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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling Portland Harbor Superfund Site Standard Reference Material
Laboratory:	ALS Environmental, Burlington, Ontario, Canada
Laboratory Groups:	L2125039 and L2159262
Analyses/Method:	Chlorinated Pesticides
Validation Level:	Stage 4
AECOM Project Number:	60566335 Task #2.12
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Completed on:	December 21, 2018
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File Name:	L2125039/L2159262 DVR

SUMMARY

The data quality review of three National Institute of Standards and Technology (NIST) standard reference materials (SRMs) has been completed. The samples were analyzed for chlorinated pesticides by EPA Method 1699-modified (GC/HRMS) at ALS Environmental (ALS) located in Burlington, Ontario, Canada. The analysis was performed in general accordance with the method 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). 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 groups L2125039 and L2159262:

Sample ID	Laboratory ID
NIST 1944	L2125039-01
NIST 1944 Duplicate	L2125039-02
NIST 1944	L2159262-01

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 and *USEPA National Functional Guidelines for Organic Superfund Methods Data Review*, January 2017. Data qualifiers assigned to this sample set are included in Table 1.

SAMPLE RECEIPT

The samples were received by ALS in good condition. The NIST SRMs were analyzed three times and reported in two laboratory groups.

Laboratory group L2125039 was analyzed on July 20, 2018. The results for 2,4'-DDD were quantified using the internal standard 4,4'-DDE-13C12 instead of the appropriate internal standard, 4,4'-DDD-13C12, due to issues with instrument drift. The results for 2,4'-DDD in the SRMs analyzed in laboratory group L2125039 were rejected and flagged 'R' based on this quantification issue.

Laboratory group L2159262 was analyzed on September 17, 2018 and no issues with QC or with calculations were found.

ORGANIC ANALYSIS

The 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 labeled compounds in the continuing calibration verification (CCV) analyzed on July 20, 2018 were outside the control limits of 70-130%.

Analyte	% Recovery
4,4'-DDT, 13C12	131%
4,4'-DDT, 13C12	69%

   2,4'-DDT was not reported for the samples in laboratory group L2125039. The results for 4,4'-DDT in NIST 1944 and NIST 1944 Duplicate for laboratory group L2125039 were qualified as estimated and flagged 'J' based on these CCV results.

3. Blanks – Acceptable except as noted below:

   4,4'-DDE (0.35 ng/g) was detected in the method blank extracted on July 6, 2018. The results for 4,4'-DDE were reported at concentrations significantly higher than the blank concentration; therefore, data were not qualified based on this method blank result.

4. Labeled compounds – Acceptable except as noted below:

   The percent recovery for 4,4'-DDT-13C12 (132%) exceeded the control limits of 47-160% in NIST 1944 associated with laboratory group L2125039. The result for 4,4'-DDT in NIST 1944 was qualified as estimated based on CCV results as described in Section 2; therefore, no further qualification based on the labeled compound recovery was required.

5. Internal Standards – Acceptable

6. Laboratory Control Sample (LCS) – Acceptable

7. Laboratory Duplicate

   A laboratory duplicate was performed using NIST 1944 associated with laboratory group L2125039 and was presented as a second analytical sample. Results were comparable.

8. Reporting Limits – Acceptable

OVERALL ASSESSMENT OF DATA

The data reported in these laboratory groups is considered usable for meeting project objectives. The completeness for laboratory group L2125039 is 80% and L2159262 is 100%.

Table 1
QA/QC Data Summary Review
Portland Harbor
Standard Reference Material
ALS Burlington Laboratory Group: L2125039/L2159262

Sample ID	Laboratory ID	Method	Analyte	Laboratory Result	Units	Final Result	Reason Code
NIST 1944 (A)	L2125039-1	E1699M	4,4'-DDT	189	ng/g	189 J	c
NIST 1944 (A)	L2125039-1	E1699M	2,4'-DDD	77.8	ng/g	R	q
NIST 1944 (B)	L2125039-2	E1699M	4,4'-DDT	199	ng/g	199 J	c
NIST 1944 (B)	L2125039-2	E1699M	2,4'-DDD	57.8	ng/g	R	q

Notes:
c - calibration issue
J - estimated value
ng/g - nanogram per gram
q - quantitation issue
R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.