

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: 580-79055-1 and 580-79055-6

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
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SUMMARY

The data quality review of two surface sediment samples collected on July 21, 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, zinc, and/or manganese) 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 580-79055-1 and 580-79055-6:

Sample ID	Laboratory ID
PDI-SG-B471	580-79055-1
PDI-SG-B472	580-79055-2

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 temperatures were recorded. The coolers were received at temperatures within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C. AECOM added rush analysis of metals including manganese, TOC, and total solids for PDI-SG-B472. The rush analyses were reported in laboratory group 580-79055-6 on 8/2/18. Both samples were authorized for the on hold 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 blanks associated with prep batches 284043 (3.89 ug/kg) and 284408 (5.71 ug/kg) at concentrations between the method detection limits (MDLs) and reporting limits. bis(2-Ethylhexyl)phthalate was not detected in the associated samples; therefore, data were not qualified based on these method blank results.

PAHs by EPA Method 8270D-SIM – Fluoranthene (0.373 ug/kg), phenanthrene (0.734 ug/kg), and pyrene (0.314 ug/kg) were detected in the method blank associated with prep batch 284059 at concentrations between the MDLs and reporting limits. Fluoranthene, phenanthrene, and pyrene were detected in PDI-SG-B471 at concentrations significantly greater than the method blank results; therefore, data were not qualified based on these method blank results.

Fluoranthene (0.386 ug/kg) was detected in the method blank associated with prep batch 286035 at a concentration between the MDL and reporting limit. Fluoranthene was detected in PDI-SG-B472 at a concentration between the reporting limit and MDL with an elevated reporting limit due to a dilution that was required prior to analysis; therefore, the result for fluoranthene in PDI-SG-B472 was qualified as estimated and flagged 'J' based on this method blank result

3. Surrogates – Acceptable except as noted below:

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The surrogate recovery for terphenyl-d14 in the method blank associated with prep batch 284408 (129%) exceeded the control limits of 58-120%. Data were not qualified based on the surrogate recoveries in QC samples (method blank).

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

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-B472. The percent recoveries in the MS (147%) and MSD (145%) exceeded the control limits of 59-123%. bis(2-Ethylhexyl)phthalate was not detected in PDI-SG-B472; therefore, data were not qualified based on the elevated MS/MSD results.

PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B472. The percent recoveries for indeno[1,2,3-cd]pyrene in the MS (127%) and MSD (122%) exceeded the control limits of 65-121%. The result for indeno[1,2,3-cd]pyrene in PDI-SG-B472 was qualified as estimated and flagged 'J' based on the MS/MSD results.

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.

6. Laboratory Duplicate

TPHs by Method NWTPH-Dx – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSD.

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 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 with the following exception. The reporting limits and MDLs for dibenz(a,h)anthracene in PDI-SG-B471 and chrysene in PDI-SG-B472 exceeded the cleanup level for carcinogenic PAHs.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – 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 limit for the result reported as not detected in PDI-SG-B472 exceeded the cleanup level (135 ug/kg), but the MDL did not. The reporting limit and MDL exceeded the cleanup level for bis(2-ethylhexyl)phthalate in PDI-SG-B471.

8. Other Items of Note:

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The laboratory noted that the percent differences (%Ds) for the surrogate terphenyl-d14 in the continuing calibration verifications (CCVs) associated with analytical batches 284567 and 284395 were outside the control limits

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of $\pm 20\%$ (high). As the surrogate recoveries in the associated samples were acceptable, data were not qualified based on these high surrogate %Ds.

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-B471 and PDI-SG-B472.

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 in PDI-SG-B471 was exceeded by 5 days due to a delay in authorization for the analysis. The result for mercury in PDI-SG-B471 was 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 – Zinc (1.55 mg/kg) was detected in the method blank associated with prep batch 280435 at a concentration between the reporting limit and MDL. Zinc was detected in PDI-SG-B472 at a concentration greater than the reporting limit and greater than ten times the method blank result; 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) – Acceptable except as noted below:

Metals by Method 6020B – An MS/MSD and PDS were performed using PDI-SG-B472. The percent recoveries for manganese in the MS (123%), MSD (144%), and PDS (-18%) were outside of the control limits of 80-120%. The sample concentration for manganese in PDI-SG-B472 was greater than four times the spike added; therefore, data were not qualified based on these MS/MSD and PDS results.

Mercury by Method 7471A – An MS/MSD was performed using PDI-SG-B472. Results were acceptable.

5. Laboratory Duplicate – Acceptable

Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B472. Results were comparable.

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Mercury by Method 7471A – A laboratory duplicate was performed using PDI-SG-B472. Results were comparable.

6. Serial Dilution – Acceptable

Metals by Method 6020B – A serial dilution was performed using PDI-SG-B472. 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.

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 and Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for both samples in this laboratory group by 1-47 days. No data qualifiers were assigned based on this holding time exceedance.

2. Blanks – Acceptable except as noted below:

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.

TOC (119 mg/kg) was detected in the method blank associated with analytical batch 284391 at a concentration between the reporting limit and MDL. TOC was detected in PDI-SG-B471 at a concentration significantly greater than the method blank result; 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 and Moisture Content at 70°C – Laboratory duplicates were performed using PDI-SG-B471. Results were comparable.



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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.

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 580-79055-1 and 580-79055-6 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Downtown/Upriver
TestAmerica Laboratory Groups: 580-79055-1 and 580-79055-6

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B471	580-79055-1	SW7471A	Mercury	0.11	mg/kg	0.11 J	h
PDI-SG-B472	580-79055-2	SW8270DSIM	Fluoranthene	40 J	ug/kg	40 J	bl
PDI-SG-B472	580-79055-2	SW8270DSIM	Indeno(1,2,3-cd)pyrene	30 J	ug/kg	30 J	m

Notes:

bl - laboratory blank contamination

h - holding time

J - estimated value

m - matrix spike recovery

mg/kg - milligram per kilogram

ug/kg - microgram per kilogram