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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Subsurface Sediment – Deep Core Stations	
Laboratory:	TestAmerica Laboratories, Incorporated, Tacoma, WA	
Laboratory Group:	580-79329-5 and 580-79329-7	
Analyses/Method:	Polycyclic Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyls (PCBs), Total Organic Carbon (TOC), Total Solids, and Grain Size	
Validation Level:	Stage 2A	
AECOM Project Number:	60566335, Task #2.12	
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Reviewed by:	Ann Bridge/AECOM	File Name: 580-79329-5 and -7 DVR

SUMMARY

The data quality review of 4 subsurface sediment samples collected on August 8, 2018, has been completed. Samples were analyzed for PAHs by EPA Method 8270D modified by selected ion monitoring (SIM), PCBs by EPA Method 8082A, TOC by EPA Method 9060, total solids by American Society for Testing and Materials (ASTM) Method D-2216, moisture content at 70 degrees centigrade (°C), and 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)* and Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, Pennsylvania. 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 580-79329-5 and -7:

Sample ID	Laboratory ID
PDI-SC-S221-0to2	580-79329-40
PDI-SC-S221-2to4	580-79329-41
PDI-SC-S221-4to6	580-79329-42
PDI-SC-S221-6to8.1	580-79329-43

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 results reported in this sample set are included in Table 1.

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Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Subsurface Sediment – Deep Core Stations
TA Lab Group: 580-79329-5 and -7

SAMPLE RECEIPT

Upon receipt by TA, the sample jar information was compared to the associated 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.

The samples were frozen by the TestAmerica Sacramento laboratory and shipped to the Tacoma laboratory on September 26, 2018, where they remained frozen until thawed for analysis.

ORGANIC ANALYSES

Samples were analyzed for PAHs and PCBs by the methods identified in the introduction to this report.

1. Holding Times – Acceptable

2. Initial and Continuing Calibration Verifications – Acceptable except as noted below:

   PCBs by Method 8082A – The percent difference (%D) for the following analytes were recovered outside the control limits of ±20% for individual peaks in the continuing calibration verifications (CCVs) associated with the analytical batches below:

Analytical Batch	Analyte	Column 1C %D	Column 2C %D
287725	PCB-1016	low	low
	PCB-1221	low	low
	PCB-1232	ok	low
	PCB-1248	ok	low
	PCB-1260	ok	low

Notes:
ok = acceptable

The laboratory narrative only noted if the average %D for initial calibration check samples (ICVs) and CCVs did not meet the ±20% criteria. As part of this review, all CCV results were reviewed and the individual peaks were assessed using the ±20% criteria. For PCB-1232, PCB-1248, and PCB-1260, the results were reported from the passing column; therefore, data were not qualified based on the CCV %Ds. For PCB-1016 and PCB-1221, the non-detect results were qualified as having estimated reporting limits and flagged “UJ” and the detected results were flagged “J” due to the low CCV %Ds:

Sample ID	Analyte	Final Result (µg/kg)
PDI-SC-S221-0to2	PCB-1016	ND UJ
PDI-SC-S221-0to2	PCB-1221	ND UJ
PDI-SC-S221-2to4	PCB-1016	20 J
PDI-SC-S221-2to4	PCB-1221	ND UJ
PDI-SC-S221-4to6	PCB-1016	23 J
PDI-SC-S221-4to6	PCB-1221	ND UJ
PDI-SC-S221-6to8.1	PCB-1016	12 J
PDI-SC-S221-6to8.1	PCB-1221	ND UJ

Notes:
µg/kg = micrograms per kilogram
ND = not detected
PCB = polychlorinated biphenyl
UJ = material analyzed for but not detected and sample quantitation limit estimated.

3. Blanks – Acceptable

4. Surrogates – Acceptable except as noted below:

   PCBs by EPA Method 8082A – The percent recoveries for tetrachloro-m-xylene in the following samples were outside of the control limits of 58–122%:

Sample	Surrogate	% Recovery
PDI-SC-S221-0to2	Tetrachloro-m-xylene (2C)	55%
PDI-SC-S221-2to4	Tetrachloro-m-xylene (1C/2C)	56%/51%
PDI-SC-S221-4to6	Tetrachloro-m-xylene (1C/2C)	55%51%

   Because only one of the two surrogate spike recoveries did not meet project criteria, no results were qualified.

5. Laboratory Control Sample – Acceptable except as noted below:

   PAHs by Method 8270D-SIM – The LCS recovery for anthracene in analytical batch 286695 was outside of the control limits:

Analyte	LCS	Control Limits
Anthracene	141%	73–125%

   The detected results for anthracene in samples PDS-SC-S221-4to6 and PDI-SC-S221-6to8.1 are qualified as estimated concentrations and flagged “J” due to the high LCS recovery. Non-detect results are not qualified based on the high LCS recovery.

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

PAHs by Method 8270D-SIM – An MS/MSD was performed using PDI-SC-S221-2to4. The percent recoveries and RPDs for the following analytes were outside of the control limits:

Analyte	MS	MSD	RPD	Control Limits (Matrix Spike / RPD)
2-Methylnaphthalene	ok	66%	ok	68–120% / 12%
Benzo[a]pyrene	ok	70%	ok	72–124%/12%
Benzo[g,h,i]perylene	ok	62%	ok	63–120% / 14%
Chrysene	ok	ok	19%	69–120%/10%
Dibenz(a,h)anthracene	59%	60%	ok	70–125% / 13%
Indeno[1,2,3-cd]pyrene	ok	63%	ok	65–121/10%
Naphthalene	ok	65%	ok	70–120%/11%

Notes:
MS = Matrix Spike
MSD = Matrix Spike Duplicate
ok = acceptable
RPD = relative percent difference

As two of the three quality control parameters (MS, MSD, and RPD) were acceptable for 2-methylnaphthalene, benzo[a]pyrene, benzo[g,h,i]perylene, chrysene, indeno[1,2,3-cd]pyrene, and naphthalene, these data were not qualified. The result for dibenz(a,h)anthracene was qualified as having as estimated reporting limit and flagged 'UJ' in PDI-SC-S221-2to4 based on the MS/MSD results.

PCBs by EPA Method 8082A – An MS/MSD was performed using PDI-SC-S221-2to4. The percent recoveries for the following analytes were outside of the control limits:

Analyte	MS	MSD	RPD	Control Limits (Matrix Spike / RPD)
PCB-1016	38%	19%	ok	64–120% / 21%
PCB-1260	52%	37%	ok	63–130% / 25%

Notes:
MS = Matrix Spike
MSD = Matrix Spike Duplicate
ok = acceptable
RPD = relative percent difference

The results for PCB-1016 and PCB-1260 were qualified as estimated and flagged "J" in PDI-SC-S221-2to4 based on the MS/MSD results.

7. Field Duplicate – Field duplicate not included with this data set.

8. Reporting Limits and Chromatographic review – Acceptable except as noted below:

   General – One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the MDLs but below the reporting limits. Laboratory 'J'-flagged results are considered estimated. As the result is between the MDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

   PCBs by EPA Method 8082A – Chromatograms were reviewed to confirm target analytes were properly identified. The review confirmed target analytes were properly identified and reported by the laboratory.

   PCBs by EPA Method 8082A – All samples required a copper clean-up to reduce matrix interferences caused by sulfur.

   PAHs by Method 8270D-SIM – All samples required dilution prior to analysis due to the nature of the sample matrix. The reporting limits have been adjusted accordingly.

9. Other Items of Note: None.

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.

   TOC by Method 9060 – Samples were frozen to preserve hold time and removed from freezer and thawed prior to analysis.

   Moisture Content at 70°C – The 7-day holding time indicated for total solids in the QAPP was exceeded for all samples in the laboratory group. No data qualifiers were assigned based on the holding time exceedances.

2. Blanks – Acceptable.

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-SC-S221-2to4. Results were acceptable.

5. Field Duplicate – Field duplicate not included with this data set.

6. Laboratory Replicate – Acceptable

   TOC by Method 9060 – Laboratory duplicate and triplicate were performed using PDI-SC-S221-2to4. Results were comparable.

Total Solids by Method D2216 – Laboratory duplicate not provided in this laboratory group.

Moisture Content at 70°C – Laboratory duplicate not provided in this laboratory group.

7. 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 – Acceptable.

   The laboratory performed duplicate analysis at a rate of 1 per 20 samples per their internal requirements. A laboratory duplicate was performed on PDI-SC-S221-0to2. Results were comparable.

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory groups 580-79329-5 and -7 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Subsurface Sediment – Deep Core Stations
TestAmreica Lab Group: 580-79329-5 and -7

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S221-0to2	580-79329-40	SW8082A	PCB-1016	4.5 U	µg/kg	4.5 UJ	c
PDI-SC-S221-0to2	580-79329-40	SW8082A	PCB-1221	4.5 U	µg/kg	4.5 UJ	c
PDI-SC-S221-2to4	580-79329-41	SW8270DSIM	Dibenz(a,h)anthracene	100 U	µg/kg	100 UJ	m
PDI-SC-S221-2to4	580-79329-41	SW8082A	PCB-1016	20	µg/kg	20 J	m,c
PDI-SC-S221-2to4	580-79329-41	SW8082A	PCB-1221	3.9 U	µg/kg	3.9 UJ	c
PDI-SC-S221-2to4	580-79329-41	SW8082A	PCB-1260	7.9	µg/kg	7.9 J	m
PDI-SC-S221-4to6	580-79329-42	SW8082A	PCB-1016	23	µg/kg	23 J	c
PDI-SC-S221-4to6	580-79329-42	SW8082A	PCB-1221	3.5 U	µg/kg	3.5 UJ	c
PDI-SC-S221-4to6	580-79329-42	SW8270DSIM	Anthracene	5.8 J	µg/kg	5.8 J	l
PDI-SC-S221-6to8.1	580-79329-43	SW8082A	PCB-1016	12	µg/kg	12 J	c
PDI-SC-S221-6to8.1	580-79329-43	SW8082A	PCB-1221	3.5 U	µg/kg	3.5 UJ	c
PDI-SC-S221-6to8.1	580-79329-43	SW8270DSIM	Anthracene	2.9 J	µg/kg	2.9 J	l

µg/kg = micrograms per kilogram
c = calibration issue
ID = identification
J = estimated concentration
l = laboratory control sample recovery
m = matrix spike recovery
U = not detected
UJ = estimated reporting limit