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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
Laboratory:	TestAmerica Laboratories, Incorporated, Seattle, WA
Laboratory Group:	580-78153-1
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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Reviewed by: Karen Mixon/AECOM	File Name: 580-78153-1 DVR

SUMMARY

The data quality review of nine surface sediment samples and one rinsate blank collected between June 15 and June 17, 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 (surface sediments) and EPA Method 7470A (water), TOC by EPA Method 9060 (surface sediments) and Standard Method (SM) 5310B (water), 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 centigrade (°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, American Society for Testing & Materials (ASTM), Philadelphia, Pennsylvania, 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, and Standard Methods for the Examination of Water and Wastewater. 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 580-78153-1:

Sample ID	Laboratory ID
PDI-SG-B301-BL1	580-78153-1
PDI-SG-B297-BL1	580-78153-2
PDI-SG-B293-BL1	580-78153-3
PDI-SG-B310-BL1	580-78153-4
PDI-SG-B309-BL1	580-78153-5
PDI-SG-B314-BL1	580-78153-6
PDI-SG-B030-BL1	580-78153-7
PDI-SG-B031-BL1	580-78153-8
PDI-SG-B042-BL1	580-78153-9

Sample ID	Laboratory ID
PDI-RB-VV-20160616 (rinsate blank)	580-78153-10

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 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. No discrepancies related to sample identification were noted by TA. PAHs, bis(2-ethylhexyl)phthalate, and tributyltin analyses were authorized for sample PDI-SG-B030-BL1 by AECOM on July 2, 2018, past the holding time indicated in the QAPP. A sample in a different laboratory group was originally requested but the sample container was broken in the laboratory and insufficient sample volume remained 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 except as noted below:

   PAHs by EPA Method 8270D-SIM – As noted under sample receipt, this analysis was authorized and prepped outside the 14-day holding time indicated for PAHs in the QAPP. The results for all PAHs in PDI-SG-B030-BL1 were qualified as estimated and flagged 'J' based on the holding time exceedance.

   bis(2-Ethylhexyl)phthalate by EPA Method 8270D – As noted under sample receipt, this analysis was authorized and prepped outside the 14-day holding time indicated for bis(2-ethylhexyl)phthalate in the QAPP. The result for bis(2-ethylhexyl)phthalate in PDI-SG-B030-BL1 was qualified as estimated and flagged 'J' based on the holding time exceedance.

   Tributyltin by Krone et al. – As noted under sample receipt, this analysis was authorized and prepped outside the 14-day holding time indicated for tributyltin in the QAPP. The result for tributyltin in PDI-SG-B030-BL1 was qualified as estimated and flagged 'J' based on the holding time exceedance.

2. Blanks – Acceptable except as noted below:

   PAHs by EPA Method 8270D-SIM – The following analytes were detected in the method blank that was extracted on July 9, 2018 at concentrations between the method detection limits (MDLs) and the reporting limits:

Analyte	Result
Anthracene	0.213 ug/kg
Benzo[a]anthracene	0.458 ug/kg

Analyte	Result
Benzo[a]pyrene	0.391 ug/kg
Benzo[b]fluoranthene	0.653 ug/kg
Benzo[g,h,i]perylene	0.407 ug/kg
Benzo[k]fluoranthene	0.309 ug/kg
Chrysene	0.796 ug/kg
Dibenz(a,h)anthracene	0.297 ug/kg
Fluorene	0.157 ug/kg
Indeno[1,2,3-cd]pyrene	0.581 ug/kg
Naphthalene	0.194 ug/kg
Phenanthrene	0.712 ug/kg

The following analytes were detected in the method blank that was extracted on July 9, 2018 at concentrations above the reporting limits:

Analyte	Result
Fluoranthene	1.50 ug/kg
Pyrene	1.34 ug/kg

The analytes listed above were reported at concentrations significantly above the blank contamination in PDI-SG-B030-BL1; therefore, no data were qualified based on these method blank results.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – bis(2-Ethylhexyl)phthalate (6.92 ug/kg) was detected in the method blank that was extracted on July 3, 2018 at a concentration below the reporting limit but above the MDL. bis(2-Ethylhexyl)phthalate was not detected in the associated samples; therefore, data were not qualified based on this method blank result.

TPHs by Method NWTPH-Dx – Two rinsate blanks were collected on June 16 and June 21 2018, were reported with laboratory groups 580-78278 (ID 580-78278-4) and 580-78153 (ID 580-78153-10), and are applicable to the samples reported in this laboratory group. TPHs were not detected in these rinsate blanks.

3. Surrogates – Acceptable except as noted below:

PAHs by EPA Method 8270D-SIM – The percent recovery for the surrogate terphenyl-d14 (2%) was outside of the control limits of 57-120% in the method blank associated with analytical batch 278837. Data were not qualified based on QC surrogate recoveries.

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

Tributyltin by Krone et al. – The relative percent difference (RPD) for the LCS/LCSD pair for tributyltin (23%) in the LCS/LCSD associated with analytical batch 279252 exceeded the control limit of 20%. The percent recoveries in the LCS and LCSD were acceptable; therefore, data were not qualified based on the LCS/LCSD RPD.

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

TPHs by Method NWTPH-Dx – An MS/MSD was performed using PDI-SG-B031-BL1. Results were acceptable.

PAHs by EPA Method 8270D-SIM – An MS/MSD was performed using PDI-SG-B030-BL1. The RPD for the MS/MSD pair for anthracene (14%) exceeded the control limit of 12%. The percent recoveries in the MS and MSD were acceptable; therefore, data were not qualified based on this MS/MSD RPD.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – An MS/MSD was performed using PDI-SG-B030-BL1. The percent recoveries in the MS (227%), MSD (180%), and the RPD for the MS/MSD pair (25%) exceeded the control limits of 59-123% and 13%, respectively. The result for bis(2-ethylhexyl)phthalate in PDI-SG-B030-BL1 was previously qualified as estimated and flagged 'J' based on the holding time exceedance and no additional qualification was necessary based on the MS/MSD results.

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 – Acceptable

TPHs by Method NWTPH-Dx – A laboratory duplicate was performed using PDI-SG-B301-BL1. Results were comparable.

7. Reporting Limits – Acceptable except as noted below:

General – Analyte concentrations detected between the MDL and the reporting limit are reported by the laboratory with a 'J' flag. Laboratory 'J'-flagged results are considered estimated results. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

TPHs by Method NWTPH-Dx – The reporting limits for PDI-SG-B042-BL1 were raised due to dilution required to bring the concentration of target analytes within the calibration range of the instrument.

PAHs by EPA Method 8270D-SIM – The reporting limits for PDI-SG-B030-BL1 were raised because of the dilution that was required prior to analysis due to the nature of the sample matrix.

bis(2-Ethylhexyl)phthalate by EPA Method 8270D – The reporting limit for PDI-SG-B030-BL1 was raised because of the dilution that was required prior to analysis due to the nature of the sample matrix. The reporting limit for bis(2-ethylhexyl)phthalate reported as not detected in PDI-SG-B030-BL1 exceeded the cleanup level. The MDL did not exceed the cleanup level in the sample noted above.

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-B301-BL1, PDI-SG-B297-BL1, PDI-SG-B293-BL1, PDI-SG-B310-BL1, PDI-SG-B309-BL1, PDI-SG-B314-BL1, PDI-SG-B030-BL1, PDI-SG-B031-BL1, and PDI-SG-B042-BL1.

METALS ANALYSES

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

1. Holding Times – Acceptable

2. Blanks – Acceptable

   General – Two rinsate blanks were collected on June 16 and June 21 2018, were reported with laboratory groups 580-78278 (ID 580-78278-4) and 580-78153 (ID 580-78153-10), and are applicable to the samples reported in this laboratory group. Metals were not detected in these rinsate blanks.

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-B031-BL1. The percent recovery for copper in the MSD (121%) exceeded the control limits of 80-120%. The percent recovery in the MS and the RPD for the MS/MSD pair were acceptable; therefore, data were not qualified based on this MSD recovery.

   Mercury by Method 7471A – An MS/MSD was performed using PDI-SG-B031-BL1. Results were acceptable.

   Mercury by Method 7470A – An MS/MSD was not performed using a sample from this laboratory group. Accuracy and precision were assessed using the LCS/LCSD.

5. Laboratory Duplicate – Acceptable

   Metals by Method 6020B – A laboratory duplicate was performed using PDI-SG-B031-BL1. Results were comparable.

   Mercury by Method 7471A – A laboratory duplicate was performed using PDI-SG-B031-BL1. Results were comparable.

   Mercury by Method 7470A – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was assessed using the LCS/LCSD.

6. Serial Dilution – Acceptable

   Metals by Method 6020B – A serial dilution was performed using PDI-SG-B031-BL1. 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:

   Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for PDI-SG-B309-BL1, PDI-SG-B314-BL1, PDI-SG-B030-BL1, PDI-SG-B031-BL1, and PDI-SG-B042-BL1 by 4-6 days due to an oversight by the laboratory. No data qualifiers were assigned based on the holding time exceedance.

2. Blanks – Acceptable where applicable, except as noted below:

   TOC by Method 9060 – Two rinsate blanks were collected on June 16 and June 21 2018, were reported with this laboratory groups 580-78278 (ID 580-78278-4) and 580-78153 (ID 580-78153-10), and are applicable to the samples reported in this laboratory group. TOC was detected in 580-78278-4 (0.33 mg/L) at a concentration below the reporting limit but above the MDL. 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 performed using PDI-SG-B031-BL1. Results were acceptable.

5. Laboratory Replicate – Acceptable

   TOC by Method 9060 – A laboratory duplicate and triplicate was performed using PDI-SG-B031-BL1. Results were comparable.

   Total Solids by ASTM Method D-2216 – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was not assessed.

   Moisture Content at 70°C – A laboratory duplicate was not performed using a sample from this laboratory group. Precision was not assessed.

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 group 580-78153-1 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Stratified Random
TestAmerica Laboratory Group: 580-78153-1

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B030-BL1	580-78153-7	SW8270D	Bis(2-ethylhexyl)phthalate	400 U	ug/kg	400 UJ	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Anthracene	27 J	ug/kg	27 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Pyrene	170	ug/kg	170 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Benzo(g,h,i)perylene	47 J	ug/kg	47 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Indeno(1,2,3-cd)pyrene	68 J	ug/kg	68 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Benzo(b)fluoranthene	82 J	ug/kg	82 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Dibenz(a,h)anthracene	37 J	ug/kg	37 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Benz(a)anthracene	58 J	ug/kg	58 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Acenaphthene	120 U	ug/kg	120 UJ	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Phenanthrene	93 J	ug/kg	93 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Fluorene	20 J	ug/kg	20 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Naphthalene	120 U	ug/kg	120 UJ	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	2-Methylnaphthalene	120 U	ug/kg	120 UJ	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Fluoranthene	190	ug/kg	190 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Benzo(k)fluoranthene	36 J	ug/kg	36 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Acenaphthylene	120 U	ug/kg	120 UJ	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Chrysene	95 J	ug/kg	95 J	h
PDI-SG-B030-BL1	580-78153-7	SW8270DSIM	Benzo(a)pyrene	44 J	ug/kg	44 J	h
PDI-SG-B030-BL1	580-78153-7	TA-MS-0346	Tributyltin	190 U	ug/kg	190 UJ	h

h - holding time
J - estimated value
ug/kg - microgram per kilogram
U - Compound was analyzed for, but not detected above the value shown.