

# 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: L2187236

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

Number: 60566335 Task #2.12

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File Name: L2187236 DVR

## SUMMARY

The data quality review of 2 subsurface sediment samples collected on July 24 and July 26, 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 L2187236:

Sample ID	Laboratory ID
PDI-SC-S154-4TO6	L2187236-01
PDI-SC-S185-5.5TO6.5	L2187236-02

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.

The samples were collected on July 24 and July 26, 2018, and were held frozen in the AECOM storage facility freezer until shipment to ALS-Burlington. The samples were received by the laboratory on October 25, 2018.

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## ORGANIC ANALYSIS

Samples were analyzed for chlorinated pesticides by EPA Method 1699.

1. Holding Times – Acceptable

The samples were extracted 81 days past the method-recommended holding time of 14 days. As described above, the samples were frozen in archive until shipment; therefore, the samples were not extracted outside the holding time.

2. Initial and Continuing Calibration Verifications – Acceptable

The percent recoveries for the following labeled compounds were outside the control limits of 70-130%.

Analysis Date and Time	Analyte	% Recovery
November 12, 2018 18:49	4,4'-DDT, 13C12	155%
November 12, 2018 22:30	4,4'-DDD, 13C12	137%
	4,4'-DDT, 13C12	176%

The results for 2,4'-DDD, 4,4'-DDD, 2,4'-DDT, and 4,4'-DDT in PDI-SC-S154-4TO6 and PDI-SC-S185-5.5TO6.5 were qualified as estimated and flagged 'UJ' based on these continuing calibration verification (CCV) results.

3. Blanks – Acceptable

4. Labeled compounds – Acceptable except as noted below:

The percent recoveries for 4,4'-DDT-13C12 exceeded the control limits of 5-120%.

Sample	Labeled Compound	Percent Recovery
PDI-SC-S154-4TO6	4,4'-DDT-13C12	132%
Laboratory Duplicate of PDI-SC-S154-4TO6	4,4'-DDT-13C12	142%
PDI-SC-S185-5.5TO6.5	4,4'-DDT-13C12	125%

Data were not qualified based on the labeled compound recovery in a QC sample (laboratory duplicate). The results for 2,4'-DDT and 4,4'-DDT in PDI-SC-S154-4TO6 and PDI-SC-S185-5.5TO6.5 were qualified as estimated based on CCV results as described in section 2; therefore, no further qualification based on labeled compound recoveries was required.

5. Internal Standards – Acceptable

6. Laboratory Control Sample (LCS) – Acceptable



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

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

8. Laboratory Duplicate

A laboratory duplicate was performed using PDI-SC-S154-4TO6. Results were comparable.

9. Field Duplicate

A field duplicate was not collected with this laboratory group. Precision was not assessed.

10. Reporting Limits – Acceptable except as noted below:

The reporting limits for the chlorinated pesticides reported as not detected in these samples were elevated due to dilution required for matrix interference. The reporting limits do not exceed the cleanup levels.

**CONVENTIONAL ANALYSIS**

The samples were analyzed for total solids by ASTM D-2974.

1. Holding Times – Acceptable

The 7-day holding time indicated in the QAPP was exceeded. Sample data was not qualified based on the holding time exceedance.

2. Laboratory Duplicate

A laboratory duplicate was performed using PDI-SC-S154-4TO6. Results were comparable.

3. Field Duplicate

A field duplicate was not collected with this laboratory group. Precision was not assessed.

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 L2187236 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Subsurface Sediment**  
**ALS Burlington Laboratory Group: L2187236**

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S154-4TO6	L2187236-1	E1699M	4,4'-DDT	0.23 U	ng/g	0.23 UJ	c
PDI-SC-S154-4TO6	L2187236-1	E1699M	2,4-DDT	0.12 U	ng/g	0.12 UJ	c
PDI-SC-S154-4TO6	L2187236-1	E1699M	4,4'-DDD	0.14 U	ng/g	0.14 UJ	c
PDI-SC-S154-4TO6	L2187236-1	E1699M	2,4-DDD	0.18 U	ng/g	0.18 UJ	c
PDI-SC-S185-5.5TO6.5	L2187236-2	E1699M	4,4'-DDT	0.27 U	ng/g	0.27 UJ	c
PDI-SC-S185-5.5TO6.5	L2187236-2	E1699M	2,4-DDD	0.21 U	ng/g	0.21 UJ	c
PDI-SC-S185-5.5TO6.5	L2187236-2	E1699M	4,4'-DDD	0.16 U	ng/g	0.16 UJ	c
PDI-SC-S185-5.5TO6.5	L2187236-2	E1699M	2,4-DDT	0.14 U	ng/g	0.14 UJ	c

**Notes:**

- c - calibration issue
- J - estimated value
- ng/g - nanogram per gram
- U - Compound was analyzed for, but not detected above the value shown.
- UJ - reported quantitation limit is approximate