

# 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: 580-79202-1 and 580-79202-6

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

Number: 60566335, Task #2.12

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File Name: 580-79202-1\_6 DVR

## SUMMARY

The data quality review of six surface sediment samples collected on July 27 and July 28, 2018, has been completed. 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), mercury by EPA Method 7471A, 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 580-79202-1 and 580-79202-6:

Sample ID	Laboratory ID
PDI-SG-B485	580-79202-1
PDI-SG-B484	580-79202-2
PDI-SG-B482	580-79202-3
PDI-SG-B487	580-79202-4
PDI-SG-B488	580-79202-5
PDI-SG-B486	580-79202-6

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*,



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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.

## 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 within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C. All six samples were received by the lab on 7/30/18 for rush grain size analyses and the other analyses were on hold. Rush grain size was reported in laboratory report 580-79202-6 on 8/8/18. All six samples in this laboratory group were authorized for the other analyses on 8/16/18, but due to laboratory oversight the samples were not frozen upon receipt at TA Tacoma. Frozen samples were shipped from TA Sacramento, where samples were properly frozen upon receipt, to TA Tacoma on 9/10/18. These frozen samples were used for analysis.

## 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
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 284043 (3.89 ug/kg) at a concentration between the method detection limit (MDL) and reporting limit. bis(2-Ethylhexyl)phthalate was not detected in the associated samples; therefore, data were not qualified based on this method blank result.

3. Surrogates – Acceptable
4. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable
5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

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.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – 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.

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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 – Acceptable

TPHs by Method NWTPH-Dx – A laboratory duplicate was performed using PDI-SG-B486. 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 all 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 PAHs reported as not detected for multiple analytes in multiple samples exceeded the cleanup level for carcinogenic PAHs (12 ug/kg) but the MDLs did not. The reporting limit and MDL for chrysene reported as not detected in PDI-SG-B486 exceeded the cleanup level for carcinogenic PAHs.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The reporting limits for all 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 results reported as not detected in PDI-SG-B485, PDI-SG-B484, PDI-SG-B482, PDI-SG-B487, PDI-SG-B488, and PDI-SG-B486 and the MDLs in PDI-SG-B485, PDI-SG-B484, and PDI-SG-B486 exceeded the cleanup level (135 ug/kg).

8. Other Items of Note:

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-B485, PDI-SG-B484, PDI-SG-B482, PDI-SG-B487, PDI-SG-B488, and PDI-SG-B486.

The laboratory noted that the percent difference (%D) for the surrogate o-terphenyl in the continuing calibration verification (CCV) associated with analytical batch 284139 was outside the control limits of  $\pm 20\%$  (high). As the surrogate recovery in the associated sample was acceptable, data were not qualified based on this high surrogate %D.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The laboratory noted that the internal standard response in the method blank and LCS associated with analytical batch 284043 were outside of the acceptance limits. Data were not qualified based on internal standard responses in QC samples.

The laboratory noted that the %Ds for the surrogate terphenyl-d14 in the CCVs associated with analytical batches 284395 and 284567 were outside the control limits of  $\pm 20\%$  (high). As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds.

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## 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 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 all samples in this laboratory group by 2-3 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.

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) – Acceptable

Metals by Method 6020B – An MS/MSD and PDS were performed using PDI-SG-B485. Results were acceptable.

Mercury by Method 7471A – An MS/MSD was not performed on a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSDs.

5. Laboratory Duplicate

Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B485. Results were comparable.

Mercury by Method 7471A – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSD.

6. Serial Dilution

Metals by Method 6020B – A serial dilution was performed using PDI-SG-B485. Results were comparable.

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.



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## 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 – The 7-day holding time indicated for total solids in the QAPP was exceeded for the samples in this laboratory group. No data qualifiers were assigned based on this 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.

TOC by Method 9060 – TOC was detected in the method blank associated with analytical batch 284391 (119 mg/kg) at a concentration between the MDL and the RL. TOC was detected in the associated samples at concentrations greater than the RL and greater than 2x 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)

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

TOC by Method 9060 – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSD.

Total Solids by ASTM Method D-2216 – A laboratory duplicate was performed using PDI-SG-B485. Results were comparable.

6. 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 reported for each sample.



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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 a 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 groups 580-79202-1 and 580-79202-6 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Surface Sediment - Downtown/Upriver**  
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B485	580-79202-1	SW7471A	Mercury	0.037 J	mg/kg	0.037 J	h
PDI-SG-B484	580-79202-2	SW7471A	Mercury	0.032 J	mg/kg	0.032 J	h
PDI-SG-B482	580-79202-3	SW7471A	Mercury	0.034 J	mg/kg	0.034 J	h
PDI-SG-B487	580-79202-4	SW7471A	Mercury	0.034 J	mg/kg	0.034 J	h
PDI-SG-B488	580-79202-5	SW7471A	Mercury	0.031 J	mg/kg	0.031 J	h
PDI-SG-B486	580-79202-6	SW7471A	Mercury	0.038	mg/kg	0.038 J	h

Notes:

h - holding time

J - estimated value

mg/kg - milligram per kilogram