

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Sediment Management Area			
Laboratory:	ALS Environmental, Kelso, WA			
Laboratory Group:	K1804770			
Analyses/Method:	Chlorinated Pesticides, Polycyclic Aromatic Hydrocarbons (PAHs), and Total Solids			
Validation Level:	Stage 2A			
AECOM Project Number:	60566335 Task #2.12			
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SUMMARY

The data quality review of 13 surface sediment samples collected on May 16 and May 17, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS), PAHs by EPA Method 8270D modified by selected ion monitoring (SIM), and total solids by EPA Method 160.3-modified at ALS Environmental (ALS) located in Kelso, Washington. The analyses were performed in general accordance with the methods specified in EPA's *Test Methods for Evaluating Solid Waste (SW-846), Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP SVM-PESTMS2), and *Methods for Chemical Analysis of Water and Wastes*, March 1983. 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 K1804770:

Sample ID	Laboratory ID		
PDI-SG-S165	K1804770-001		
PDI-SG-S166	K1804770-002		
PDI-SG-S190	K1804770-003		
PDI-SG-S196	K1804770-004		
PDI-SG-S151	K1804770-005		
PDI-SG-S153	K1804770-006		
PDI-SG-S136	K1804770-007		
PDI-SG-S137	K1804770-008		
PDI-SG-S138	K1804770-009		
PDI-SG-S139	K1804770-010		
PDI-SG-S141	K1804770-011		
PDI-SG-S146	K1804770-012		
PDI-SG-S148	K1804770-013		

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,



Data Validation Report Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Surface Sediment – Sediment Management Area ALS Lab Group: K1804770

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 ALS, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperatures were recorded. No discrepancies related to sample identification were noted by ALS and the coolers were received at temperatures within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C.

ORGANIC ANALYSES

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

1. Holding Times – Acceptable except as noted below:

<u>Chlorinated Pesticides by EPA Method 1699-modified</u> – PDI-SG-S146, PDI-SG-S138 for 2,4'-DDD and 4,4'-DDD, and PDI-SG-S136 for 2,4'-DDT, 4,4'-DDD, and 4,4'-DDT were re-extracted 36-61 days past the method-recommended holding time of 14 days after sample collection. Per ALS-Kelso protocol, the samples were frozen in archive after the initial extraction and the samples were thawed for less than 14 days; therefore, the samples were not re-extracted outside the holding time.

- 2. Initial and Continuing Calibration Verifications Acceptable
- 3. Blanks Acceptable

<u>General</u> - 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.

<u>PAHs by EPA Method 8270D-SIM</u> – The following analytes were detected between the method detection limit (MDL) and the reporting limit in the method blank extracted on May 29, 2018:

Analyte	Result (ug/kg)		
Fluoranthene	0.062		
Pyrene	0.052		
Benz(a)anthracene	0.058		
Benzo(g,h,i)perylene	0.11		

The results for the PAHs noted in the above table were reported at concentrations significantly higher than the method blank concentrations in the associated samples; therefore, data were not qualified based on these method blank results.

- 4. Surrogates Acceptable
- 5. Internal Standards Acceptable



Data Validation Report Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Surface Sediment – Sediment Management Area ALS Lab Group: K1804770

6. Laboratory Control Sample (LCS) – Acceptable except as noted below:

<u>Chlorinated Pesticides by EPA Method 1699-modified</u> – The percent recoveries for the following analytes in two LCSs were outside the control limits:

Extraction Date	Analyte	LCS	Control Limits
May 29, 2018	2,4'-DDT	127%	77-118%
July 6, 2018	2,4'-DDT	133%	77-118%
	4,4'-DDT	123%	78-116%

The results for 2,4'-DDT in PDI-SG-S153, PDI-SG-S139, PDI-SG-S148, and PDI-SG-S146 and 4,4'-DDT in PDI-SG-S146 were qualified as estimated and flagged 'J' based on these LCS results.

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

<u>Chlorinated Pesticides by EPA Method 1699-modified</u> – MS/MSDs were performed using PDI-SG-S138. The MS/MSDs were extracted for two different batches using 20 grams (batch KQ1806778) and 1 gram (batch KQ1810351). The following percent recoveries and/or relative percent differences (RPDs) for the following analytes were outside the control limits:

					RPD
Batch	Analyte	MS	MSD	Control limit	CL = 40%
KQ1806843	2,4'-DDD	-6,198%	-6,423%	32-169%	ok
(20 grams)	2,4'-DDT	538%	429%	55-161%	ok
	4,4'-DDD	-16,563%	-17,333%	10-190%	ok
	4,4'-DDE	-57%	-58%	35-162%	ok
	4,4'-DDT	-17,863%	3,070%	24-183%	122%
KQ1810351	2,4'-DDD	-288%	-353%	32-169%	ok
(1 gram)	4,4'-DDD	-953%	-108%	10-190%	ok
	4,4'-DDT	-1,233%	-1,337%	24-183%	ok

ok – acceptable CL – control limits

The results for the chlorinated pesticides noted in the table above were not reported from the associated extraction batch, were reported as not detected, or the concentrations were more than four times the spike concentrations in the associated extraction; therefore, data were not qualified for these analytes in PDI-SG-S138 based on the MS/MSD results.

<u>PAHs by EPA Method 8270D-SIM</u> – An MS/MSD was performed using PDI-SG-S138. The percent recovery for fluoranthene in the MSD (35%) was outside the control limits of 42-130%. The percent recovery for fluoranthene in the MS and the RPD for the MS/MSD pair were acceptable; therefore, data were not qualified based on this MSD result.

8. Reporting Limits– Acceptable except as noted below:

<u>Chlorinated Pesticides by EPA Method 1699-modified</u> – 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.



Data Validation Report Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Surface Sediment – Sediment Management Area ALS Lab Group: K1804770

The results for 4,4'-DDT in PDI-SG-S151, PDI-SG-S153, PDI-SG-S138, and PDI-SG-S139 were flagged 'E' by the laboratory to indicate that the concentrations exceeded the linear range of the instrument. The results for 4,4'-DDT in these samples were qualified as estimated and flagged 'J' based on the calibration range exceedance.

The reporting limits for one or more pesticides reported as not detected in multiple samples were elevated due to the moisture content and/or dilution due to matrix interference. The elevated reporting limits and MDLs do not exceed the cleanup level.

CONVENTIONAL ANALYSIS

Samples were analyzed for total solids by EPA Method 160.3-modified.

- 1. Holding Times Acceptable
- 2. Laboratory Duplicate Acceptable

Laboratory duplicates were performed using PDI-SG-S138 and PDI-SG-S148. Results were comparable.

3. Reporting Limits – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group K1804770 is 100%.

Table 1 QA/QC Data Summary Review Portland Harbor Surface Sediment - Sediment Management Area ALS Kelso Laboratory Group: K1804770

				Laboratory		Final	Reason
Sample ID	Laboratory ID	Method	Analyte	Result	Units	Result	Code
PDI-SG-S151	K1804770-005	CWA1699M	4,4'-DDT	200 E	ug/kg	200 J	q
PDI-SG-S153	K1804770-006	CWA1699M	2,4-DDT	7.5	ug/kg	7.5 J	I
PDI-SG-S153	K1804770-006	CWA1699M	4,4'-DDT	230 E	ug/kg	230 J	q
PDI-SG-S138	K1804770-009	CWA1699M	4,4'-DDT	410 E	ug/kg	410 J	q
PDI-SG-S139	K1804770-010	CWA1699M	2,4-DDT	13	ug/kg	13 J	I
PDI-SG-S139	K1804770-010	CWA1699M	4,4'-DDT	240 E	ug/kg	240 J	q
PDI-SG-S146	K1804770-012	CWA1699M	2,4-DDT	4.9	ug/kg	4.9 J	
PDI-SG-S146	K1804770-012	CWA1699M	4,4'-DDT	160	ug/kg	160 J	
PDI-SG-S148	K1804770-013	CWA1699M	2,4-DDT	4.0	ug/kg	4.0 J	

Notes:

E - result exceeded calibration range of the instrument

J - estimated value

I - LCS recovery

ug/kg - microgram per kilogram

q - quantitation issue