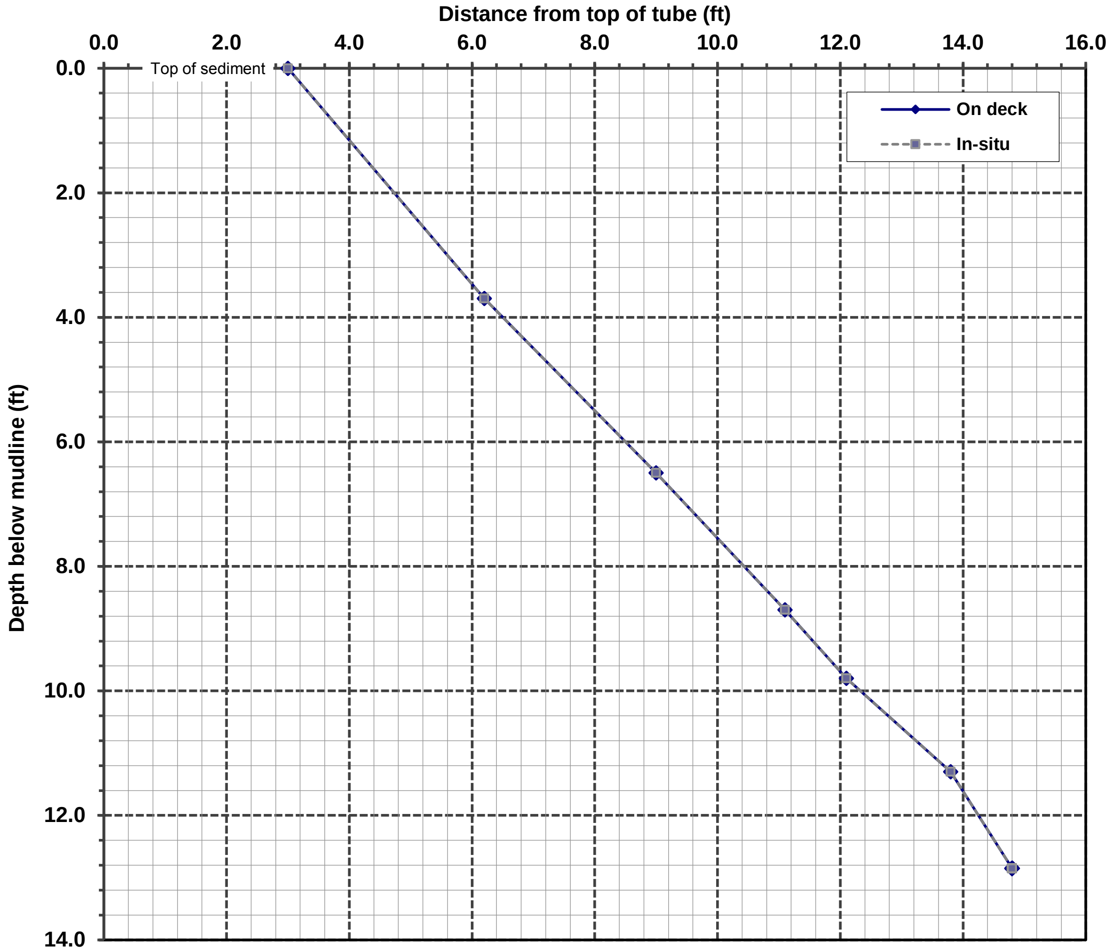
Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	2
1	2.86
2	3.94
3	5.06
4	5.92
5	6.78
6	7.78
7	8.79
8	9.88
9	No sample
10	No sample
11	No sample
12	No sample
13	No sample
14	No sample
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

Mudmole™ Bore Log

Project: Grays Habor Dredge Characterization **Station:** SS-2
Project No: BergerABAM **Position:** NAD83 WA S
Collected by: GSM 615425 Northing
Date: 2/26/2018 **Time:** 15:00 787743 Easting
Water depth: 15.0 ft **Mudline:** -15.0 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: N/A



Penetration 12.85 ft/ On deck recovery 11.8 ft = 92% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-3.7	3.2	86%
3.7-6.5	2.8	100%
6.5-8.7	2.1	95%
8.7-9.8	1	91%
9.8-11.3	1.7	113%
11.3-12.85	1	65%

Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	3
1	3.86
2	4.73
3	5.59
4	6.50
5	7.50
6	8.50
7	9.48
8	10.43
9	11.37
10	12.33
11	13.46
12	14.25
13	No sample
14	No sample
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

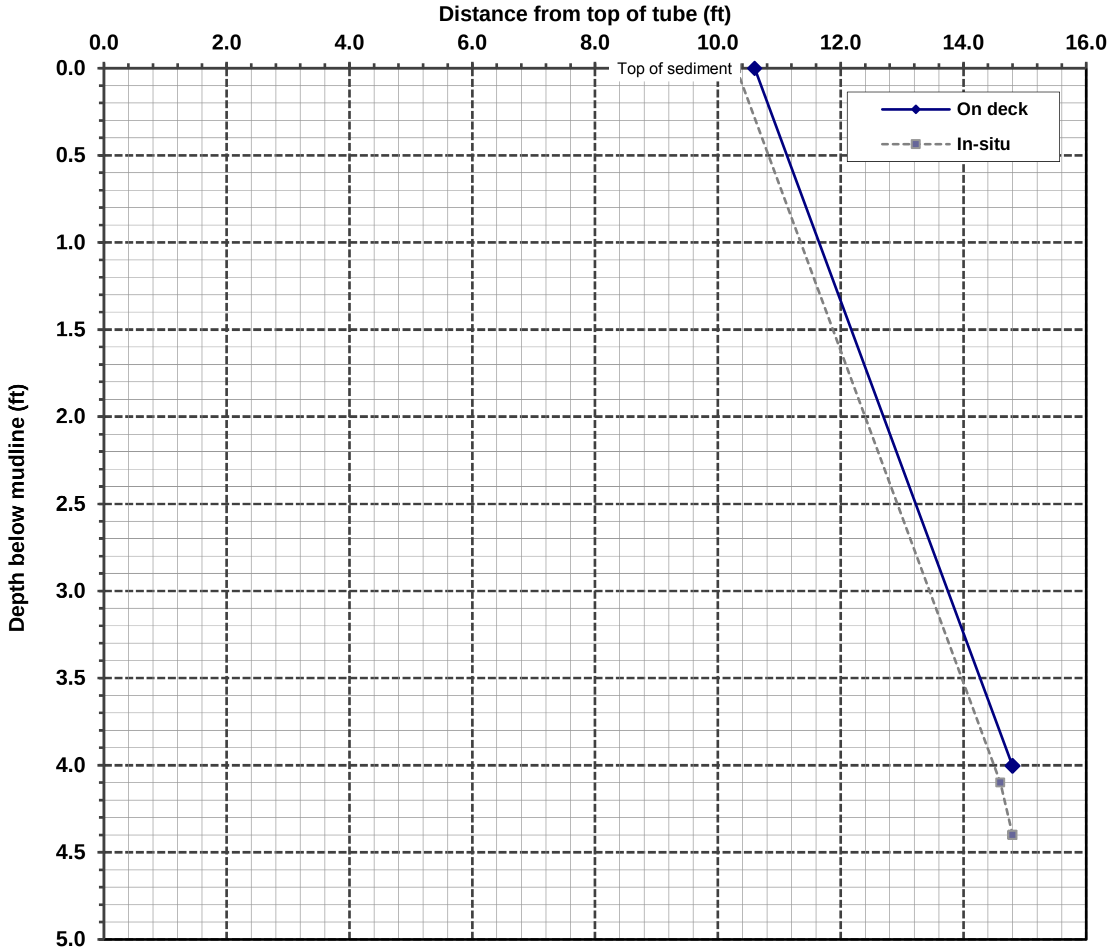
Mudmole™ Bore Log

Project: Grays Habor Dredge Chaaracterization **Station:** SS-3
Project No: BergerABAM **Position:** NAD83 WA S
Collected by: GSM 615258 Northing
Date: 2/27/2018 **Time:** 10:07 787616 Easting
Water depth: 43.0 ft **Mudline:** -33.0 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: Windy

Driven to refusal.



Penetration 4.4 ft/ On deck recovery 4.2 ft = 95% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-4.1	4.3	105%
4.1-4.4	0.2	67%

Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	10.6
1	11.65
2	12.70
3	13.75
4	14.80
5	No sample
6	No sample
7	No sample
8	No sample
9	No sample
10	No sample
11	No sample
12	No sample
13	No sample
14	No sample
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

Mudmole™ Bore Log

Project: Grays Habor Dredge Chaaracterization **Station:** SS-5

Project No: BergerABAM **Position:** NAD83 WA S

Collected by: GSM 615366 Northing

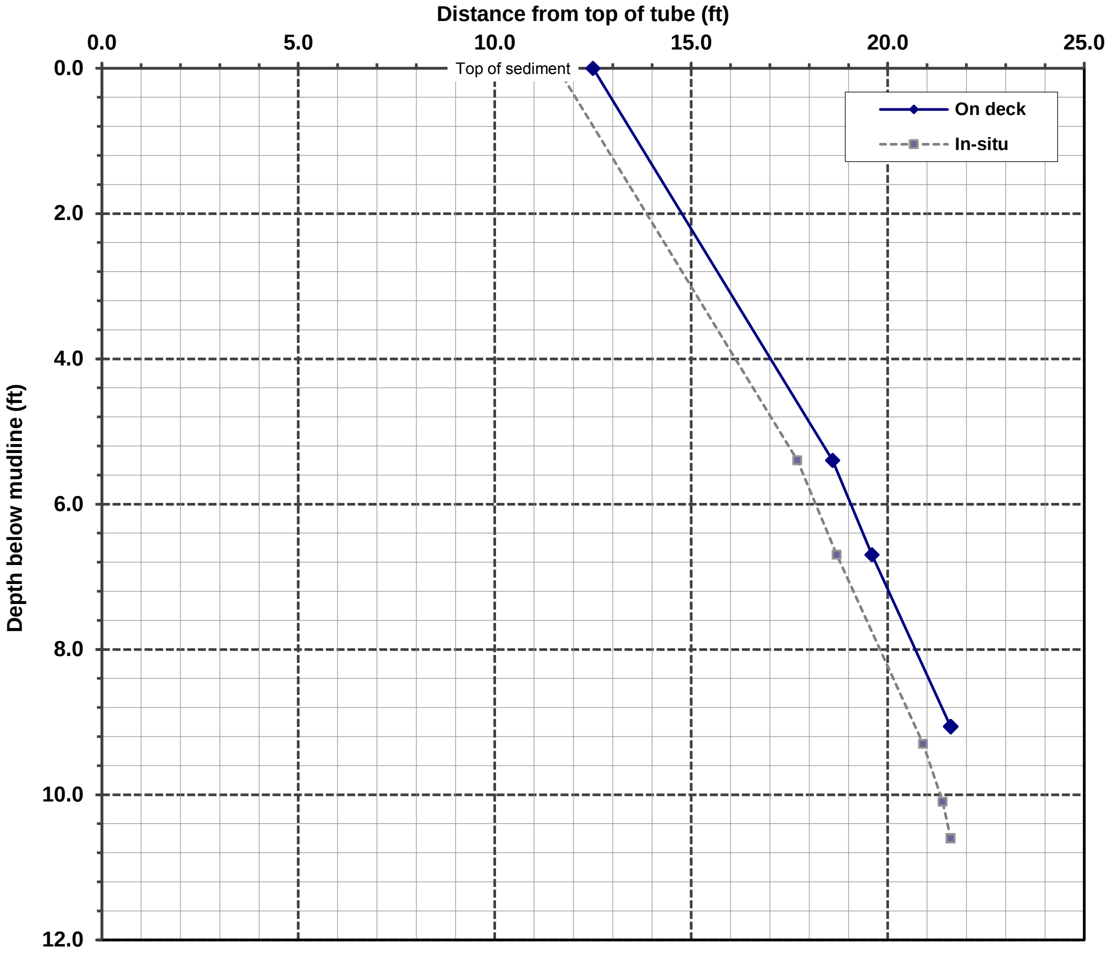
Date: 5/2/2018 **Time:** 11:52 788095 Easting

Water depth: 33.0 ft **Mudline:** -24.0 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: N/A

Driven to refusal



Penetration 10.6 ft/ On deck recovery 9.1 ft = 86% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-5.4	6.1	113%
5.4-6.7	1	77%
6.7-9.3	2.2	85%
9.3-10.1	0.5	62%
10.1-10.6	0.2	40%

Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	12.5
1	13.63
2	14.76
3	15.89
4	17.02
5	18.15
6	19.06
7	19.85
8	20.70
9	21.55
10	No sample
11	No sample
12	No sample
13	No sample
14	No sample
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

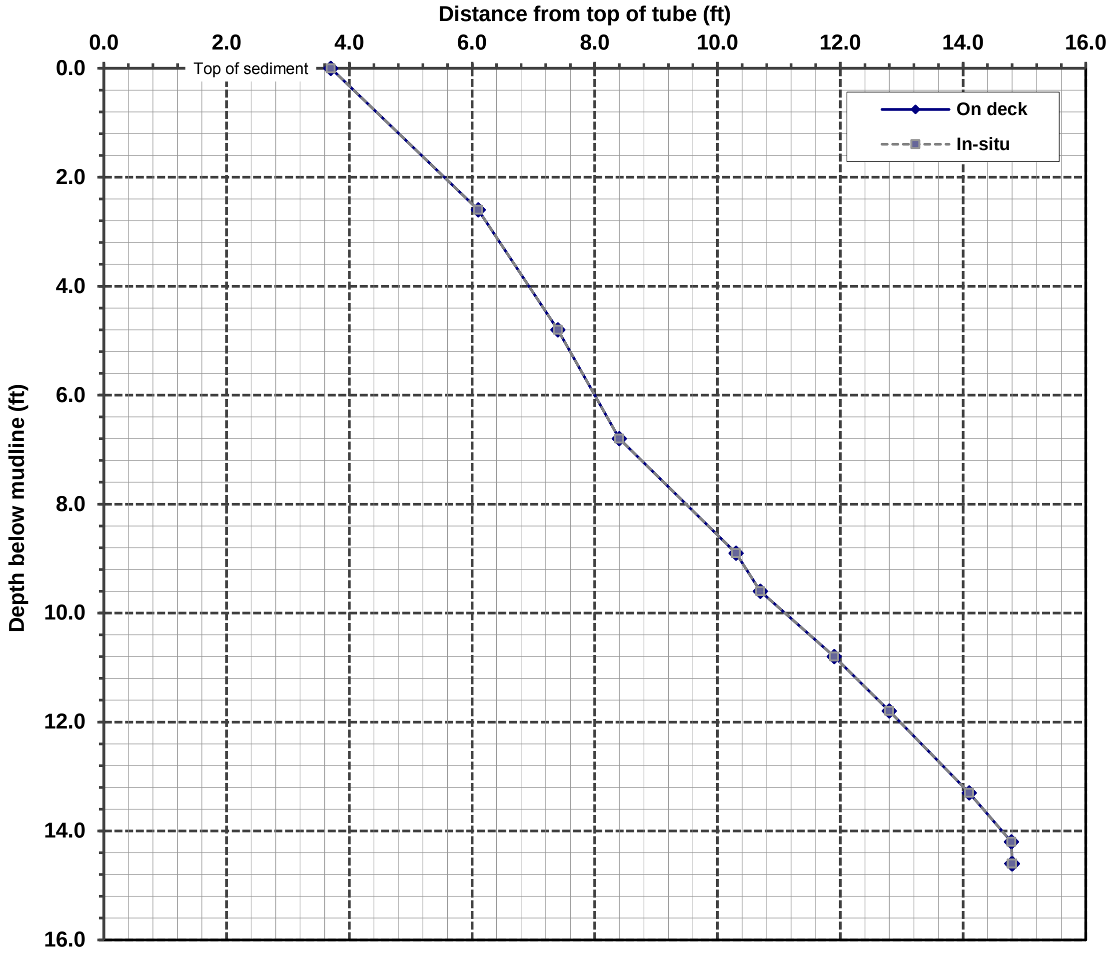
Mudmole™ Bore Log

Project: Grays Habor Dredge Chaaracterization **Station:** SS-6
Project No: BergerABAM **Position:** NAD83 WA S
Collected by: GSM 615431 Northing
Date: 5/2/2018 **Time:** 13:11 788362 Easting
Water depth: 5.6 ft **Mudline:** -0.6 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: N/A

Moved off station. 3rd attempt



Penetration 14.6 ft/ On deck recovery 11.1 ft = 76% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-2.6	2.4	92%
2.6-4.8	1.3	59%
4.8-6.8	1	50%
6.8-8.9	1.9	90%
8.9-9.6	0.4	57%
9.6-10.8	1.2	100%
10.8-11.8	0.9	90%
11.8-13.3	1.3	87%
13.3-14.2	0.69	77%
14.2-14.6	0.01	2%

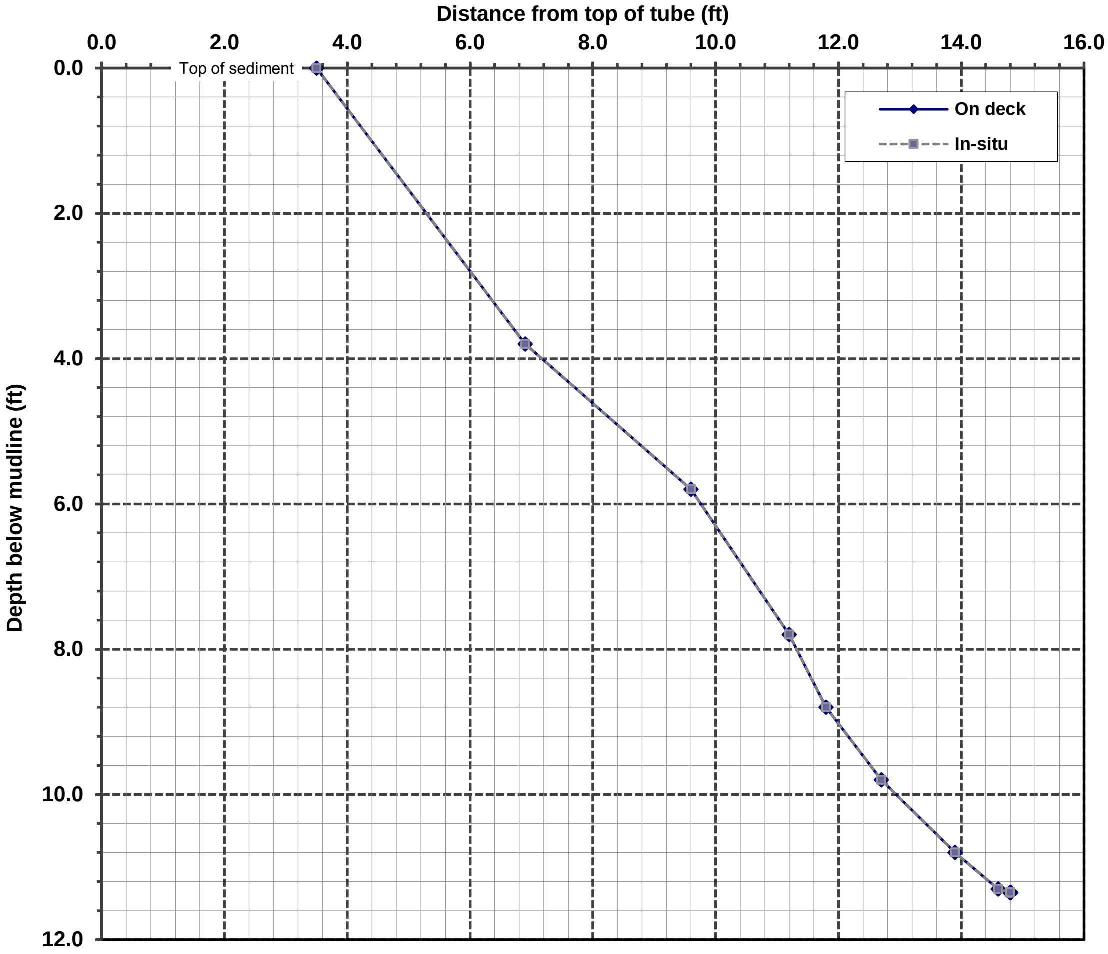
Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	3.7
1	4.62
2	5.55
3	6.34
4	6.93
5	7.50
6	8.00
7	8.58
8	9.49
9	10.36
10	11.10
11	12.08
12	12.97
13	13.84
14	14.64
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

Mudmole™ Bore Log

Project: Grays Habor Dredge Chaaracterization **Station:** SS-8
Project No: BergerABAM **Position:** NAD83 WA S
Collected by: GSM 615266 Northing
Date: 2/26/2018 **Time:** 10:05 788387 Easting
Water depth: 45.0 ft **Mudline:** -35.0 ft MLLW (estimated using tide tables)

Place Field ID Label Here

Weather/Comments: N/A



Penetration 11.35 ft/ On deck recovery 11.3 ft = 100% Recovery

Penetration interval (ft)	Interval recovery (ft)	Percent recovery
0-3.8	3.4	89%
3.8-5.8	2.7	135%
5.8-7.8	1.6	80%
7.8-8.8	0.6	60%
8.8-9.8	0.9	90%
9.8-10.8	1.2	120%
10.8-11.3	0.7	140%
11.3-11.35	0.2	400%

Depth below mudline (ft)	Distance from top of tube (ft)
Mudline	3.5
1	4.39
2	5.29
3	6.18
4	7.17
5	8.52
6	9.76
7	10.56
8	11.32
9	11.98
10	12.94
11	14.18
12	No sample
13	No sample
14	No sample
15	No sample
16	No sample
17	No sample
18	No sample
19	No sample
20	No sample

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix E

Bore Logs and Photographs

SOIL CLASSIFICATION CHART

MAJOR DIVISONS			SYMBOLS		TYPICAL DESCRIPTIONS
			GRAPH	LETTER	
COARSE GRAINED SOILS MORE THAN 50% RETAINED ON NO. 200 SIEVE	GRAVEL AND GRAVELLY SOILS MORE THAN 50% OF COARSE FRACTION RETAINED ON NO. 4 SIEVE	CLEAN GRAVELS (LITTLE OR NO FINES)		GW	WELL-GRADED GRAVELS, GRAVEL-SAND MIXTURES
				GP	POORLY-GRADED GRAVELS, GRAVEL-SAND MIXTURES
		GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)		GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES
				GC	CLAYEY GRAVELS, GRAVEL-SAND-CLAY MIXTURES
	SAND AND SANDY SOILS MORE THAN 50% OF COARSE FRACTION PASSING NO. 4 SIEVE	CLEAN SANDS (LITTLE OR NO FINES)		SW	WELL-GRADED SANDS, GRAVELLY SAND
				SP	POORLY-GRADED SANDS, GRAVELLY SAND
		SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)		SM	SILTY SANDS, SAND-SILT MIXTURES
				SC	CLAYEY SANDS, SAND-CLAY MIXTURES
FINE GRAINED SOILS MORE THAN 50% PASSING NO. 200 SIEVE	SILTS AND CLAYS	LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS, ROCK FLOUR, CLAYEY SILTS WITH SLIGHT PLASTICITY
				CL	INORGANIC SILTS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
				OL	INORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
	SILTS AND CLAYS	LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS SILTY SOILS
				CH	INORGANIC CLAYS OF HIGH PLASTICITY
				OH	ORGANIC CLAYS AND SILTS OF MEDIUM TO HIGH PLASTICITY
HIGHLY ORGANIC SOILS				PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENTS

NOTE: Multiple symbols are used to indicate borderline or dual soil classifications.

ADDITIONAL MATERIAL SYMBOLS

SYMBOLS		TYPICAL DESCRIPTIONS
GRAPH	LETTER	
	CC	CEMENT CONCRETE
■	AC	ASPHALT CONCRETE
▒	CR	CRUSHED ROCK/QUARRY SPALLS
	TS	TOPSOILS/FOREST DUFF/SOD

▽	MEASURED GROUNDWATER LEVEL IN EXPLORATION, WELL, OR PIEZOMETER
▼	GROUNDWATER OBSERVED AT TIME OF EXPLORATION
≡	MEASURED FREE PRODUCT IN WELL OR PIEZOMETER
▼	MEASURED FREE PRODUCT IN WELL OR PIEZOMETER

STRATIGRAPHIC CONTACT

———	DISTINCT CONTACT BETWEEN SOIL STRATA OR GEOLOGIC UNITS
／	GRADUAL CHANGE BETWEEN SOIL STRATA OR GEOLOGIC UNITS
– – – – – –	APPROXIMATE LOCATION OF SOIL STRATA CHANGE WITHIN A GEOLOGIC SOIL UNIT

SHEEN CLASSIFICATION

NS	NO VISIBLE SHEEN
SS	SLIGHT SHEEN
MS	MODERATE SHEEN
HS	HIGH SHEEN
NT	NOT TESTED

NOTE: The reader must refer to the discussion in the report text and the logs of explorations for a proper understanding of subsurface conditions. Descriptions on the logs apply only at the specific exploration locations and at the time the explorations were made; they are not warranted to be represented of subsurface conditions at other locations or times.

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

Date Drilled: 2/26/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 8	Surface Elevation (ft): -25.9	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
	▨			ML	Dark gray silt with sand lenses (stiff)			
2	▨							
4	▨							
6	▨							
7	▨			SM	Sand with silt			
8	▨			ML	Silt with sand lenses			

LOG OF BORING # SS-1

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility

Project Location: Hoquiam, WA

Project Number: A17.0202.00

bgs = below ground surface

ft = feet

Date Drilled: 2/26/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 14.25	Surface Elevation (ft): -15	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
				ML	Dark gray silt with sand lenses (stiff)			
2								
4								
6								
8								
10								
12								

LOG OF BORING # SS-2

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 2/27/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 4	Surface Elevation (ft): -33	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
2	▨			ML	Dark Gray silt with sand lenses (stiff)			
4								
								Subsurface recovery unachievable

LOG OF BORING # SS-3

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 2/27/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 9	Surface Elevation (ft): -24	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0	▨			ML	Dark gray silt with sand lenses			
2								
4								
6								
8								
9								

LOG OF BORING # SS-5

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 2/27/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 14	Surface Elevation (ft): -0.6	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
				ML	Dark gray silt with sand lenses (soft to dense)			
2								
4								
6								
8								
10								
11								
				SP	Dark gray sand (dense)			
12								
14								

LOG OF BORING # SS-6

BergerABAM	Project: Proposed Grays Harbor Potash Export Facility	bgs = below ground surface ft = feet
	Project Location: Hoquiam, WA	
	Project Number: A17.0202.00	

Date Drilled: 2/26/18	Logged By: GR	Checked By: SLF
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: MudMole
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 11	Surface Elevation (ft): -35	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
	▨			ML	Dark gray silt with sand (soft to medium stiff)			
2								
4								
6								
8								
10								
	▨			SP-SM	Sand with silt pockets (medium dense to dense)			
11								

LOG OF BORING # SS-8

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility

Project Location: Hoquiam, WA

Project Number: A17.0202.00

bgs = below ground surface

ft = feet

SOIL CLASSIFICATION CHART

MAJOR DIVISONS			SYMBOLS		TYPICAL DESCRIPTIONS
			GRAPH	LETTER	
COARSE GRAINED SOILS	GRAVEL AND GRAVELY SOILS	CLEAN GRAVELS (LITTLE OR NO FINES)	▨	GW	WELL-GRADED GRAVELS, GRAVEL-SAND MIXTURES
			▨	GP	POORLY-GRADED GRAVELS, GRAVEL-SAND MIXTURES
	MORE THAN 50% OF COARSE FRACTION RETAINED ON NO. 4 SIEVE	GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)	▨	GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES
			▨	GC	CLAYEY GRAVELS, GRAVEL-SAND-CLAY MIXTURES
	SAND AND SANDY SOILS	CLEAN SANDS (LITTLE OR NO FINES)	▨	SW	WELL-GRADED SANDS, GRAVELLY SAND
MORE THAN 50% RETAINED ON NO. 200 SIEVE			▨	SP	POORLY-GRADED SANDS, GRAVELLY SAND
	MORE THAN 50% OF COARSE FRACTION PASSING NO. 4 SIEVE	SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)	▨	SM	SILTY SANDS, SAND-SILT MIXTURES
			▨	SC	CLAYEY SANDS, SAND-CLAY MIXTURES
FINE GRAINED SOILS	SILTS AND CLAYS	LIQUID LIMIT LESS THAN 50	▨	ML	INORGANIC SILTS, ROCK FLOUR, CLAYEY SILTS WITH SLIGHT PLASTICITY
			▨	CL	INORGANIC SILTS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
			▨	OL	INORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
	SILTS AND CLAYS	LIQUID LIMIT GREATER THAN 50	▨	MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS SILTY SOILS
MORE THAN 50% PASSING NO. 200 SIEVE			▨	CH	INORGANIC CLAYS OF HIGH PLASTICITY
			▨	OH	ORGANIC CLAYS AND SILTS OF MEDIUM TO HIGH PLASTICITY
HIGHLY ORGANIC SOILS			▨	PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENTS

NOTE: Multiple symbols are used to indicate borderline or dual soil classifications.

ADDITIONAL MATERIAL SYMBOLS

SYMBOLS		TYPICAL DESCRIPTIONS
GRAPH	LETTER	
▨	CC	CEMENT CONCRETE
■	AC	ASPHALT CONCRETE
▒	CR	CRUSHED ROCK/QUARRY SPALLS
▦	TS	TOPSOILS/FOREST DUFF/SOD

▽	MEASURED GROUNDWATER LEVEL IN EXPLORATION, WELL, OR PIEZOMETER
▼	GROUNDWATER OBSERVED AT TIME OF EXPLORATION
≡	MEASURED FREE PRODUCT IN WELL OR PIEZOMETER
▼	MEASURED FREE PRODUCT IN WELL OR PIEZOMETER

STRATIGRAPHIC CONTACT

────	DISTINCT CONTACT BETWEEN SOIL STRATA OR GEOLOGIC UNITS
／	GRADUAL CHANGE BETWEEN SOIL STRATA OR GEOLOGIC UNITS
– – – – – –	APPROXIMATE LOCATION OF SOIL STRATA CHANGE WITHIN A GEOLOGIC SOIL UNIT

SHEEN CLASSIFICATION

NS	NO VISIBLE SHEEN
SS	SLIGHT SHEEN
MS	MODERATE SHEEN
HS	HIGH SHEEN
NT	NOT TESTED

NOTE: The reader must refer to the discussion in the report text and the logs of explorations for a proper understanding of subsurface conditions. Descriptions on the logs apply only at the specific exploration locations and at the time the explorations were made; they are not warranted to be represented of subsurface conditions at other locations or times.

Date Drilled: 8/15/18	Logged By: GR	Checked By: AR
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: Vibracore
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 7	Surface Elevation (ft): -35.1	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
	▨			ML	Gray silt (very soft to soft)			No odor
								Shrimp at top of core when opened
3				SM	Gray silty fine sand (medium stiff)			
4								
6								
				ML	Gray silt with fine sand lenses (soft)			
7								

LOG OF BORING # SS-3A

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 8/15/18	Logged By: GR	Checked By: AR
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: Vibracore
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 8.3	Surface Elevation (ft): -36	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
				ML	Gray silt (soft)			Original recovery was 8.3 ft, ~1 ft lost when core tube was cut open due to it bending when being pushed into sediment
2								
4				ML	Gray silt with fine sand lenses (medium stiff to stiff)			
6								
7								

LOG OF BORING # SS-3B

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 8/14/18	Logged By: GR	Checked By: AR
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: Vibracore
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 10.5	Surface Elevation (ft): -24.57	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0	▨			ML	Gray sandy silt (very soft to stiff)			
2								
4								
6								Wood chips at 6 ft (no odor), archive samples taken below and above the wood chips
8								
10								

LOG OF BORING # SS-4B

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility
Project Location: Hoquiam, WA
Project Number: A17.0202.00

bgs = below ground surface
ft = feet

Date Drilled: 8/14/18	Logged By: GR	Checked By: AR
Drilling Contractor: N/A	Drilling Method: N/A	Sampling Methods: Vibracore
Auger Data: N/A	Hammer Data: N/A	Groundwater Level (ft bgs): N/A
Total Depth (ft bgs): 2.9	Surface Elevation (ft): -36.6	Datum: MLLW

Depth (ft)	Graphic Log	Standard Penetration Test (SPT) Blows/foot	Water Level (ft)	Group Symbol	Material Description	Sheen	Headspace Vapor PID (ppm)	Notes/Sample ID
0								
	▨			ML	Gray sandy silt (soft to medium stiff)			
1	▨							Wood chips at 1 ft (no odor)
	▨							
2	▨							
	▨							
3	▨							

LOG OF BORING # SS-7

BergerABAM

Project: Proposed Grays Harbor Potash Export Facility

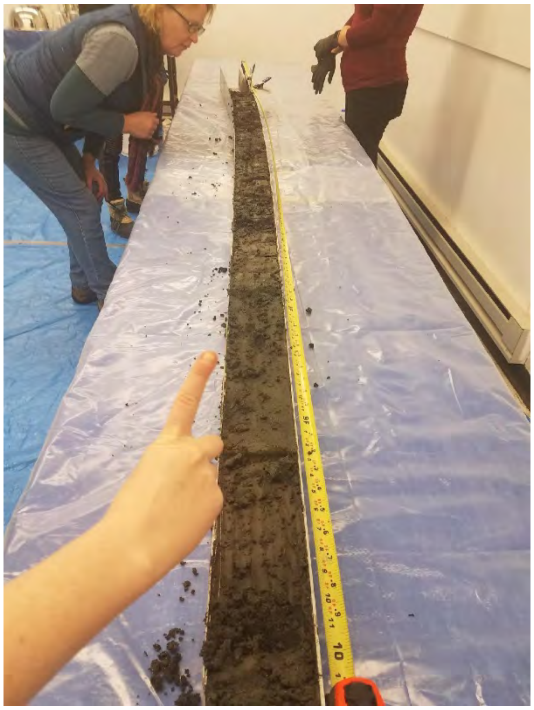
Project Location: Hoquiam, WA

Project Number: A17.0202.00

bgs = below ground surface

ft = feet

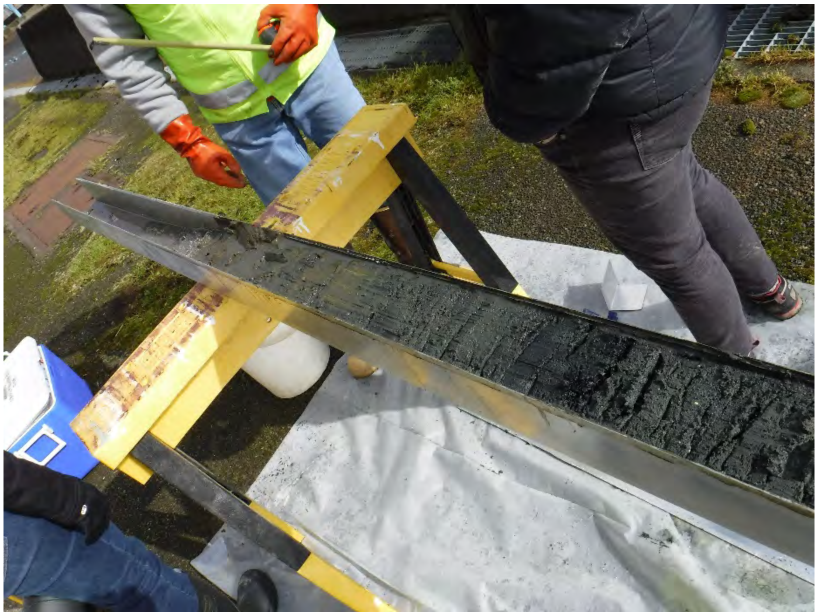
Photograph 1. Sampler Equipment



Photograph 2. Subsample SS-1

Photograph 3. Subsample SS-2

Photograph 4. Subsample SS-3

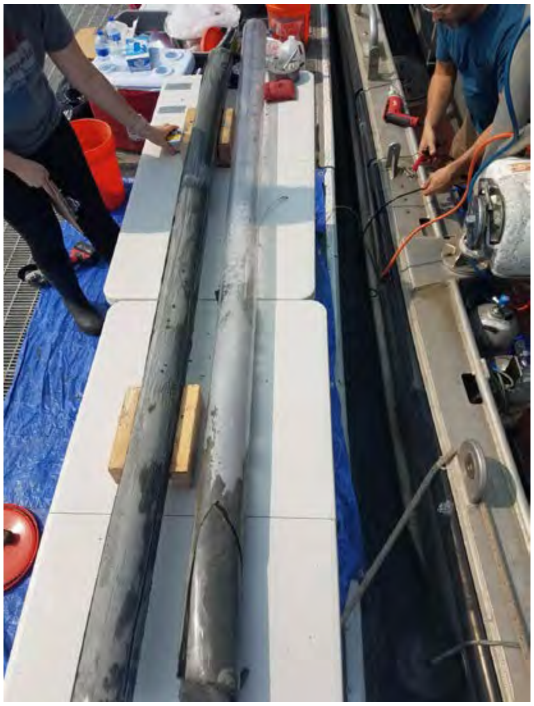


Photograph 5. Subsample SS-5

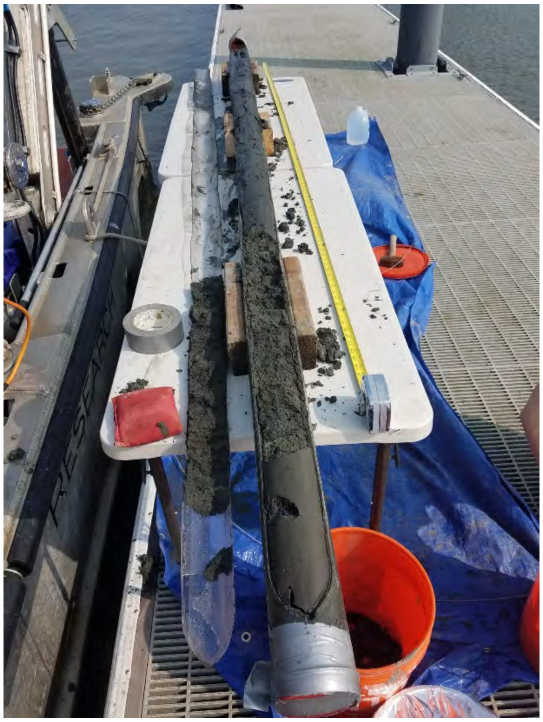
Photograph 6. Subsample SS-6

Photograph 7. Subsample SS-8

Photograph 8. Subsample SS-3A



Photograph 9. Subsample SS-3B



Photograph 10. Subsample SS-4B

Photograph 11. Subsample SS-7

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix F
February 2018 - Chemical Analytical Data Report

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix G
August 2018 - Chemical Analytical Data Report

Revised Dredged Material Characterization Report
Proposed Grays Harbor Potash Export Facility
U.S. Army Corps of Engineers, Seattle District

Appendix H
Data QA/QC Review Summary

APPENDIX H
DATA QA/QC REVIEW SUMMARY

QUALITY ASSURANCE REVIEW

This appendix summarizes the results of a Level 1 quality assurance (QA1) review of the analytical data for sediment samples collected February 2018 and August 2018 from the proposed dredged material associated with the BHP Billiton Canada Inc. (BHP) Proposed Grays Harbor Potash Export Facility. Field procedures used for sample collection are discussed in the Sampling and Analysis Plan (SAP)[1]. BergerABAM submitted sediment samples to Analytical Resources Inc. (ARI), of Tukwila, Washington, for chemical analysis. Copies of the analytical laboratory reports are included in Appendix F. Based on our review, the analytical data are valid with minor qualifications for their intended use. A data completeness checklist is included in Table H-1 of this appendix.

The quality assurance review included examination and validation of the following information from the laboratory's summary reports (ARI Report 18B0374 found in Appendix F).

- Holding times
- Method blanks
- Surrogate recoveries
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) recoveries
- Standard reference material (SRM) recoveries
- Calibration criteria
- Internal standard recoveries
- Laboratory duplicate relative percent difference (RPD)
- Laboratory replicate relative standard deviation

ANALYTICAL METHODS AND DETECTION LIMITS

Chemical Analysis

Six subsamples in February 2018 and four subsamples in August 2018 were collected during the sediment characterization activities. The subsamples were composited into four dredged material management unit (DMMU) samples in February 2018 (DMMU-1 through -4) and two DMMUs in August 2018 (DMMU-2A and -4A) and were analyzed for the following.

- Total organic carbon by SM5310B/Environmental Protection Agency (EPA) Method 9060 (modified for sediments)
- Total solids/total volatile solids by Puget Sound Estuary Program (PSEP)/SM2540G
- Ammonia by Plumb (1981)

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[1] BergerABAM. February 2018. Dredged Material Management Program Sampling and Analysis Plan, Proposed Grays Harbor Potash Export Facility.

- Sulfides by PSEP and Plumb (1981)
- Grain size by PSEP/ASTM D-422 (modified)
- Total metals and mercury using EPA Methods 6010/6020/7440/7471
- Semi-volatile organic compounds (SVOCs) using EPA Method 8270D
- Polycyclic aromatic hydrocarbons (PAHs) using EPA Method 8270D
- Chlorinated hydrocarbons using EPA Methods 8260B/8270D/8081
- Phthalates, phenols, and miscellaneous extractables using EPA Methods 8270D/8081
- Pesticides using EPA Method 8081
- Polychlorinated biphenyls (PCBs) using EPA Method 8082
- Bulk tributyltin (TBT) using PSEP, Krone (1989), and Unger (1986)
- Dioxins/furans using EPA Method 1613B

Detection and Reporting Limits

Method detection limits (MDLs) are the minimum concentration of a chemical compound that can be measured and reported. The MDL is based on instrumentation abilities and the sample matrix. Method reporting limits (MRL) represent the concentration that can be accurately quantified. MRLs are set by the laboratory and are based on the low standard of the initial calibration curve or low-level calibration check standard.

In some cases, the MRL is raised because of high concentrations of analytes in the samples or matrix interferences. MRLs were consistent with industry standards. Tables 4 and 5 of this report list the MDLs for undetected samples. The MDLs are sufficient in achieving the Dredged Material Management Program/Sediment Management Standards criteria. Analytes that were detected between the MDL and MRL are qualified as estimated (J qualifier).

QA REVIEW RESULTS

The laboratory provided quality control (QC) sample results that underwent a QA review. Laboratory QC samples were consistent with those specified in the SAP to evaluate precision, accuracy, representativeness, comparability, and completeness. The sample data and laboratory QC data are suitable for their intended use with minor qualifications. The following summary lists the results of the QA review by analyte or test.

General Chemistry Parameters (total organic carbon, total solids, sulfide, and ammonia)

All hold times were met. The method blanks were clean at the reporting limits. The SRM and LCS percent recoveries were within control limits. All initial calibrations were within method requirements.

The duplicate RPD of sulfide was outside the control limit for sample DMMU-3. All other QC parameters were met for this analysis. No corrective action was taken.

Total Metals

All required holding times were met. The February 2018 method blank had lead detected below the reporting limit, but above the MDL. Lead was flagged with a "J" qualifier on the method blank. No further corrective action was taken. The August 2018 method blanks were clean at the reporting limits. The February 2018 LCS percent recoveries were within control limits. The August 2018 LCS BGH0475 mis-spiked and had no recovery for silver. The LCS was reprepped with silver within control limits. All other LCS percent recoveries were within control limits. No further corrective action was taken.

The February 2018 SRM percent recoveries were within QC limits. The August 2018 SRM mis-spiked and had no recovery for silver. The SRM was reprepped with silver within QC limits. All other SRM percent recoveries were within QC limits. No further corrective action was taken.

The duplicate RPDs were within control limits.

The matrix spike percent recovery of antimony fell outside the control limits low for sample DMMU-3. A post-digestion spike was performed, and the recovery was within the control limits. The lab did not take any further corrective action.

Total Metals UCT-KED by 6020A

The samples were digested and analyzed within the recommended holding times.

The February 2018 initial calibration BC00026 has a linear COD limit for copper at the COD limit. The linear COD does not exceed the COD limit. No corrective action was taken. The August 2018 initial and continuing calibrations were within method requirements.

The February 2018 method blank has copper and zinc detected below the reporting limits, but above the MDLs. These metals have been flagged with "J" qualifiers on the method blank. No further corrective action was taken. The August 2018 method blank was clean at the reporting limits.

The LCS percent recoveries were within control limits.

The matrix spike, matrix spike duplicate, and duplicate percent recoveries and/or RPDs were within QC limits.

The SRM percent recoveries were within QC limits.

Mercury

All required holding times were met for the DMMU samples. The method blank was clean at the reporting limit. The LCS percent recovery was within control limits. The SRM percent recoveries were within QC limits. The matrix spike percent recovery and duplicate RPD were within control limits.

SVOCs by 8270

The samples and associated laboratory QC were extracted and analyzed within the holding times. Internal calibration were within method requirements. The surrogate percent recoveries were within control limits. The method blank was clean at the reporting limits. The LCS percent recoveries were within control limits. Initial standard areas were within limits.

The February 2018 initial calibration verification fell outside control limits low for benzoic acid. Associated detected results and QC were flagged with “Q” qualifiers. No further corrective action was taken.

The February 2018 certified reference material (CRM) percent recoveries were within QC limits. The August 2018 SRM had low percent recoveries for 1,4-dichlorobenzene, naphthalene, and anthracene. All the other percent recoveries were within QC limits. No corrective action was taken.

The matrix spike and matrix spike duplicate percent recoveries of benzoic acid fell outside the control limits low for sample DMMU-3. All other matrix spike/matrix duplicate percent recoveries and RPD were within QC limits. No corrective action was taken.

SVOCs/TBT by 8270-SIM

The samples were extracted and analyzed within the recommended holding times. Initial calibrations were within method requirements.

The February 2018 dual scan initial calibration verification (ICV) for NT14 on 9 March 2018 is outside of control limits high for butylbenzylphthalate and the surrogate p-terphenyl-d14. Associated detected results and QC have been flagged with "Q" qualifiers. No further corrective action was taken. The August 2018 initial and continuing calibrations were within method requirements.

Internal standard areas were within limits.

Sample DMMU-2 has low surrogate percent recovery for p-terphenyl-d14 in association with the dual scan analysis. All other surrogate percent recoveries were within control limits. No corrective action was taken.

The method blanks were clean at the reporting limits. The LCS percent recoveries were within control limits.

A dual scan matrix spike and matrix spike duplicate were prepared in conjunction with sample DMMU-3. The matrix spike/matrix spike duplicate percent recoveries and RPD were within QC limits.

A TBT matrix spike and matrix spike duplicate were prepared in conjunction with sample DMMU-1. The matrix spike/matrix spike duplicate percent recoveries and RPD were within QC limits.

The August 2018 dual scan SRM F011621 has low percent recovery for 1,4-dichlorobenzene. The TBT SRM F011117 had low percent recovery for the tributyltin ion. All other percent recoveries were within QC limits. No corrective action was taken.

The CRM percent recoveries were within QC limits.

Pesticides

The samples and associated laboratory QC were extracted and analyzed within the method holding times. Initial calibrations were within method requirements. Internal standard areas were within limits. The surrogate percent recoveries were within control limits.

The method blank was clean at the reporting limits. The LCS percent recoveries were within control limits.

A matrix spike and matrix spike duplicate were prepared in conjunction with sample DMMU-3. The matrix spike has high spike recovery for heptachlor and trans-chlordane. The matrix spike duplicate has high spike recovery for trans-chlordane. The results are advisory. All other matrix spike/matrix spike duplicate percent recoveries were within QC limits. No corrective action was taken.

The August 2018 matrix spike had low spike recovery for 4,4'-DDD and the second column 4,4'-DDT. The matrix spike duplicate has high RPD for cis-chlordane and the second column hexachlorobenzene, and low spike recovery for 4,4'-DDT. Both the matrix spike and matrix spike duplicate have reported values for heptachlor and trans-chlordane that are greater than 40 percent difference between the concentration determined on two GC columns where applicable. These compounds have been flagged with "P1" qualifiers on the matrix QC. The sample is non-detect for all target compounds. The results are advisory.

No further corrective action was taken.

The February 2018 CRM has low recovery for the first column of 4,4'-DDD. All other percent recoveries were within QC limits. No corrective action was taken.

The August 2018 SRM has various compounds that are over range and have been flagged with "E" qualifiers. The SRM is not appropriate for Puget Sound Dredged Disposal Analysis level reporting. The SRM has low percent recovery for both columns of 4,4'-DDD. No further corrective action was taken.

PCBs

The samples and associated laboratory QC were extracted and analyzed within the method holding times. Initial calibrations were within method requirements. Internal standard areas were within limits. The surrogate percent recoveries were within control limits.

The February 2018 ICV SGC0142-ICV2 is outside of control limits high for the first column, Aroclor 1260. All other aroclors were within control limits. No corrective action was taken. The August 2018 initial and continuing calibrations were within method requirements.

The method blank was clean at the reporting limits. The LCS percent recoveries were within control limits.

A matrix spike and matrix spike duplicate were prepared in conjunction with sample DMMU-3. The matrix spike/matrix spike duplicate percent recoveries and RPD were within QC limits.

The SRM percent recoveries were within QC limits.

Dioxins/Furans

The samples were extracted and analyzed within the recommended holding times. Analysis was performed using an application specific column developed by Restek. The RTX-DIoxin2 column has unique isomer separation for the 2378-TCDF, eliminating the need for confirmation analysis.

Initial and continuing calibrations were within method requirements. Labeled internal standard areas were within limits. The cleanup surrogate percent recoveries were within control limits.

The February 2018 method blank BGD0106 has OCDD detected above the reporting limits and 1,2,3,4,6,7,8-HpCDD flagged with a "J" qualifier on the method blank detected below the reporting limit, but above the method detection limit. Associated detected results have been flagged with "B" qualifiers. No further corrective action was taken.

The August 2018 method blank had reportable responses for various compounds below the reporting limits. Associated detected results and QC have been flagged with "B" qualifiers. No further corrective action was taken.

The Ongoing Precision and Recovery standard percent recoveries were within control limits.

A duplicate was prepared in conjunction with sample DMMU-3. The duplicate results are advisory. No corrective action was taken.

The SRM percent recoveries were within QC limits.

Grain Size

All required holding times were met.

An assumed specific gravity of 2.65 was used in the hydrometer calculations. One sample was run in triplicate for each sampling event. There were no noted anomalies.

Table H-1. QA1 Data Checklist

Sample Locations and Compositing	Test Sediment	Reference Sediment
Latitude and Longitude	NAD 83	NAD 83
NAD 1983 HARN	Yes	Yes
Station Name	Yes	Yes
Water Depth	Yes	NA
Drawing Showing Sampling Locations and ID Numbers	Yes	NA
Compositing Scheme (sampling locations/depths for composites)	Yes	NA
Sampling Method	Yes	NA
Sampling Dates	Yes	NA
Estimated Volume of Dredged Material Represented by each DMMU	Yes	NA
Positioning Method	Yes	NA
Sediment Conventionals		
Preparation and Analysis Methods	Yes	NA
Sediment Conventional Data and QA/QC Qualifiers	Yes	NA
QA Qualifier Code Definitions	Yes	NA
Units (dry weight except for total solids)	Yes	NA
Method Blank Data (sulfides, ammonia, TOC)	Yes	NA
Method Blank Units (dry weight)	Yes	NA
Analysis Dates (sediment conventionals, blanks)	Yes	NA
Grain Size Analysis		
Fine Grain Analysis Method	Yes	NA
Analysis Dates	Yes	NA
Triplicate	Yes	NA
Grain Size Data (complete sieve and phi size distribution)	Yes	NA

NAD=North American Datum; NA=Not Applicable; DMMU=Dredged Material Management Unit; QA/QC=quality assurance/quality control; TOC= Total Organic Carbon

Sediment Chemical Analyses

	Metals	SVOCs/ PAHs	Pesticides/ PCBs	VOCs	Dioxins/ Furans
Extraction/digestion method	NA	NA	NA	NA	Yes
Extraction/digestion dates (test sediment, blanks, matrix spike, reference material)	Yes	Yes	Yes	NA	Yes
Analysis method	Yes	Yes	Yes	Yes	Yes
Data and QA qualifier included for:					
Test sediments	Yes	Yes	Yes	Yes	Yes
Reference materials, including 95% confidence interval	NA	NA	Yes	NA	Yes

	Metals	SVOCs/ PAHs	Pesticides/ PCBs	VOCs	Dioxins/ Furans
Method blanks	Yes	Yes	Yes	Yes	Yes
Matrix spikes	Yes	Yes	Yes	NA	NA
Matrix spike added (dry weight basis)	Yes	Yes	Yes	Yes	Yes
Laboratory control sample	Yes	Yes	Yes	Yes	Yes
Laboratory control sample duplicate	NA	NA	NA	Yes	NA
Replicates	Yes	NA	NA	Yes	NA
Continuing calibration verification	Yes	Yes	Yes	Yes	Yes
Units (dry weight)	Yes	Yes	Yes	Yes	Yes
Method blank units (dry weight)	Yes	Yes	Yes	Yes	Yes
QA/QC qualifier definitions	Yes	Yes	Yes	Yes	Yes
Surrogate recovery for test sediment, blank, matrix spike, ref. material	Yes	Yes	Yes	Yes	Yes
Analysis dates (test sediment, blanks, matrix spike, reference material)	Yes	Yes	Yes	Yes	Yes

SVOC=semi-volatile organic compound; PAH=polycyclic aromatic hydrocarbon; PCB=polychlorinated biphenyl; NA=Not Applicable

LIST OF ACRONYMS AND ABBREVIATIONS

ARI	Analytical Resources Inc.
BHP	BHP Billiton Canada Inc.
CRM	certified reference material
DMMU	dredged material management unit
EPA	Environmental Protection Agency
ICV	initial calibration verification
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDL	method detection limit
MRL	method reporting limit
NAD	North American Datum
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl

PSEP	Puget Sound Estuary Program
QA1	Level 1 quality assurance
QC	quality control
RPD	relative percent difference
SAP	Sampling and Analysis Plan
SRM	standard reference material
SVOC	semi-volatile organic compounds
TBT	tributyltin