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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS-AXYS, Sydney, British Columbia, Canada
Laboratory Group:	WG67275-PEST
Analyses/Method:	Pesticides by HRGC/HRMS / EPA Method 1699
Validation Level:	Stage 2A
AECOM Project Number:	60566335.2.12
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SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on January 26-27, 2019 and February 17-18, 2019.

Sample ID	Matrix/Sample Type
PDI-RB-XF-190127	Equipment Blank
PDI-WS-T01-1902	Surface Water
PDI-WS-T02-1902	Surface Water
PDI-WS-T03-1902	Surface Water
PDI-WS-T04-1902	Surface Water
PDI-WS-T05-1902	Surface Water
PDI-WS-T06-1901	Surface Water
PDI-WS-T07-1901	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
✗	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 or negated 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.

Laboratory Blanks/Equipment Blanks

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

Compounds detected in the laboratory method blank are summarized in Attachment A in Table A-1. There were no compounds detected in the equipment blank PDI-RB-XF-190127 after method blank actions (as described below) were applied.

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 following manner on the basis of laboratory method blank contamination. As allowed in the NFG, a blank action limit (BAL) was determined as five times the blank result:

- When the sample results were < the blank result, the sample result was qualified as nondetect (U) at the sample result.
- When the sample result was $\geq$ the blank result and $\leq$ 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. 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.

Nonconformances are summarized in Attachment A in Table A-2. Samples were qualified as follows:

Actions: (Based on NFG 2016)

Criteria[1]	Actions[2]	
	Detected	Nondetected
%R > Upper Acceptance Limit	J	UJ
%R >10% but < Lower Acceptance Limit	J	UJ
%R <10%	See below	
<10% and S/N >10:1	J	R
<10% and S/N <10:1	R	R

Criteria[1]		Actions[2]	
		Detected	Nondetected
Ion abundance ratio criteria not met	Calibration compliant	J	UJ
	Calibration non-compliant	J	R
Clean-up Standard Recovery < Lower Acceptance Limit		J	UJ

[1]See Table 7 in method 1613B for acceptance criteria

[2]The dioxin method is performed using isotope dilution technique; therefore, professional judgment was applied and bias codes were not included in data qualification.

Qualified sample results are summarized in Table 1.

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 exception. In cases where the EDL is less than the nominal concentration of 0.04 ng, then the EDL is raised to this nominal concentration and is adjusted to include the appropriate preparation factors.

Lock Mass Interferences

The laboratory identified the presence of interferences of the mass ion as indicated by the monitored lock mass by qualifying the affected sample result with a "G" laboratory qualifier. These interferences may impact compound quantitation; therefore, the positive and nondetect results for affected samples were qualified as estimated (J/UJ).

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 1/5th 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. All QC acceptance criteria were met.

Estimated Maximum Possible Concentrations (EMPCs)

The data were reviewed to identify sample results that were indicated by the laboratory to be estimated maximum possible concentrations (EMPCs) because of identification criteria not being met.

The laboratory identified the presence of EMPCs for the samples in this data set by qualifying affected results with a “K” laboratory qualifier. Samples were qualified as follows:

Actions: (Based on 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 where there is a conflicting low and high bias 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 summarized in Table 1.

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-190127	WQ	cis-Nonachlor		0.415	ng/sample	U	bl
PDI-RB-XF-190127	WQ	Hexachlorobenzene		0.146	ng/sample	U	bl
PDI-RB-XF-190127	WQ	trans-Nonachlor		0.303	ng/sample	U	bl
PDI-WS-T01-1902	WS	4,4'-DDT	53.5	1.67	ng/sample	J	lc
PDI-WS-T01-1902	WS	Aldrin	0.438	0.302	ng/sample	JN	k
PDI-WS-T01-1902	WS	alpha-Chlordane	2.76	0.302	ng/sample	JN	k
PDI-WS-T01-1902	WS	cis-Nonachlor	1.26	0.302	ng/sample	JN	bl,k
PDI-WS-T01-1902	WS	Oxychlordane	0.479	0.396	ng/sample	JN	k
PDI-WS-T01-1902	WS	trans-Chlordane	3.26	0.302	ng/sample	JN	k
PDI-WS-T02-1902	WS	cis-Nonachlor	1.66	0.292	ng/sample	J+	bl
PDI-WS-T02-1902	WS	Oxychlordane	0.436	0.323	ng/sample	JN	k
PDI-WS-T03-1902	WS	Aldrin	0.492	0.301	ng/sample	JN	k
PDI-WS-T03-1902	WS	cis-Nonachlor	2.06	0.301	ng/sample	JN	bl,k
PDI-WS-T03-1902	WS	Oxychlordane		0.301	ng/sample	UJ	q
PDI-WS-T03-1902	WS	trans-Chlordane	3.92	0.301	ng/sample	JN	k
PDI-WS-T03-1902	WS	trans-Nonachlor	4.79	0.301	ng/sample	JN	k
PDI-WS-T04-1902	WS	Aldrin	0.330	0.284	ng/sample	JN	k
PDI-WS-T04-1902	WS	cis-Nonachlor	1.26	0.284	ng/sample	JN	bl,k
PDI-WS-T04-1902	WS	trans-Chlordane	2.29	0.284	ng/sample	JN	k
PDI-WS-T04-1902	WS	trans-Nonachlor	2.62	0.284	ng/sample	JN	k
PDI-WS-T05-1902	WS	cis-Nonachlor	2.13	0.301	ng/sample	J+	bl
PDI-WS-T05-1902	WS	trans-Chlordane	2.60	0.301	ng/sample	JN	k
PDI-WS-T05-1902	WS	trans-Nonachlor	2.79	0.301	ng/sample	JN	k
PDI-WS-T06-1901	WS	alpha-Chlordane	1.38	0.286	ng/sample	JN	k
PDI-WS-T06-1901	WS	cis-Nonachlor	0.722	0.286	ng/sample	JN	bl,k
PDI-WS-T06-1901	WS	trans-Nonachlor	1.58	0.286	ng/sample	JN	bl,k
PDI-WS-T07-1901	WS	cis-Nonachlor	1.75	0.295	ng/sample	J+	bl
PDI-WS-T07-1901	WS	Oxychlordane		0.295	ng/sample	UJ	q
PDI-WS-T07-1901	WS	trans-Nonachlor	3.09	0.295	ng/sample	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 - Lab Blanks

Blank ID	Compound	Result	QL	BAL	Units	Associated Samples
WG67275-101	Hexachlorobenzene	0.276	14.7	1.38	ng/sample	PDI-RB-XF-190127 PDI-WS-T01-1902 PDI-WS-T02-1902 PDI-WS-T03-1902 PDI-WS-T04-1902 PDI-WS-T05-1902 PDI-WS-T06-1901 PDI-WS-T07-1901
	trans-Nonachlor	0.372	25.0	1.86	ng/sample	
	cis-Nonachlor	0.568	29.3	2.84	ng/sample	

Table A-2 - Labeled Compound Recoveries

Sample ID	Labeled Compound	% Recovery	Lower Limit	Upper Limit
PDI-WS-T01-1902	13C-4,4'-DDT	39.2	40	150

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