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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Surface Sediment – Downtown/Upriver Reaches
Laboratory:	ALS Environmental, Kelso, WA
Laboratory Group:	K1806207
Analyses/Method:	Chlorinated Pesticides and Total Solids
Validation Level:	Stage 2A
AECOM Project Number:	60566335 Task #2.12
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Completed on:	October 16, 2018
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File Name:	K1806207 DVR

SUMMARY

The data quality review of 25 surface sediment samples and one rinsate blank collected between June 29 and July 1, 2018, has been completed. The samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/MS/MS) and/or 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 *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 were associated with laboratory group K1806207:

Sample ID	Laboratory ID
PDI-SG-B434	K1806207-001
PDI-SG-B435	K1806207-002
PDI-SG-B441	K1806207-003
PDI-SG-B442	K1806207-004
PDI-SG-B439	K1806207-005
PDI-SG-B440	K1806207-006
PDI-SG-B445	K1806207-007
PDI-SG-B446	K1806207-008
PDI-SG-B447	K1806207-009
PDI-SG-B449	K1806207-010
PDI-SG-B443	K1806207-011
PDI-SG-B444	K1806207-012
PDI-SG-B448	K1806207-013
PDI-SG-B451	K1806207-014
PDI-SG-B455	K1806207-015
PDI-SG-B450	K1806207-016
PDI-SG-B454	K1806207-017
PDI-SG-B453	K1806207-018
PDI-SG-B453-D (Duplicate of PDI-SG-B453)	K1806207-019

Sample ID	Laboratory ID
PDI-SG-B452	K1806207-020
PDI-SG-B457	K1806207-021
PDI-SG-B459	K1806207-022
PDI-SG-B460	K1806207-023
PDI-SG-B461	K1806207-024
PDI-SG-B461-D (Duplicate of PDI-SG-B461)	K1806207-025
PDI-SG-RB-20180630 (Rinsate blank)	K1806207-026

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.

SAMPLE RECEIPT

Upon receipt by ALS, the sample jar information was compared to the chain-of-custody (COC) and the cooler temperature was recorded. No discrepancies related to sample identification were noted by ALS and the cooler was received at a temperature within the EPA-recommended limits of greater than 0°C and less than or equal to 6°C. The samples were received by the laboratory on July 2, 2018, and placed on frozen hold. Samples were authorized for analysis on August 16, 2018.

ORGANIC ANALYSIS

The samples were analyzed for chlorinated pesticides by EPA Method 1699-modified.

1. Holding Times – Acceptable

   The samples in this laboratory group were extracted 47-53 days past the method-recommended holding time of 14 days. Per ALS-Kelso protocol, the samples were frozen in archive until extraction and were thawed for less than 14 days; therefore, the samples were not extracted outside the holding time.

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

   The percent differences (%Ds) for 2,4'-DDE (38.1%) and 4,4'-DDE (54.5%) exceeded the control limit of ±25% in the continuing calibration verification (CCV) analyzed on September 18, 2018. 2,4'-DDE was not detected in the associated samples; therefore, data were not qualified for this analyte based on this CCV result. The results for 4,4'-DDE in PDI-SG-B443, PDI-SG-B444, PDI-SG-B448, PDI-SG-B451, PDI-SG-B455, PDI-SG-B454, PDI-SG-B453, PDI-SG-B453-D, PDI-SG-B452, PDI-SG-B457, PDI-SG-B459, PDI-SG-B460, PDI-SG-B461, and PDI-SG-B461-D were qualified as estimated and flagged 'J' based on this CCV result.

3. Blanks – Acceptable except as noted below:

   One rinsate blank was reported with this laboratory group. 4,4'-DDT (0.11 ng/L) was detected at a concentration between the method detection limit (MDL) and the reporting limit. Additional rinsate blanks associated with these samples were 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 detections.

4. Surrogates – Acceptable except as noted below:

   The percent recoveries for 4,4'-DDD-d4 in PDI-SG-RB-20180630 (125%) and the laboratory control sample extracted on July 3, 2018 (125%) exceeded the control limits of 5-120%. Data were not qualified based on surrogate exceedances in QC samples. The results for 2,4'-DDD, 2,4'-DDE, 4,4'-DDD, and 4,4'-DDE in PDI-SG- RB-20180630 were qualified as estimated and flagged 'UJ' based on this surrogate recovery.

5. Internal Standards – Acceptable except as noted below:

   The internal standard area counts for pyrene-d10 exceeded the control limits of 50-200% (high) in multiple samples reported in this laboratory group and associated QC samples. Pyrene-d10 is associated with the labeled surrogate compound recoveries which are within control limits in the samples referenced above; therefore, data were not qualified based on these internal standard outliers.

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

   The percent recoveries and/or relative percent differences (RPDs) for the following analytes in the LCSs and/or the LCSDs were outside the control limits:

Extraction Date	Analyte	LCS	LCSD	Control limits	RPD CL = 30%
7/3/18 (rinsate blank)	2,4'-DDD	ok	78%	79-113%	ok
	2,4'-DDE	51%	49%	75-117%	ok
	4,4'-DDE	65%	63%	76-115%	ok
	4,4'-DDT	114%	116%	85-113%	ok
	Aldrin	ok	114%	81-113%	ok
	alpha-Chlordane	ok	ok	69-130%	39%
	cis-Nonachlor	270%	188%	59-138%	36%
	gamma-Chlordane	130%	ok	72-127%	37%
	Oxychlordane	125%	ok	77-123%	ok
8/31/18 (sediments)	2,4'-DDD	124%	NA	73-122%	NA
9/6/18 (sediments)	2,4'-DDD	126%	NA	73-122%	NA

ok – acceptable NA – not applicable CL – control limits

2,4'-DDD, aldrin, alpha-chlordane, cis-nonachlor, gamma-chlordane, and oxychlordane were either not detected in the rinsate blank, or 2 out of 3 quality control parameters (LCS, LCSD, and/or RPD) were acceptable; therefore, data were not qualified based on the LCS/LCSD

extracted on July 3, 2018. The results for 2,4'-DDE, and 4,4'-DDE were qualified base on surrogate results as described in Section 4; therefore, no further qualification was necessary. The result for 4,4'-DDT in PDI-SG-RB-180630 was qualified as estimated and flagged 'J' based on the LCS/LCSD extracted on July 3, 2018.

The results for 2,4'-DDD in PDI-SG-B439, PDI-SG-B448, PDI-SG-B457, and PDI-SG-B459 were qualified as estimated and flagged 'J' based on the LCSs extracted on August 31, 2018 and September 6, 2018.

7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

An MS/MSD was not performed in association with the rinsate blank. Precision and accuracy were assessed using the LCS/LCSD results.

An MS/MSD was performed using PDI-SG-B434. Results were acceptable.

An MS/MSD was performed using PDI-SG-B457. The following percent recoveries were outside the control limits:

Analyte	MS	MSD	Control limit
4,4'-DDT	-301%	-305%	24-183%
Aldrin	231%	225%	52-151%

ok - acceptable

Aldrin was reported as not detected in PDI-SG-B457; therefore, no data were qualified based on this MS/MSD result. The result for 4,4'-DDT in PDI-SG-B457 was qualified as estimated and flagged 'J' based on this MS/MSD result.

8. Field Duplicate – Acceptable except as noted below:

Field duplicates were submitted for PDI-SG-B453 and PDI-SG-B461 and identified as PDI-SG-B453-D and PDI-SG-B461-D. The RPD for 4,4'-DDT (75%) was more than 50% in the PDI-SG-B461/ PDI-SG-B461-D field duplicate pair. The results for 4,4'-DDT in this field duplicate pair were less than five times the reporting limits; therefore, data were not qualified based on the field duplicate RPD.

9. Reporting Limits – Acceptable except as noted below:

One or more results in multiple samples 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.

The reporting limits for the chlorinated pesticides reported as not detected in these sediment samples were elevated due to the moisture content and/or lower extraction volume used due to matrix interference. The reporting limits and MDLs for dieldrin exceeded the cleanup level in all sediment samples reported in laboratory group K1806207.

CONVENTIONAL ANALYSIS

The 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-B443 and PDI-SG-B457. Results were comparable.

3. Field Duplicate – Acceptable

   Field duplicates were submitted for PDI-SG-B453 and PDI-SG-B461 and identified as PDI-SG-B453-D and PDI-SG-B461-D. Results were comparable.

4. 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 K1806207 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Surface Sediment - Downtown/Upriver Reaches
ALS Kelso Laboratory Group: K1806207

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SG-B439	K1806207-005	CWA1699M	2,4-DDD	0.71	ug/kg	0.71 J	l
PDI-SG-B443	K1806207-011	CWA1699M	4,4'-DDE	1.2	ug/kg	1.2 J	c
PDI-SG-B444	K1806207-012	CWA1699M	4,4'-DDE	1.3	ug/kg	1.3 J	c
PDI-SG-B448	K1806207-013	CWA1699M	2,4-DDD	0.26 J	ug/kg	0.26 J	l
PDI-SG-B448	K1806207-013	CWA1699M	4,4'-DDE	2.3	ug/kg	2.3 J	c
PDI-SG-B451	K1806207-014	CWA1699M	4,4'-DDE	0.49	ug/kg	0.49 J	c
PDI-SG-B455	K1806207-015	CWA1699M	4,4'-DDE	0.85	ug/kg	0.85 J	c
PDI-SG-B454	K1806207-017	CWA1699M	4,4'-DDE	0.91	ug/kg	0.91 J	c
PDI-SG-B453	K1806207-018	CWA1699M	4,4'-DDE	1.2	ug/kg	1.2 J	c
PDI-SG-B453-D	K1806207-019	CWA1699M	4,4'-DDE	1.3	ug/kg	1.3 J	c
PDI-SG-B452	K1806207-020	CWA1699M	4,4'-DDE	1.1	ug/kg	1.1 J	c
PDI-SG-B457	K1806207-021	CWA1699M	2,4-DDD	0.37 J	ug/kg	0.37 J	l
PDI-SG-B457	K1806207-021	CWA1699M	4,4'-DDE	3.7	ug/kg	3.7 J	c
PDI-SG-B457	K1806207-021	CWA1699M	4,4'-DDT	29	ug/kg	29 J	m
PDI-SG-B459	K1806207-022	CWA1699M	2,4-DDD	0.76	ug/kg	0.76 J	l
PDI-SG-B459	K1806207-022	CWA1699M	4,4'-DDE	1.8	ug/kg	1.8 J	c
PDI-SG-B460	K1806207-023	CWA1699M	4,4'-DDE	1.6	ug/kg	1.6 J	c
PDI-SG-B461	K1806207-024	CWA1699M	4,4'-DDE	1.5	ug/kg	1.5 J	c
PDI-SG-B461-D	K1806207-025	CWA1699M	4,4'-DDE	1.2	ug/kg	1.2 J	c
PDI-SG-RB-20180630	K1806207-026	CWA1699M	2,4-DDD	0.51 U	ng/L	0.51 UJ	s
PDI-SG-RB-20180630	K1806207-026	CWA1699M	2,4-DDE	0.51 U	ng/L	0.51 UJ	s
PDI-SG-RB-20180630	K1806207-026	CWA1699M	4,4'-DDD	0.51 U	ng/L	0.51 UJ	s
PDI-SG-RB-20180630	K1806207-026	CWA1699M	4,4'-DDE	0.51 U	ng/L	0.51 UJ	s
PDI-SG-RB-20180630	K1806207-026	CWA1699M	4,4'-DDT	0.11 J	ng/L	0.11 J	l

Notes:
c - calibration issue
J - estimated value
l - LCS recovery
m - matrix spike recovery
ng/L - nanogram per liter
s - surrogate recovery
ug/kg - microgram per kilogram
U - Compound was analyzed for, but not detected above the value shown.