

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	SGS-AXYS, Sydney, British Columbia, Canada	
Laboratory Group:	WG65521-PEST_2	
Analyses/Method:	Pesticides by HRGC/HRMS / EPA Method 1699	
Validation Level:	Stage 4	
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SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 20-25, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-XD-180820	Equipment blank
PDI-WS-T01-1808	Surface Water
PDI-WS-T02-1808	Surface Water
PDI-WS-T03-1808	Surface Water
PDI-WS-T04-1808	Surface Water
PDI-WS-T05-1808	Surface Water
PDI-WS-T06-1808	Surface Water
PDI-WS-T07-1808	Surface Water

Data validation activities were conducted with reference to:

- EPA Method 1699: *Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (December 2007) ,
- *Quality Assurance Project Plan, Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling, Portland Harbor Superfund Site (March 2018)*, and the
- laboratory quality control (QC) limits.

The National Functional Guidelines were modified to accommodate the non-CLP methodologies. In the absence of method-specific information, laboratory QC limits, project-specific requirements and/or AECOM professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

- ✓ Data completeness (chain-of-custody (COC)/sample integrity)
- ✓ Holding times and sample preservation
- ✓ Mass and Chromatographic Resolution/DDT Breakdown Results
- ✓ Initial calibration/continuing calibration verification
- ✗ Laboratory blanks/equipment blanks
- NA Matrix spike (MS) and/or matrix spike duplicate (MSD) results
- ✓ Ongoing precision and recovery (OPR) results
- NA Field duplicate results
- ✓ Labeled compound recoveries
- ✗ Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An NA indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as qualified and may be used for decision making purposes. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below). Qualified sample results are presented in Table 1.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with method criteria. All method QC acceptance criteria were met.

Mass and Chromatographic Resolution/DDT Breakdown Results

The data were reviewed to ensure that

- the perfluorokerosene (PFK) molecular leak was performed at the correct frequency (at the beginning and end of a 12-hour shift) and the mass resolution was at a resolving power of > 8,000;
- the separation between 4,4'-DDD and 2,4'-DDT must be $\leq 35\%$ of the valley height, and
- the 4,4'-DDT breakdown must be $\leq 15\%$.

All method QC acceptance criteria were met.

Initial Calibration/Continuing Calibration Verification

The data were reviewed to ensure that

- the ion abundance ratio method acceptance criteria were met;
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for all native and labeled compounds;
- and the calibration verification standard (VER) method acceptance criteria were met.

All method QC acceptance criteria were met.

Laboratory Blanks/Equipment Blanks

Method and equipment rinsate blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL). Target compounds were detected in the method blank and equipment blank associated with the samples in this data set.

Compounds detected in the laboratory method blank and the equipment blank are summarized in Attachment A in Tables A-1 and A-2, respectively. It should be noted that significant contamination was found in the equipment blank associated with the samples in this data set. Consequently, the sample data were qualified on the basis of the equipment blank contamination as well as the laboratory method blank contamination.

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of data and the reporting of false negative results should be avoided. Therefore, in order to avoid the reporting of false negative results, professional judgment was used to qualify the data in the manner summarized below.

The data were first qualified for laboratory method blank contamination on the following basis. As allowed in the NFG, a blank action limit (BAL) was determined as five times the method blank result.

- When the sample results were < the method blank result, the sample result was qualified as nondetect (U) at the sample result.
- When the sample result was \geq the method blank result but \leq the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was > the BAL, the sample result was not qualified.

Qualified sample results are summarized in Table 1.

The data were subsequently qualified for equipment blank contamination on the following basis. Again, as allowed in the NFG, a blank action limit (BAL) was determined as five times the equipment blank result.

- When the sample result was \leq the BAL, the sample result was qualified as estimated and potentially biased high (J+).
- When the sample result was $>$ the BAL, the sample result was not qualified.

Qualified sample results are summarized in Table 1.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

OPR Results

The OPR %Rs were reviewed for conformance with the method QC acceptance criteria.

Field Duplicate Results

A field duplicate pair was not submitted with this data set. No data validation actions were taken on this basis.

Labeled Compound Recoveries

The labeled compound %Rs were reviewed for conformance with the QC acceptance criteria. All method QC acceptance criteria were met.

The laboratory does not spike the XAD resin with a field spike prior to deployment to the field. No data validation actions were taken on this basis.

Sample Results/Reporting Issues

All sample results detected at concentrations less than the lowest calibration standard but greater than the EDL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

It should be noted that the sample reported detection limit is the sample specific estimated detection limit (EDL) with the following exceptions. In cases where the EDL is less than the nominal concentration of 0.2 ng for hexachlorobenzene or 0.4 ng for the remaining compounds, then the EDL is raised to these nominal concentrations and are adjusted to include the appropriate preparation factors

Compound Identification

The data were reviewed to ensure that

- the relative retention time, ion abundance ratios, SIM ion co-maximization, and S/N method acceptance criteria were met for compound identification.

Samples were qualified as follows:

Actions: (Based on NFG 2016 and AECOM professional judgment)

Criteria	Actions
A native target compound was reported by the laboratory as an EMPC.	Report result as an EMPC and qualify as estimated and presumptively present (JN).
A labeled compound was flagged by the laboratory indicating all identification criteria were not met.	Qualify associated positive and nondetect results as estimated (J/UJ).

It should be noted that in instances of multiple nonconformances, the bias is considered indeterminate in cases where a conflicting low and high bias exists or when a result does not exhibit a consistent bias. These results have an overall qualification of estimated (J) with the exception noted below.

When applicable, the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where EMPC results were qualified for multiple quality control nonconformances. Qualified sample results are shown in Table 1.

Lock Mass Interferences

The positive and nondetect results for the following compounds for the listed samples were qualified as estimated and potentially biased low (J-/UJ) as a result of ion suppression as indicated by the monitored lock mass:

4,4'-DDT: PDI-RB-XD-180820

alpha-chlordane: PDI-WS-T07-1808

oxychlordane: PDI-WS-T02-1808, PDI-WS-T03-1808, PDI-WS-T04-1808, PDI-WS-T05-1808, PDI-WS-T06-1808, PDI-WS-T07-1808

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. All actions are described above.

ATTACHMENTS

Attachment A: Nonconformance Summary Tables

Attachment B: Qualifier Codes and Explanations

Attachment C: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	EDL	Units	Validation Qualifiers	Validation Reason
PDI-RB-XD-180820	WQ	4,4'-DDE	0.164	0.0620	ng/sample	J+	bl
PDI-RB-XD-180820	WQ	4,4'-DDT	0.465	0.338	ng/sample	JN	k,su
PDI-RB-XD-180820	WQ	Hexachlorobenzene	0.274	0.0263	ng/sample	J+	bl
PDI-RB-XD-180820	WQ	trans-Nonachlor	0.322	0.0525	ng/sample	JN	k
PDI-WS-T01-1808	WS	4,4'-DDT	0.518	0.100	ng/sample	J+	be
PDI-WS-T01-1808	WS	alpha-Chlordane	1.17	0.0528	ng/sample	J+	be
PDI-WS-T01-1808	WS	Oxychlordane	0.231	0.158	ng/sample	JN	k
PDI-WS-T01-1808	WS	trans-Chlordane	1.07	0.0528	ng/sample	J+	be
PDI-WS-T01-1808	WS	trans-Nonachlor	0.965	0.0528	ng/sample	J+	be
PDI-WS-T02-1808	WS	4,4'-DDT	0.772	0.0678	ng/sample	J+	be
PDI-WS-T02-1808	WS	alpha-Chlordane	1.29	0.0534	ng/sample	J+	be
PDI-WS-T02-1808	WS	Oxychlordane		0.256	ng/sample	UJ	su
PDI-WS-T02-1808	WS	trans-Chlordane	1.09	0.0534	ng/sample	J+	be
PDI-WS-T02-1808	WS	trans-Nonachlor	1.14	0.0534	ng/sample	J+	be
PDI-WS-T03-1808	WS	4,4'-DDT	1.82	0.0941	ng/sample	J+	be
PDI-WS-T03-1808	WS	cis-Nonachlor	0.692	0.0534	ng/sample	JN	k
PDI-WS-T03-1808	WS	Oxychlordane		0.247	ng/sample	UJ	su
PDI-WS-T03-1808	WS	trans-Chlordane	1.69	0.0534	ng/sample	J+	be
PDI-WS-T04-1808	WS	Aldrin	0.576	0.0902	ng/sample	JN	k
PDI-WS-T04-1808	WS	Oxychlordane		0.342	ng/sample	UJ	su
PDI-WS-T04-1808	WS	trans-Chlordane	2.23	0.0532	ng/sample	J+	be
PDI-WS-T05-1808	WS	2,4-DDT	0.149	0.0949	ng/sample	JN	k
PDI-WS-T05-1808	WS	4,4'-DDT	0.255	0.0997	ng/sample	J+	be
PDI-WS-T05-1808	WS	alpha-Chlordane	1.79	0.0523	ng/sample	J+	be
PDI-WS-T05-1808	WS	Oxychlordane		0.275	ng/sample	UJ	su
PDI-WS-T05-1808	WS	trans-Chlordane	1.57	0.0523	ng/sample	J+	be
PDI-WS-T05-1808	WS	trans-Nonachlor	1.43	0.0523	ng/sample	J+	be
PDI-WS-T06-1808	WS	4,4'-DDT	0.397	0.103	ng/sample	J+	be
PDI-WS-T06-1808	WS	alpha-Chlordane	1.89	0.0531	ng/sample	J+	be
PDI-WS-T06-1808	WS	Oxychlordane	0.268	0.201	ng/sample	JN	k,su
PDI-WS-T06-1808	WS	trans-Chlordane	1.43	0.0531	ng/sample	J+	be
PDI-WS-T07-1808	WS	4,4'-DDT	0.457	0.0740	ng/sample	J+	be
PDI-WS-T07-1808	WS	alpha-Chlordane	1.61	0.0525	ng/sample	J	be,su
PDI-WS-T07-1808	WS	Oxychlordane		0.338	ng/sample	UJ	su
PDI-WS-T07-1808	WS	trans-Chlordane	1.26	0.0525	ng/sample	J+	be

Attachment A

Nonconformance Summary Tables

Table A-1 Laboratory Blank

Blank ID	Compound	Result	RL	Units	BAL	Associated Samples
WG65521-101	Hexachlorobenzene	0.085	0.0265	ng/sample	0.425	PDI-RB-XD-180820 PDI-WS-T01-1808 PDI-WS-T02-1808 PDI-WS-T03-1808 PDI-WS-T04-1808 PDI-WS-T05-1808 PDI-WS-T06-1808 PDI-WS-T07-1808
	4,4'-DDE	0.089	0.0630	ng/sample	0.445	

Table A-2 Field Blank

Sample ID	Compound	Result	RL	Units	BAL	Associated Samples
PDI-RB-XD-180820	Hexachlorobenzene	0.274	0.0263	ng/sample	1.37	PDI-WS-T01-1808 PDI-WS-T02-1808 PDI-WS-T03-1808 PDI-WS-T04-1808 PDI-WS-T05-1808 PDI-WS-T06-1808 PDI-WS-T07-1808
	4,4'-DDT	0.465	0.338	ng/sample	2.32	
	alpha-Chlordane	0.417	0.0525	ng/sample	2.08	
	trans-Chlordane	0.887	0.0525	ng/sample	4.44	
	trans-Nonachlor	0.322	0.0525	ng/sample	1.61	
	2,4-DDD	0.184	0.140	ng/sample	0.920	
	4,4'-DDD	0.512	0.174	ng/sample	2.56	
	4,4'-DDE	0.164	0.0620	ng/sample	0.820	

Attachment B**Qualifier Codes and Explanations**

Qualifier	Explanation
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential low bias.
J+	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential high bias.
JN	The analyte was tentatively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
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.

Attachment C

Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
bf	Field blank contamination
bl	Laboratory blank contamination
c	Calibration issue
cl	Clean-up standard recovery
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
h	Holding times
i	Internal standard areas
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
lc	Labeled compound recovery
ld	Laboratory duplicate RPDs
lp	Laboratory control sample/laboratory control sample duplicate RPDs
m	Matrix spike recovery
md	Matrix spike/matrix spike duplicate RPDs
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results