

## Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Portland Harbor Superfund Site  
 Subsurface Sediment – Nearshore Core Stations

Laboratory: TestAmerica Laboratories, Incorporated, Seattle, WA

Laboratory Group: 580-79057-1

Analyses/Method: Polycyclic Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyls (PCBs),  
 Total Organic Carbon (TOC), Total Solids, and Grain Size

Validation Level: Stage 2A/Stage 4 on EPA split sample

AECOM Project Number: 60566335, Task #2.12

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Reviewed by: Stacy Louie/AECOM File Name: 580-79057-1 DVR

### SUMMARY

The data quality review of 6 subsurface sediment samples collected on July 20, 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 group 580-79057-1:

Sample ID	Laboratory ID
PDI-SC-S036-0to1.4	580-79057-1
PDI-SC-S036-1.4to3.4	580-79057-2
PDI-SC-S036-3.4to5.2	580-79057-3
PDI-SC-S022-0to2	580-79057-4
PDI-SC-S022-2to4	580-79057-5
PDI-SC-S022-4to6	580-79057-6

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*, 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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**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. Six samples were listed on the chain-of-custody but were not received by the laboratory. AECOM clarified that those samples were submitted in an earlier shipment.

**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/CCV or CCB ID	Analyte	%D
CCV 580-281356	PCB-1232	high
	PCB-1242	high
	PCB-1248	high
	PCB-1221(c)	high
	PCB-1254(c)	high
	PCB-1016(c)	high
	PCB-1260(c)	high
CCV 580-281356	TCMX (ss)	high
CCV 580-280527	PCB-1232	high
CCB 580-281356/7	ss(c)	out

(c) = confirmation column only  
(ss) = surrogate spike

For all samples associated with these CCVs, the above analytes were not detected; therefore, data were not qualified based on the high CCV %Ds.

The percent differences (%D) for the following analytes were outside (low) the control limits of ±20% in the continuing calibration verifications (CCVs) associated with the analytical batches listed below:

Analytical Batch/CCV or CCB ID	Analyte	%D
CCV 530-280273/3	PCB-1248(c)	low
	PCB-1260(c)	low

(c) = confirmation column only

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For the two samples associated with the low CCV recoveries listed above (PDI-SC-S036-0to1.4 and PDI-SC-S036-1.4to3.4), PCB-1248 was not detected; however, the results are not qualified due to the low CCV recovery because that low CCV recovery was on the confirmation column, not the primary column.

The laboratory narrative only noted cases where the average CCV %D did not meet the ±20% criteria. As part of this review, all individual CCV %Ds were reviewed. As a result of that extended review, the PCB-1254 result of 85 µg/kg for PDI-SC-S036-3.4to5.2 was qualified as an estimated concentration because the result was reported from the primary column (where the CCV recovery was high), not from the second column (where the CCV %D met project criteria).

- 3. Blanks – Acceptable except as noted below:

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

Extraction Date	Analyte	Result
7/27/2018	2-Methylnaphthalene	0.590 J µg/kg
	Acenaphthene	0.237 J µg/kg
	Acenaphthylene	0.160 J µg/kg
	Anthracene	0.140 J µg/kg
	Benzo[a]anthracene	0.203 J µg/kg
	Benzo[a]pyrene	0.146 J µg/kg
	Benzo[k]fluoranthene	0.162 J µg/kg
	Fluorene	0.226 J µg/kg
	Naphthalene	0.391 J µg/kg
	Phenanthrene	0.653 J µg/kg
	Pyrene	0.212 J µg/kg

J = reported concentrations were above the MDLs but below the reporting limit  
 µg/kg = micrograms per kilogram

All eleven compounds detected in the method blank were detected in one or more of the associated project samples. Because all the samples were diluted prior to analysis (5X and 10X), sample results that were flagged by the laboratory as “JB” (detected in associated blank and reported at a concentration less than the RL but greater than the MDL) were flagged as estimated “J” with a reason code of “bl” due to potential high bias due to method blank contamination. The following results were qualified:

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Sample ID	Analyte	Final Result	Reason Code
PDI-SC-S036-0to1.4	Acenaphthylene	2.7 J	bl
PDI-SC-S022-0to2	2-Methylnaphthalene	1.5 J	bl
PDI-SC-S022-0to2	Acenaphthene	1.1 J	bl
PDI-SC-S022-0to2	Acenaphthylene	0.9 J	bl
PDI-SC-S022-0to2	Anthracene	3.3 J	bl
PDI-SC-S022-0to2	Benzo[a]anthracene	5.2 J	bl
PDI-SC-S022-0to2	Fluorene	2.0 J	bl
PDI-SC-S022-0to2	Naphthalene	2.8 J	bl
PDI-SC-S022-2to4	Benzo[a]anthracene	3.2 J	bl
PDI-SC-S022-2to4	Phenanthrene	3.0 J	bl
PDI-SC-S022-4to6	2-Methylnaphthalene	1.5 J	bl
PDI-SC-S022-4to6	Phenanthrene	2.9 J	bl
PDI-SC-S022-4to6	Benzo[a]anthracene	2.5 J	bl

4. Surrogates – Acceptable except as noted below:

PCBs by EPA Method 8082A – The percent recoveries for decachlorobiphenyl and tetrachloro-m-xylene in the following samples were outside of the control limits of 54–142% and 58–122%, respectively:

Sample	Surrogate	% Recovery
PDI-SC-S036-0to1.4	Decachlorobiphenyl	50%
	Tetrachloro-m-xylene	54%
PDI-SC-S036-1.4to3.4	Decachlorobiphenyl	52%
	Tetrachloro-m-xylene	29%
PDI-SC-S036-3.4to5.2	Decachlorobiphenyl	150%
	Tetrachloro-m-xylene	57%

The PCB results in samples PDI-SC-S036-0to1.4, PDI-SC-S036-1.4to3.4, and PDI-SC-S036-3.4to5.2 were qualified as estimated and flagged ‘J’ or ‘UJ’ based on the surrogate spike recoveries.

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

PAHs by Method 8270D-SIM – The percent recoveries in the LCS were outside of the control limits in the LCS July 27, 2018:

Analyte	LCS	LCSD	RPD	LCS Control Limits
Indeno[1,2,3-cd]pyrene	127%	NA	NC	65–121%

NA = not analyzed  
 NC = not calculated

Since the only laboratory quality control sample analyzed was the LCS, the detected results for indeno[1,2,3-cd]pyrene in PDI-SC-S036-0to1.4, PDI-SC-S036-1.4to3.4, PDI-SC-S036-

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3.4to5.2, and PDI-SC-022-0to2 were qualified as estimated and flagged 'J' based on the LCS recovery.

In addition, no LCSD was reported, and no MS/MSD was performed, so precision data is not available for this SDG.

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

PAHs by Method 8270D-SIM – An MS/MSD was not performed on a sample from this laboratory group. In addition, an LCSD was not performed. Therefore, precision for the analytical batch could not be assessed and accuracy was assessed using the LCS.

PCBs by EPA Method 8082A – An MS/MSD was performed using PDI-SC-S022-0to2. The percent recoveries for the following analytes were outside of the control limits:

Analyte	MS	MSD	RPD	Control Limits (Matrix Spike / RPD)
PCB-1016	63%	59%	ok	64-120%/21
PCB-1260	52%	55%	ok	63-130%/25

ok = acceptable

The results for PCB-1016 and PCB-1260 were qualified as estimated and flagged 'UJ' in PDI-SC-S022-0to2 based on the MS/MSD results.

7. Field Duplicate – None submitted with this sample group.
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.

PAHs by Method 8270D-SIM – All samples required dilution prior to analysis due to the nature of the sample matrix.

PCBs by EPA Method 8082A – All samples and the associated MS/MSD (on sample PDI-SC-S022-0to2) required a copper clean-up to reduce matrix interferences caused by sulfur.

9. Other Items of Note: None

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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:

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 by 2 days due to an oversight by the laboratory. No data qualifiers were assigned based on the holding time exceedance.

2. Blanks – Acceptable.
3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Acceptable.
4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Not Performed.
5. Field Duplicate – None submitted with this sample group.
6. Laboratory Replicate – Acceptable.
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 using PDI-SC-S022-4to6. Results were within the project limits of 20%, with the exception of coarse sand (29%). However, because the coarse sand was less than 1 percent of the total fraction; this result is not qualified.

## **OVERALL ASSESSMENT OF DATA**

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group 580-79057-1 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
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Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S036-0to1.4	580-79057-1	SW8270D	Indeno[1,2,3-cd]pyrene	230	µg/Kg	230 J	l
PDI-SC-S036-0to1.4	580-79057-1	SW8270D	Acenaphthylene	27 J	µg/Kg	27 J	bl
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1016	3.8 U	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1221	3.8 U	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1232	3.8 U	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1242	3.8 U	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1248	3.8 U	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1254	ND	µg/Kg	3.8 UJ	s
PDI-SC-S036-0to1.4	580-79057-1	SW8082A	PCB-1260	11	µg/Kg	11 J	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8270D	Indeno[1,2,3-cd]pyrene	630	µg/Kg	630 J	l
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1016	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1221	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1232	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1242	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1248	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1254	3.0 U	µg/Kg	3.0 UJ	s
PDI-SC-S036-1.4to3.4	580-79057-2	SW8082A	PCB-1260	21	µg/Kg	21 J	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8270D	Indeno[1,2,3-cd]pyrene	2,100	µg/Kg	2,100 J	l
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1016	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1221	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1232	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1242	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1248	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1254	85	µg/Kg	85 J	s,c
PDI-SC-S036-3.4to5.2	580-79057-3	SW8082A	PCB-1260	3.1 U	µg/Kg	3.1 UJ	s
PDI-SC-S022-0to2	580-79057-4	SW8270D	Indeno[1,2,3-cd]pyrene	3.1 J	µg/Kg	3.1 J	l
PDI-SC-S022-0to2	580-79057-4	SW8270D	2-Methylnaphthalene	1.5 J	µg/Kg	1.5 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Acenaphthene	1.1 J	µg/Kg	1.1 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Acenaphthylene	0.9 J	µg/Kg	0.9 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Anthracene	3.3 J	µg/Kg	3.3 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Benzo[a]anthracene	5.2 J	µg/Kg	5.2 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Fluorene	2.0 J	µg/Kg	2.0 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8270D	Naphthalene	2.8 J	µg/Kg	2.8 J	bl
PDI-SC-S022-0to2	580-79057-4	SW8082A	PCB-1016	2.9 U	µg/Kg	2.9 UJ	m

**Table 1**  
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PDI-SC-S022-0to2	580-79057-4	SW8082A	PCB-1260	2.9 U	µg/Kg	2.9 UJ	m
PDI-SC-S022-2to4	580-79057-5	SW8270D	Benzo[a]anthracene	3.2 J	µg/Kg	3.2 J	bl
PDI-SC-S022-2to4	580-79057-5	SW8270D	Phenanthrene	3.0 J	µg/Kg	3.0 J	bl
PDI-SC-S022-4to6	580-79057-6	SW8270D	2-Methylnaphthalene	1.5 J	µg/Kg	1.5 J	bl
PDI-SC-S022-4to6	580-79057-6	SW8270D	Phenanthrene	2.9 J	µg/Kg	2.9 J	bl
PDI-SC-S022-4to6	580-79057-6	SW8270D	Benzo[a]anthracene	2.5 J	µg/Kg	2.5 J	bl

µg/Kg - micrograms per kilogram

bl - blank contamination

c - continuing calibrations

ID - identification

J - estimated value

I - laboratory control sample

m - matrix spike/matrix spike duplicate

s - surrogate spike

U - not detected

UJ - estimated reporting limit