

# Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Portland Harbor Superfund Site  
 Subsurface Sediment – Deep/Nearshore Cores

Laboratory: ALS Environmental, Burlington, Ontario, Canada

Laboratory Group: L2160292

Analyses/Method: Chlorinated Pesticides and Total Solids

Validation Level: Stage 2

AECOM Project

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File Name: L2160292 DVR

## SUMMARY

The data quality review of 13 subsurface sediment samples, one field duplicate, and one rinsate blank collected on September 5, 2018, has been completed. Samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/HRMS) and/or 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 L2160292:

Sample ID	Laboratory ID
PDI-SC-S129-0TO2	L2160292-1
PDI-SC-S129-2TO4	L2160292-2
PDI-SC-S129-4TO5.3	L2160292-3
PDI-SC-S155-0TO2.1	L2160292-4
PDI-SC-S155-2.1TO4.2	L2160292-5
PDI-SC-S155-4.2TO5.3	L2160292-6
PDI-SC-S121-0TO1.8	L2160292-7
PDI-SC-S121-1.8TO3.4	L2160292-8
PDI-SC-S255-0TO2.1	L2160292-9
PDI-SC-S255-0TO2.1D (Duplicate of PDI-SC-S255-0TO2.1)	L2160292-10
PDI-SC-S255-2.1TO4.3	L2160292-11
PDI-SC-S112-0TO2	L2160292-12
PDI-SC-S112-2TO4	L2160292-13
PDI-SC-S112-4TO6	L2160292-14
PDI-RB-SS-180905 (rinsate blank)	L2160292-15



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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.

**ORGANIC ANALYSIS**

Samples were analyzed for chlorinated pesticides by EPA Method 1699.

1. Holding Times – Acceptable
2. Initial and Continuing Calibration Verifications – Acceptable
3. Blanks – Acceptable
4. Labeled compounds – Acceptable except as noted below:

The percent recoveries for 4,4'-DDE-13C12 were below the control limits of 21-125% in the following samples.

Sample	Labeled Compound	Percent Recovery
PDI-SC-S121-0TO1.8	4,4'-DDE-13C12	19%
PDI-SC-S112-2TO4	4,4'-DDE-13C12	16%
PDI-SC-S112-4TO6	4,4'-DDE-13C12	8%

The results for 2,4'-DDE and 4,4'-DDE in the samples listed above were qualified as estimated and flagged 'J' or 'UJ' based on these labeled compound recoveries.

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

The percent recovery for 2,4'-DDT (122%) in the LCS extracted on September 14, 2018 exceeded the control limits of 50-120%. 2,4'-DDT was not detected in the associated sample; therefore, no qualification was required based on this elevated LCS result.



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

An MS/MSD was not performed in association with the rinsate blank. Accuracy was assessed using the LCS results. Precision was not assessed.

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

8. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S129-0TO2. Results greater than five times the reporting limits (RLs) were evaluated. Results were comparable.

9. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SC-S255-0TO2.1 and identified as PDI-SC-S255-0TO2.1D. Results greater than five times the RL were evaluated. Results were comparable.

10. 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 estimated detection limits (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 exceed 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). Results were qualified as tentatively identified and flagged 'JN' based on this laboratory flag as identified in Table 1.

11. Other Items:

Samples PDI-SC-S155-4.2TO5.3 and PDI-SC-S255-0TO2.1 were re-analyzed due to potential instrument carryover. Results from the re-analysis were reported.

**CONVENTIONAL ANALYSIS**

Sediment 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 indicated in the QAPP. No data qualifiers were assigned based on the holding time exceedance.



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2. Laboratory Duplicate – Acceptable

A laboratory duplicate was performed using PDI-SC-S129-0TO2. Results were comparable.

3. Field Duplicate – Acceptable

A field duplicate was submitted for PDI-SC-S255-0TO2.1 and identified as PDI-SC-S255-0TO2.1D. Results greater than five times the RL were evaluated. 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 L2160292 is 100%.

**Table 1**  
**QA/QC Data Summary Review**  
**Portland Harbor**  
**Subsurface Sediment**  
**ALS Burlington Laboratory Group: L2160292**

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
PDI-SC-S129-0TO2	L2160292-1	E1699M	2,4-DDD	0.44 M,J,R	ng/g	0.44 JN	k
PDI-SC-S129-0TO2	L2160292-1	E1699M	4,4'-DDT	0.18 M,J,R	ng/g	0.18 JN	k
PDI-SC-S129-2TO4	L2160292-2	E1699M	4,4'-DDT	0.27 M,J,R	ng/g	0.27 JN	k
PDI-SC-S129-4TO5.3	L2160292-3	E1699M	2,4-DDT	0.11 M,J,R	ng/g	0.11 JN	k
PDI-SC-S129-4TO5.3	L2160292-3	E1699M	4,4'-DDT	0.27 M,J,R	ng/g	0.27 JN	k
PDI-SC-S155-0TO2.1	L2160292-4	E1699M	2,4-DDT	0.22 M,J,R	ng/g	0.22 JN	k
PDI-SC-S121-0TO1.8	L2160292-7	E1699M	2,4'-DDE	0.056 U	ng/g	0.056 UJ	lc
PDI-SC-S121-0TO1.8	L2160292-7	E1699M	4,4'-DDE	0.104 M,J	ng/g	0.104 J	lc
PDI-SC-S255-0TO2.1	L2160292-9	E1699M	2,4-DDD	0.078 M,J,R	ng/g	0.078 JN	k
PDI-SC-S255-0TO2.1	L2160292-9	E1699M	4,4'-DDT	0.25 M,J,R	ng/g	0.25 JN	k
PDI-SC-S255-0TO2.1D	L2160292-10	E1699M	2,4-DDE	0.012 M,J,R	ng/g	0.012 JN	k
PDI-SC-S255-2.1TO4.3	L2160292-11	E1699M	2,4-DDE	0.042 M,J,R	ng/g	0.042 JN	k
PDI-SC-S255-2.1TO4.3	L2160292-11	E1699M	4,4'-DDT	0.094 M,J,R	ng/g	0.094 JN	k
PDI-SC-S112-2TO4	L2160292-13	E1699M	2,4-DDE	0.563 M,J	ng/g	0.563 J	lc
PDI-SC-S112-2TO4	L2160292-13	E1699M	4,4'-DDE	6.58 M	ng/g	6.58 J	lc
PDI-SC-S112-4TO6	L2160292-14	E1699M	2,4-DDE	0.970 M,J	ng/g	0.970 J	lc
PDI-SC-S112-4TO6	L2160292-14	E1699M	2,4-DDT	1.2 M,J,R	ng/g	1.2 JN	k
PDI-SC-S112-4TO6	L2160292-14	E1699M	4,4'-DDE	6.07 M	ng/g	6.07 J	lc

**Notes:**

- J - estimated value
- JN - tentatively identified analyte
- k - Estimated Maximum Possible Concentration (EMPC)
- lc - labeled compound recovery
- M - manual integration by laboratory
- ng/g - nanogram per gram
- ng/L -nanogram per liter
- R - Ion abundance outside acceptance criterion
- U - Compound was analyzed for, but not detected above the value shown.