

Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
 Portland Harbor Superfund Site
 Surface Sediment – Stratified Random

Laboratory: TestAmerica Laboratories, Incorporated, Seattle, WA

Laboratory Group: 580-80756-1

Analyses: Petroleum Hydrocarbons, Metals, Total Organic Carbon (TOC), Tributyltin, Polycyclic Aromatic Hydrocarbons (PAHs), bis(2-Ethylhexyl)phthalate, Total Solids, and Grain Size

Validation Level: Stage 2A

AECOM Project
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SUMMARY

The data quality review of two surface sediment samples collected on July 20, 2018, has been completed. The samples were analyzed for total petroleum hydrocarbons (TPHs, diesel-range and motor oil-range) by Washington State Department of Ecology (Ecology) Method NWTPH-Dx; metals by United States Environmental Protection Agency (EPA) Method 6020B (arsenic, cadmium, copper, lead, and zinc) and EPA Method 7471A (mercury); TOC by EPA Method 9060; tributyltin by Krone et al.; PAHs by EPA Method 8270D modified by selected ion monitoring (SIM); bis(2-ethylhexyl) phthalate by EPA Method 8270D; total solids by American Society for Testing and Materials (ASTM) Method D-2216; moisture content at 70 degrees Celsius (°C); and/or grain size by ASTM Method D7928/D6913 by TestAmerica Laboratories, Incorporated (TA) located in Tacoma, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846)*, Ecology's *Analytical Methods for Petroleum Hydrocarbons*, June 1997, Annual Book of ASTM Standards, ASTM, Philadelphia, Pennsylvania, and Krone CA et al., *A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound*, Marine Environmental Research, 1989. The laboratory provided level 2 and level 4 data packages containing sample results, and associated quality assurance (QA) and quality control (QC) data, preparation logs, and raw instrument outputs (where applicable). The following samples are associated with laboratory group 580-80756-1:

Sample ID	Laboratory ID
PDI-SG-B258-BL1	580-80756-1
PDI-SG-B258-BL1-D (field duplicate of PDI-SG-B258-BL1)	580-80756-2

Data validation is based on method performance criteria and QC criteria documented in the *Quality Assurance Project Plan (QAPP)*, dated March 23, 2018, as amended. If data qualification was required, data were qualified based on the definitions and use of qualifying flags outlined in the EPA documents *USEPA National Functional Guidelines for Organic Superfund Methods Data Review*, January 2017, and *USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review*, January 2017. Data qualifiers assigned to this sample set are included in Table 1.



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SAMPLE RECEIPT

Upon receipt by TA, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperature was recorded. The cooler was received at a temperature below the EPA-recommended limits of greater than 0°C and less than or equal to 6°C at -11.0°C. Data were not qualified based on the low cooler temperature. The samples in this laboratory group were frozen after sample collection on 7/24/18 until they were shipped to TA on 9/26/18 and then again by TA on 10/4/18.

ORGANIC ANALYSES

Samples were analyzed for TPHs, tributyltin, PAHs, and bis(2-ethylhexyl)phthalate by the methods identified in the introduction to this report.

1. Holding Times – Acceptable

General – The laboratory noted that the samples were frozen upon receipt in order to extend the holding time. The samples were thawed and prepped within holding time.

2. Blanks – Acceptable except as noted below:

General – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. Sediment data were not qualified based on rinsate blank results.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – bis(2-Ethylhexyl)phthalate was detected in the method blank associated with prep batch 286343 (4.07 ug/kg) at a concentration between the method detection limit (MDL) and the reporting limit. bis(2-Ethylhexyl)phthalate was detected at a concentration between the MDL and reporting limit in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D. The samples were diluted and the reporting limits were elevated; therefore, the results for bis(2-ethylhexyl)phthalate in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D were qualified as estimated and flagged 'J' based on the method blank result.

3. Surrogates – Acceptable

4. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable except as noted below:

PAHs by EPA Method 8270D-SIM – The percent recovery of anthracene in the LCS (71%) associated with prep batch 286335 was outside the control limits of 73-125%. The results for anthracene in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D were qualified as estimated and flagged 'UJ' based on the LCS result.

5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B258-BL1-D. The percent recoveries in the MS (-11%) and MSD (-20%) were outside the control limits of 59-123%. The results for bis(2-ethylhexyl)phthalate in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D were qualified as estimated and flagged 'J' based on the method blank results and no further qualification was necessary based on the MS/MSD results.

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TPHs by Method NWTPH-Dx – An MS/MSD was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

PAHs by EPA Method 8270D-SIM – An MS/MSD was not performed using a sample from this laboratory group. Accuracy was assessed using the LCS. Precision was not assessed using a sample from this laboratory group.

Tributyltin by Krone et al. – An MS/MSD was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

6. Field Duplicate – Acceptable except as noted below:

General – A field duplicate was submitted for PDI-SG-B258-BL1 and identified as PDI-SG-B258-BL1-D. Results were comparable with the following exceptions.

PAHs by EPA Method 8270D-SIM – The relative percent differences (RPDs) for the following analytes either exceeded the control limits of 50% or could not be calculated:

Analyte	RPD
2-Methylnaphthalene	NC
Acenaphthene	NC
Acenaphthylene	NC
Benzo[a]anthracene	152%
Benzo[a]pyrene	164%
Benzo[b]fluoranthene	145%
Benzo[g,h,i]perylene	131%
Benzo[k]fluoranthene	155%
Chrysene	NC
Dibenz(a,h)anthracene	NC
Fluoranthene	NC
Fluorene	NC
Indeno[1,2,3-cd]pyrene	142%
Naphthalene	NC
Phenanthrene	152%
Pyrene	NC

NC – not calculable

The sample concentrations for the analytes listed above were less than five times the reporting limits; therefore, data were not qualified based on field duplicate RPDs.

7. Laboratory Duplicate

TPHs by Method NWTPH-Dx – Laboratory duplicates were not performed using the sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

8. Reporting Limits – Acceptable except as noted below:

General – Analyte concentrations detected between the MDLs and the reporting limits are reported by the laboratory with 'J' flags. Laboratory 'J'-flagged results are considered

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estimated results. As the results are between the MDLs and the reporting limits, there is a greater level of uncertainty associated with the numerical results.

PAHs by EPA Method 8270D-SIM – The reporting limits for both samples in this laboratory group were raised because of the dilutions that were required prior to analysis due to the nature of the sample matrix. The reporting limits for multiple analytes in both samples reported as not detected exceeded the cleanup level for carcinogenic PAHs (12 ug/kg) but the MDLs did not.

9. Other Items of Note:

TPHs by Method NWTPH-Dx – The laboratory indicated that the diesel-range hydrocarbon elution pattern was later than the typical diesel pattern in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D.

METALS ANALYSES

Samples were analyzed for metals by the methods identified in the introduction to this report.

1. Holding Times – Acceptable except as noted below:

Mercury by Method 7471A – As noted under sample receipt, all samples in this laboratory group were frozen after collection and prior to shipment to TA, and again by TA upon receipt. The holding time for mercury is not extended by freezing; therefore the holding time remains 28 days to final analysis. The 28-day holding time for mercury was exceeded in PDI-SG-B258-BL1 and PDI-SG-B258-BL1-D by 59 days. The results for mercury in these samples were qualified as estimated and flagged 'J' based on the holding time exceedance.

2. Blanks – Acceptable

General – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. Sediment data were not qualified based on rinsate blank results.

3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable

4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Post-Digestion Spike (PDS, where applicable)

General – MS/MSDs were not performed on the sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSDs.

5. Field Duplicate – Acceptable

General – A field duplicate was submitted for PDI-SG-B258-BL1 and identified as PDI-SG-B258-BL1-D. Results were comparable.

6. Laboratory Duplicate

General – Laboratory duplicates were not performed using the sample from this laboratory group. Precision was assessed using the LCS/LCSD.



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7. Serial Dilution

Metals by Method 6020B – A serial dilution was not performed on the sample from this laboratory group. Precision was assessed using the LCS/LCSD.

8. Reporting Limits – Acceptable

General – One or more results in multiple samples were reported at concentrations between the reporting limits and the MDLs and were flagged 'J' by the laboratory. As described above, laboratory 'J'-flagged results are considered estimated results.

CONVENTIONAL ANALYSES

Samples were analyzed for TOC and total solids by the methods identified in the introduction to this report.

1. Holding Times – Acceptable except as noted below:

Total Solids by ASTM Method D-2216/Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for the sample in this laboratory group by 77 days. No data qualifiers were assigned based on this holding time exceedance.

2. Blanks – Acceptable

General – A rinsate blank was not submitted with this laboratory group. Associated rinsate blanks are reported under separate cover. Target compounds may have been detected in the rinsate blanks associated with these samples. Sediment data were not qualified based on rinsate blank results.

3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable

4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

TOC by Method 9060 – An MS/MSD was not performed using the sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

5. Field Duplicate – Acceptable

General – A field duplicate was submitted for PDI-SG-B258-BL1 and identified as PDI-SG-B258-BL1-D. Results were comparable.

6. Laboratory Replicate

General – Laboratory duplicates were not performed using the sample from this laboratory group. Precision was assessed using the LCS/LCSD where applicable.

7. Reporting Limits – Acceptable

GRAIN SIZE ANALYSES

Samples were analyzed for grain size by the methods identified in the introduction to this report. The data were reviewed to confirm that the required grain size fractions identified in the QAPP were



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reported for each sample. As indicated under sample receipt, the sample volume used for grain size analysis was frozen until shipped to TA. No data qualifiers were assigned to grain size results based on sample condition.

1. Laboratory Duplicate

The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was not performed using the sample from this laboratory group.

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group, as qualified, is considered usable for meeting project objectives. The completeness for laboratory group 580-80756-1 is 100%.

Table 1
QA/QC Data Summary Review
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B258-BL1	580-80756-1	SW7471A	Mercury	0.067	mg/kg	0.067 J	h
PDI-SG-B258-BL1	580-80756-1	SW8270D	Bis(2-ethylhexyl)phthalate	100 J	ug/kg	100 J	bl
PDI-SG-B258-BL1	580-80756-1	SW8270DSIM	Anthracene	16 U	ug/kg	16 UJ	l
PDI-SG-B258-BL1-D	580-80756-2	SW7471A	Mercury	0.063	mg/kg	0.063 J	h
PDI-SG-B258-BL1-D	580-80756-2	SW8270D	Bis(2-ethylhexyl)phthalate	150 J	ug/kg	150 J	bl
PDI-SG-B258-BL1-D	580-80756-2	SW8270DSIM	Anthracene	16 U	ug/kg	16 UJ	l

Notes:

bl - laboratory blank contamination

h - holding time

J - estimated value

l - laboratory control sample recovery

mg/kg - milligram per kilogram

U - Compound was analyzed for, but not detected above the value shown.

ug/kg - microgram per kilogram