

# Data Validation Report

Project: Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling  
 Laboratory: SGS-AXYS, Sydney, British Columbia, Canada  
 Laboratory Group: WG65583-PEST\_3  
 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-XF-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),
- USEPA Contract Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016),
- 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. The equipment blank contamination was comparable to the laboratory method blank contamination. Consequently, the sample data were qualified only on the basis of 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.

#### **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 percent recoveries (%Rs) were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

### **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 with the following exception.

The laboratory noted that approximately 1/3 of the extract for the laboratory duplicate sample PDI-WS-T03-1808 (Duplicate) was lost when transferring to the micro vial. As a result, most of the labeled compound recoveries in this sample fell below the QC acceptance limits. However, since the sample results are quantitated using isotope dilution technique, the data are not adversely impacted by these nonconformances. Data were not qualified 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.

### **Laboratory Duplicate Analysis**

The laboratory was unable to extract the entire number of filters received for each sample due to limitations of their Dean Stark apparatus. Approximately 1/5<sup>th</sup> of each homogenized original filter sample was spiked with labeled standards and extracted rather than the entire amount that was collected. Consequently, a laboratory duplicate analysis was performed to ensure that the results achieved were representative of the entire sample.

Professional judgement was applied to use a relative percent difference criterion of <20% for results greater than five times the quantitation limit.

Nonconformances are summarized in Attachment A in Table A-3. Samples were qualified as follows:

**Actions:** (Based on AECOM professional judgment)

Criteria	RPD	Action	
		Detect	Nondetect
Sample and duplicate are nondetect results	Not calculable (NC)	No qualification	No qualification
Sample and duplicate results <QL	Not applicable	No qualification	No qualification
Sample and duplicate results $\geq 5 \times \text{QL}$	>20%	J	Not Applicable
Sample and duplicate results are >QL and <5xQL	>40%	J	Not Applicable
If sample or duplicate result is >5xQL and the other is not detected	NC	J	UJ
If sample or duplicate result is <QL and the other is not detected	NC	No qualification	No qualification

Qualified sample results are shown in Table 1.

#### 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 nondetect results for the following compound for the listed samples were qualified as estimated (UJ) as a result of ion suppression as indicated by the monitored lock mass:

oxychlordan: All samples

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-XF-180820	WQ	Hexachlorobenzene	0.851	0.137	ng/sample	J+	bl
PDI-RB-XF-180820	WQ	Oxychlorane		0.275	ng/sample	UJ	su
PDI-WS-T01-1808	WS	2,4-DDT	0.876	0.338	ng/sample	JN	k
PDI-WS-T01-1808	WS	cis-Nonachlor	0.368	0.261	ng/sample	JN	k
PDI-WS-T01-1808	WS	Hexachlorobenzene	1.58	0.131	ng/sample	J+	bl
PDI-WS-T01-1808	WS	Oxychlorane		0.261	ng/sample	UJ	su
PDI-WS-T02-1808	WS	Hexachlorobenzene	1.59	0.129	ng/sample	J+	bl
PDI-WS-T02-1808	WS	Oxychlorane		0.259	ng/sample	UJ	su
PDI-WS-T03-1808	WS	4,4'-DDT	8.11	0.431	ng/sample	J	ld
PDI-WS-T03-1808	WS	cis-Nonachlor	0.672	0.268	ng/sample	JN	k
PDI-WS-T03-1808	WS	Hexachlorobenzene	1.90	0.134	ng/sample	J+	bl
PDI-WS-T03-1808	WS	Oxychlorane		0.268	ng/sample	UJ	su
PDI-WS-T04-1808	WS	Hexachlorobenzene	2.15	0.130	ng/sample	J+	bl
PDI-WS-T04-1808	WS	Oxychlorane		0.261	ng/sample	UJ	su
PDI-WS-T05-1808	WS	2,4-DDT	0.714	0.259	ng/sample	JN	k
PDI-WS-T05-1808	WS	Hexachlorobenzene	1.59	0.129	ng/sample	J+	bl
PDI-WS-T05-1808	WS	Oxychlorane		0.259	ng/sample	UJ	su
PDI-WS-T06-1808	WS	2,4-DDT	0.679	0.313	ng/sample	JN	k
PDI-WS-T06-1808	WS	cis-Nonachlor	0.443	0.265	ng/sample	JN	k
PDI-WS-T06-1808	WS	Hexachlorobenzene	1.45	0.133	ng/sample	J+	bl
PDI-WS-T06-1808	WS	Oxychlorane		0.265	ng/sample	UJ	su
PDI-WS-T06-1808	WS	trans-Nonachlor	0.902	0.265	ng/sample	JN	k
PDI-WS-T07-1808	WS	2,4-DDT	0.578	0.398	ng/sample	JN	k
PDI-WS-T07-1808	WS	cis-Nonachlor	0.448	0.257	ng/sample	JN	k
PDI-WS-T07-1808	WS	Hexachlorobenzene	1.32	0.129	ng/sample	J+	bl
PDI-WS-T07-1808	WS	Oxychlorane		0.257	ng/sample	UJ	su

## Attachment A

## Nonconformance Summary Tables

Table A-1 - Laboratory Blanks

Blank ID	Compound	Result	RL	BAL	Units	Associated Samples
WG65583-101	Hexachlorobenzene	0.568	0.132	2.84	ng/sample	PDI-RB-XF-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

Table A-2 - Field Blanks

Blank ID	Compound	Result	RL	BAL	Units	Associated Samples
PDI-RB-XF-180820	Hexachlorobenzene	0.814	0.131	4.07	ng/sample	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

Table A-3 – Laboratory Duplicate

Compound	RL	5xRL	PDI-WS-T03-1808 (pg/sample)	Lab duplicate (pg/sample)	RPD
4,4'-DDT	13.5	67.5	8.11	18.8	79.5

**Attachment B****Qualifier Codes and Explanations**

<b>Qualifier</b>	<b>Explanation</b>
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