

Data Validation Report

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

Laboratory: ALS Environmental, Burlington, Ontario, Canada

Laboratory Group: L2133758

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

Prepared by: Lucy Panteleeff/AECOM

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Reviewed by: George Kisluk/AECOM

File Name: L2133758 DVR

SUMMARY

The data quality review of 3 subsurface sediment samples collected on July 18, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/HRMS) and total solids by American Society for Testing and Materials (ASTM) Method D-2974 at ALS Environmental (ALS) located in Burlington, Ontario, Canada. The analyses were performed in general accordance with the methods specified in *Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS*, December 2007 (modified by ALS SOP BU-TM-1103 v07 OCP), 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 L2133758:

Sample ID	Laboratory ID
PDI-SC-S033-0TO2	L2133758-1
PDI-SC-S033-2TO3	L2133758-2
PDI-SC-S033-3TO4	L2133758-3

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 High Resolution Superfund Methods Data Review*, April 2016, *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 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.



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ORGANIC ANALYSIS

Samples were analyzed for chlorinated pesticides by EPA Method 1699.

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

The percent recoveries for the following analytes and labeled compounds were outside the control limits of 75-125% and 70-130%, respectively.

Analysis Date and Time	Analyte	% Recovery
August 9, 2018	4,4'-DDT-13C12	51%
August 10, 2018	2,4'-DDD	138%
	4,4'-DDD-13C12	47%
	4,4'-DDT-13C12	31%

The results associated with the continuing calibration verification (CCV) analyzed on August 9, 2018 were not reported by the laboratory; therefore, no data were qualified based on this CCV result.

The results for 2,4'-DDD, 4,4'-DDD, 2,4'-DDT and 4,4'-DDT in PDI-SC-S033-0TO2, PDI-SC-S033-2TO3, and PDI-SC-S033-3TO4 were qualified as estimated and flagged 'J' based on these CCV results.

3. Blanks – Acceptable
4. Labeled compounds – Acceptable
5. Internal Standards – Acceptable
6. Laboratory Control Sample (LCS) – Acceptable
7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

An MS/MSD was not performed in association with these samples. Accuracy was assessed using the LCS results. Precision was assessed using the laboratory duplicate results.

8. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S033-0TO2. Results were comparable.



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9. 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 EDLs but below the reporting limits. Laboratory 'J'-flagged results are considered estimated. As the result is between the EDL and the reporting limit, there is a greater level of uncertainty associated with the numerical result.

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 reporting limits do not exceeded the cleanup level.

Analytes with an ion abundance ratio outside the control limits of 1.56 +/- 25% were flagged 'R' by the laboratory indicating an 'EMPC' (estimated maximum possible concentration). These results were qualified as tentatively identified and flagged 'JN' based on this laboratory flag as identified in Table 1.

10. Other Items:

The laboratory noted PDI-SC-S033-3TO4 was re-analyzed at a dilution to confirm results in the initial analysis of the sample due to matrix interference. Results from the initial analysis of PDI-SC-S033-3TO4 were reported by the laboratory.

CONVENTIONAL ANALYSIS

Samples were analyzed for total solids by ASTM D-2974.

1. Holding Times – Acceptable except as noted below:

The sediment samples exceeded the 7-day holding time as indicated in the QAPP. No data qualifiers were assigned based on the holding time exceedance.

2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S033-0TO2. 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 L2133758 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Subsurface Sediment
ALS Burlington Laboratory Group: L2133758

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S033-0TO2	L2133758-1	E1699M	2,4-DDD	3.32	ng/g	3.32 J	c
PDI-SC-S033-0TO2	L2133758-1	E1699M	2,4-DDT	0.112 M,J	ng/g	0.112 J	c
PDI-SC-S033-0TO2	L2133758-1	E1699M	4,4'-DDD	8.51 M	ng/g	8.51 J	c
PDI-SC-S033-0TO2	L2133758-1	E1699M	4,4'-DDT	0.546 M	ng/g	0.546 J	c
PDI-SC-S033-2TO3	L2133758-2	E1699M	2,4-DDD	5.34	ng/g	5.34 J	c
PDI-SC-S033-2TO3	L2133758-2	E1699M	2,4-DDT	0.15 M,J,R	ng/g	0.15 JN	c,k
PDI-SC-S033-2TO3	L2133758-2	E1699M	4,4'-DDD	11.8 M	ng/g	11.8 J	c
PDI-SC-S033-2TO3	L2133758-2	E1699M	4,4'-DDT	1.08 M	ng/g	1.08 J	c
PDI-SC-S033-3TO4	L2133758-3	E1699M	2,4-DDD	13.0	ng/g	13.0 J	c
PDI-SC-S033-3TO4	L2133758-3	E1699M	2,4-DDT	1.47	ng/g	1.47 J	c
PDI-SC-S033-3TO4	L2133758-3	E1699M	4,4'-DDD	26.6	ng/g	26.6 J	c
PDI-SC-S033-3TO4	L2133758-3	E1699M	4,4'-DDT	10.0 M	ng/g	10.0 J	c

Notes:

- c - calibration issue
- J - estimated value
- JN - tentatively identified analyte
- k - Estimated Maximum Possible Concentration (EMPC)
- M - manual integration by laboratory
- ng/g - nanogram per gram
- R - Ion abundance outside acceptance criterion