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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Stratified Random/Sediment Management Area
Laboratory:	TestAmerica Laboratories, Incorporated, Seattle, WA
Laboratory Group:	580-79278-1
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 Number:	60566335, Task #2.12
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SUMMARY

The data quality review of five surface sediment samples collected between May 14 and June 19, 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 group 580-79278-1:

Sample ID	Laboratory ID
PDI-SG-B420-BL1	580-79278-1
PDI-SG-B423-BL1	580-79278-2
PDI-SG-S114	580-79278-3
PDI-SG-S155	580-79278-4
PDI-SG-S228	580-79278-5

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.

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 -2.9°C. Data were not qualified based on the low cooler temperature. The samples in this laboratory group were frozen after sample collection until they were shipped to TA on August 1, 2018. Samples PDI-SG-S114, PDI-SG-S155, and PDI-SG-S228 were originally marked incorrectly on the COC for NWTPH-Dx, metals, and mercury analyses. AECOM instructed TA to revise the COC and not log the samples for these analyses. TA noted that sample PDI-SG-B423-BL1 was received with a cracked jar; sufficient sample was recovered for all assigned analyses. Only one jar was received by the lab for sample PDI-SG-B420-BL1 and subsampling was required. Sample integrity was not impacted by the subsampling; therefore, data were not qualified.

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. Data were not qualified based on rinsate blank results.

   PAHs by EPA Method 8270D-SIM – The following analytes were detected in the method blank extracted on August 7, 2018, at concentrations between the method detection limits (MDLs) and the reporting limits:

Analyte	Result
2-Methylnaphthalene	0.341 ug/kg
Acenaphthene	0.218 ug/kg
Acenaphthylene	0.330 ug/kg
Anthracene	0.165 ug/kg
Fluoranthene	0.394 ug/kg
Fluorene	0.119 ug/kg
Naphthalene	0.313 ug/kg
Phenanthrene	0.672 ug/kg
Pyrene	0.403 ug/kg

   Fluoranthene, naphthalene, phenanthrene, and pyrene were detected in the associated samples at concentrations greater than the reporting limits and greater than two times the method blank detection; therefore, data were not qualified based on these method blank results. 2-Methylnaphthalene, acenaphthene, acenaphthylene, anthracene, and fluorene in PDI-SG-B423-BL1 and 2-methylnaphthalene, acenaphthylene, and fluorene in PDI-SG-S228 were detected at concentrations below the reporting limits but greater than the MDLs; therefore, the results were qualified as not detected and flagged 'U' at the reporting limits.

3. Surrogates – Acceptable

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

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

   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 performed using PDI-SG-B423-BL1. The percent recoveries in the MS/MSD and relative percent differences (RPDs) for the following analytes were outside of the control limits:

Analyte	MS	MSD	RPD	Control Limits (Matrix spike / RPD)
Benzo[a]pyrene	62%	64%	ok	72-124% / 12%
Benzo[b]fluoranthene	ok	ok	13%	63-121% / 10%
Benzo[k]fluoranthene	61%	58%	ok	63-123% / 15%
Chrysene	58%	59%	ok	69-120% / 10%
Phenanthrene	70%	ok	ok	73-120% / 11%

ok - acceptable

   As two of the three quality control parameters (MS, MSD, and RPD) were acceptable, data were not qualified for benzo[b]fluoranthene or phenanthrene based on the MS/MSD results. The results for benzo[a]pyrene, benzo[k]fluoranthene, and chrysene were qualified as estimated and flagged 'J' based on these MS/MSD results.

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B423-BL1. Results were acceptable.

   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. Laboratory Duplicate

   TPHs by Method NWTPH-Dx – A laboratory duplicate was not performed on 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.

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The reporting limits for PDI-SG-B423-BL1 were raised because of the dilution that was required prior to analysis due to the nature of the sample matrix. The reporting limit (340 ug/kg) exceeded the cleanup level (135 ug/kg), but the MDL (40 ug/kg) did not.

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-B420-BL1 and PDI-SG-B423-BL1.

   Tributyltin by Krone et al. – The laboratory noted that the percent difference (%D) for the surrogate tripentyltin in the continuing calibration verification (CCV) associated with analytical batch 281469 was outside the control limits of ±20% (high). As the surrogate recovery in the associated sample was acceptable, data were not qualified based on this high surrogate %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 holding time for mercury was exceeded in PDI-SG-B420-BL1 and PDI-SG-B423-BL1 by 23 to 50 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. Data were not qualified based on rinsate blank results.

   Metals by Method 6020B – Copper (0.124 mg/kg) was detected in the method blank prepared on August 6, 2018, at a concentration between the reporting limit and MDL. Copper was detected in the associated samples at concentrations greater than the reporting limits and greater than 10 times 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 on samples from this laboratory group. Accuracy and precision were assessed using the LCS/LCSDs.

5. Laboratory Duplicate

   General – Laboratory duplicates were 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 not performed on a sample from 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

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 samples in this laboratory group as the samples were held in freezer storage before shipping to TA. Total solids were analyzed within the time that other analyses were performed for 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. Data were not qualified based on rinsate blank results.

   TOC by Method 9060 – TOC (100 mg/kg) was detected in the method blank analyzed on August 13, 2018, at a concentration between the reporting limit and MDL. TOC was detected in the associated samples at concentrations above the reporting limits and greater than 10 times 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-B423-BL1. Results were comparable.

Moisture Content at 70°C – A laboratory duplicate was performed using PDI-SG-S155. 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. 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.

The silt fractions for samples PDI-SG-B420-BL1 and PDI-SG-B423-BL1 were reported as negative due to the difference between the recovered mass and the calculated recovery, and the low silt/clay content in the samples. This error is inherent for the method; therefore, data were not qualified based on the negative silt results.

1. Laboratory Duplicate – Acceptable except as noted below:

   The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was performed on PDI-SG-S155. The result for the silt fraction for sample PDI-SG-S155 was assigned an 'L' qualifier to indicate that the grain size fraction was greater than 5 percent of the total combined fractions and the RPD for duplicate analysis on the sample fraction was greater than 20%.

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 580-79278-1 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random/Sediment Management Area
TestAmerica Laboratory Group: 580-79278-1

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B420-BL1	580-79278-1	SW7471A	Mercury	0.029	mg/kg	0.029 J	h
PDI-SG-B423-BL1	580-79278-2	SW7471A	Mercury	0.029	mg/kg	0.029 J	h
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	2-Methylnaphthalene	0.58 J	ug/kg	1.1 U	bl
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Acenaphthene	0.51 J	ug/kg	1.1 U	bl
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Acenaphthylene	0.79 J	ug/kg	1.1 U	bl
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Anthracene	0.46 J	ug/kg	1.1 U	bl
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Benzo(a)pyrene	0.84 J	ug/kg	0.84 J	m
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Benzo(k)fluoranthene	0.40 J	ug/kg	0.40 J	m
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Chrysene	1.3	ug/kg	1.3 J	m
PDI-SG-B423-BL1	580-79278-2	SW8270DSIM	Fluorene	0.37 J	ug/kg	1.1 U	bl
PDI-SG-S155	580-79278-4	D7928/D6913	Silt	14.1	%	14.1 L	ld
PDI-SG-S228	580-79278-5	SW8270DSIM	2-Methylnaphthalene	0.71 J	ug/kg	1.3 U	bl
PDI-SG-S228	580-79278-5	SW8270DSIM	Acenaphthylene	0.90 J	ug/kg	1.3 U	bl
PDI-SG-S228	580-79278-5	SW8270DSIM	Fluorene	0.96 J	ug/kg	1.3 U	bl

Notes:
% - percent
bl - laboratory blank contamination
h - holding time
J - estimated value
L - the grain size fraction was greater than 5 percent of the total combined fractions and the RPD for duplicate analysis on the sample fraction was greater than 20%
ld - laboratory duplicate RPD
m - matrix spike recovery
mg/kg - milligram per kilogram
U - Compound was analyzed for, but not detected above the value shown.
ug/kg - microgram per kilogram

Note: Line items where the laboratory result contains a "J" and the final result contains a “U” with a data validation reason code "bl" indicate that the final result is reported as not detected (“U” flag) at the reporting limit.