

# Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Portland Harbor Superfund Site  
 Surface Sediment – Downtown/Upriver

Laboratory: TestAmerica Laboratories, Incorporated, Seattle, WA

Laboratory Groups: 580-80635-1, 580-80635-6, and 580-80635-7

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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File Name: 580-80635-1\_6\_7 DVR

## SUMMARY

The data quality review of four surface sediment samples collected between July 27 and August 17, 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 groups 580-80635-1, 580-80635-6, and 580-80635-7:

Sample ID	Laboratory ID	Analytes
PDI-SG-B436	580-80635-1	TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, Total Solids, Grain Size
PDI-SG-B474	580-80635-2	TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, Total Solids, Grain Size
PDI-SG-B480	580-80635-3	Grain Size
PDI-SG-B481	580-80635-4	TPH, Metals, Tributyltin, bis(2-Ethylhexyl) phthalate, Total Solids, Grain Size

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.



## Data Validation Report

### Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

#### Surface Sediment – Downtown/Upriver

TestAmerica Lab Groups: 580-80635-1, 580-80635-6, and 580-80635-7

## SAMPLE RECEIPT

Upon receipt by TA, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperatures were recorded. One 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 as the samples were shipped frozen. TA noted that an extra sample container was received that was not marked on the COC. AECOM confirmed that it was the Atterberg limits container for PDI-SG-B436 and requested that it be logged and put on hold. All samples were cancelled for Atterberg limits on 10/2/18. All samples in this laboratory group were frozen at the field warehouse, except grain size, after sample collection until they were shipped to TA on 9/26/18. TA froze the samples upon receipt to extend holding times until they were thawed for analysis. The rush grain size results were reported in laboratory group 580-80635-6 on 10/8/18. PDI-SG-B436 and PDI-SG-B481 were authorized for the on hold analyses on 10/9/18 and reported in laboratory group 580-80635-1 on 10/24/18. PDI-SG-B474 was authorized for the on hold analyses on 10/23/18 and reported in laboratory group 580-80635-7 on 11/21/18.

## ORGANIC ANALYSES

The 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
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.

PAHs by EPA Method 8270D-SIM – 2-Methylnaphthalene (0.150 ug/kg), acenaphthene (0.183 ug/kg), naphthalene (0.288 ug/kg), and phenanthrene (0.338 ug/kg) were detected in the method blank associated with prep batch 288111 at concentrations between the method detection limits (MDLs) and reporting limits. 2-Methylnaphthalene, naphthalene, and phenanthrene were either not detected in the associated sample or detected at a concentration greater than the reporting limit and significantly greater than the method blank result; therefore, data for these analytes were not qualified based on the method blank results. Acenaphthene was detected in PDI-SG-B474 at a concentration between the MDL and the reporting limit in a sample that was diluted prior to analysis; therefore, the result was qualified as estimated and flagged 'J' based on this method blank result.

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

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The percent recovery for bis(2-ethylhexyl) phthalate in the LCS (130%) associated with prep batch 288122 was above the control limits of 59-123%. bis(2-Ethylhexyl) phthalate was not detected in the associated sample; therefore, data were not qualified based on this elevated LCS recovery.

## Data Validation Report

### Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

#### Surface Sediment – Downtown/Upriver

TestAmerica Lab Groups: 580-80635-1, 580-80635-6, and 580-80635-7

PAHs by EPA Method 8270D-SIM – The percent recovery for anthracene in the LCS (71%) associated with prep batch 286335 was below the control limits of 73-125%. The results for anthracene in PDI-SG-B436 and PDI-SG-B481 were qualified as estimated and flagged 'UJ' based on this LCS recovery.

5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Acceptable except as noted below:

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B436. The percent recoveries in the MS (-140%) and MSD (36%) and the relative percent difference (RPD) for the MS/MSD pair (15%) were outside of the control limits of 59-123% and 13%, respectively. The sample result was more than 4 times the spiking concentration; therefore, no data were qualified based on the MS/MSD results.

An MS/MSD was analyzed using PDI-SG-B474. The percent recoveries for the MS and MSD could not be calculated because the sample was diluted and the amount that was spiked was below the elevated MDL. Results were not qualified based on MS/MSD results that could not be calculated.

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.

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.

Tributyltin by Krone et al. – 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.

6. Laboratory Duplicate

TPHs by Method NWTPH-Dx – A laboratory duplicate was performed using PDI-SG-B481. The RPD for motor oil-range hydrocarbons (85%) exceeded the control limit of 35%. The sample concentration for motor oil-range hydrocarbons in PDI-SG-B481 was less than five times the reporting limit; therefore, data were not qualified based on the elevated laboratory duplicate RPD.

A laboratory duplicate was performed using PDI-SG-B474. Results were comparable.

7. 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 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 PDI-SG-B436, PDI-SG-B474, and PDI-SG-B481 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 all samples reported as not detected exceeded the cleanup level for carcinogenic PAHs (12 ug/kg), but the MDLs did not.

## Data Validation Report

### Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

#### Surface Sediment – Downtown/Upriver

TestAmerica Lab Groups: 580-80635-1, 580-80635-6, and 580-80635-7

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The reporting limits for PDI-SG-B436, PDI-SG-B474, and PDI-SG-B481 were raised because of the dilutions that were required prior to analysis due to the nature of the sample matrix. The reporting limit and MDL for the result reported as not detected in PDI-SG-B481 exceeded the cleanup level (135 ug/kg). The reporting limit for the result reported as not detected in PDI-SG-B474 exceeded the cleanup level, but the MDL did not.

TPHs by Method NWTPH-Dx – The laboratory indicated that the diesel-range hydrocarbon elution patterns were later than the typical diesel pattern in PDI-SG-B436, PDI-SG-B474, and PDI-SG-B481.

## METALS ANALYSES

The 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 in the field warehouse after sample collection 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 holding time for mercury was exceeded in PDI-SG-B436, PDI-SG-B474, and PDI-SG-B481 by 32-52 days. The results for mercury in these samples were qualified as estimated and flagged 'J' based on the holding time exceedance.

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.

Metals by Method 6020B – Copper (0.158 mg/kg) was detected in the method blank associated with prep batch 288047 at a concentration between the MDL and the reporting limit. Copper was detected in the associated sample at a concentration greater than the reporting limit and significantly greater than the method blank detection; therefore, data were not qualified based on this method blank result.

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 using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

5. Laboratory Duplicate

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

## Data Validation Report

### Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling

#### Surface Sediment – Downtown/Upriver

TestAmerica Lab Groups: 580-80635-1, 580-80635-6, and 580-80635-7

#### 6. Serial Dilution

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

#### 7. 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

The sample was 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 and Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for PDI-SG-B436, PDI-SG-B474, and PDI-SG-B481 by 49-87 days. No data qualifiers were assigned based on this holding time exceedance.

#### 2. Blanks – Acceptable

TOC by Method 9060 – 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) – Acceptable

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

#### 5. Laboratory Replicate – Acceptable

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

#### 6. Reporting Limits – Acceptable

## GRAIN SIZE ANALYSES

The sample was 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 reported for each sample.



**Data Validation Report**

**Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling**

**Surface Sediment – Downtown/Upriver**

**TestAmerica Lab Groups: 580-80635-1, 580-80635-6, and 580-80635-7**

1. Laboratory Duplicate – Acceptable

The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was performed using PDI-SG-B436. Results were comparable.

**OVERALL ASSESSMENT OF DATA**

The data reported in this laboratory group, as qualified, is considered usable for meeting project objectives. The completeness for laboratory groups 580-80635-1, 580-80635-6, and 580-80635-7 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Surface Sediment - Downtown/Upriver**  
**TestAmerica Laboratory Groups: 580-80635-1, 580-80635-6, and 580-80635-7**

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B436	580-80635-1	SW7471A	Mercury	0.067	mg/kg	0.067 J	h
PDI-SG-B436	580-80635-1	SW8270DSIM	Anthracene	47 U	ug/kg	47 UJ	l
PDI-SG-B474	580-80635-2	SW7471A	Mercury	0.037 J	mg/kg	0.037 J	h
PDI-SG-B474	580-80635-2	SW8270DSIM	Acenaphthene	6.4 J	ug/kg	6.4 J	bl
PDI-SG-B481	580-80635-4	SW7471A	Mercury	0.052	mg/kg	0.052 J	h
PDI-SG-B481	580-80635-4	SW8270DSIM	Anthracene	42 U	ug/kg	42 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