

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: K1806305

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2A

AECOM Project

Number: 60566335 Task #2.12

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File Name: K1806305 DVR

SUMMARY

The data quality review of 10 surface sediment samples and one rinsate blank collected on July 2 and July 3, 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 K1806305:

Sample ID	Laboratory ID
PDI-SG-B458	K1806305-001
PDI-SG-B470	K1806305-002
PDI-SG-B469	K1806305-003
PDI-SG-B456	K1806305-004
PDI-SG-B462	K1806305-005
PDI-SG-B463	K1806305-006
PDI-SG-B464	K1806305-007
PDI-SG-B466	K1806305-008
PDI-SG-B468	K1806305-009
PDI-SG-B429	K1806305-010
RB-VV-180703-1720 (Rinsate Blank)	K1806305-011

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 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 COC was incorrectly marked for polychlorinated biphenyl analysis for these samples. At the request of AECOM, the sample analysis was corrected to chlorinated pesticides and the COC was revised and re-submitted to ALS. The samples were received by the laboratory on July 5, 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 sediment samples in this laboratory group were extracted 44 days past the method-recommended holding time of 14 days. As described above, 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

3. Blanks – Acceptable

One rinsate blank was reported with this laboratory group and is associated with the sediment samples reported in K1806305. No target analytes were detected in RB-VV-180703-1720.

4. Surrogates – Acceptable except as noted below:

The following percent recoveries for 4,4'-DDD-d4 exceeded the control limits of 5-120% in the QC samples associated with the rinsate blank:

Analyte	% Recovery
Method Blank	132%
Laboratory Control Sample	131%
Laboratory Control Sample Duplicate	132%

Data were not qualified based on surrogate exceedances in QC samples.

5. Internal Standards – Acceptable except as noted below:

The internal standard area counts for pyrene-d10 were outside the control limits of 50-200% (high) in all sediment samples reported in this laboratory group, as well as the associated laboratory control sample, matrix spike, and matrix spike duplicate samples. Pyrene-d10 is associated with the labeled surrogate compound recoveries which were within control limits; therefore, data were not qualified based on these internal standard outliers.

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

The percent recoveries for the following analytes were outside the control limits in the LCS/LCSD extracted on July 6, 2018, and associated with the rinsate blank:

Analyte	LCS	LCSD	Control limits
2,4'-DDE	47%	50%	75-117%
4,4'-DDE	60%	69%	76-115%
4,4'-DDT	ok	117%	85-113%
cis-Nonachlor	213%	227%	59-138%

ok – acceptable

4,4'-DDT and cis-nonachlor were either not detected in the rinsate blank or 2 out of 3 quality control parameters (LCS, LCSD, and/or relative percent difference [RPD]) were acceptable; therefore, data were not qualified for these analytes based on these LCS/LCSD results. The results for 2,4'-DDE, and 4,4'-DDE in RB-VV-180703-1720 were qualified as estimated and flagged 'UJ' based on these LCS/LCSD results.

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

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-B463. The percent recoveries for 4,4'-DDT in the MS (15%) and the MSD (9%) were outside the control limits of 24-183%. The result for 4,4'-DDT in PDI-SG-B463 was qualified as estimated and flagged 'J' based on these MS/MSD results.

8. Reporting Limits – Acceptable except as noted below:

One or more results were flagged 'J' by the laboratory to indicate the reported concentrations were above the method detection limits (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 K1806305.

CONVENTIONAL ANALYSIS

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

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

A laboratory duplicate was performed using PDI-SG-B463. Results were comparable.

3. Reporting Limits – Acceptable



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OVERALL ASSESSMENT OF DATA

The data reported in this laboratory group is considered usable for meeting project objectives. The completeness for laboratory group K1806305 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-SG-B463	K1806305-006	CWA1699M	4,4'-DDT	4.9	ug/kg	4.9 J	m
RB-VV-180703-1720	K1806305-011	CWA1699M	2,4-DDE	0.51 U	ng/L	0.51 UJ	I
RB-VV-180703-1720	K1806305-011	CWA1699M	4,4'-DDE	0.51 U	ng/L	0.51 UJ	I

Notes:

- J - estimated value
- I - laboratory control sample/laboratory control sample duplicate recovery
- m - matrix spike recovery
- ng/L - nanogram per liter
- ug/kg - microgram per kilogram
- U - Compound was analyzed for, but not detected above the value shown.