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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS AXYS Analytical Services Ltd, Sidney, BC, Canada
Laboratory Group:	WG65124-PEST
Analyses/Method:	Organochlorine Pesticides by HRGC/HRMS/E1699
Validation Level:	Stage 4
AECOM Project Number:	60566335.2.12
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File Name:	WG65124-PEST DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 13, 14, and 15, 2018.

Sample ID	Matrix/Sample Type
PDI-TF-SMB063	Fish Tissue
PDI-TF-SMB073	Fish Tissue
PDI-TF-SMB114	Fish Tissue
PDI-TF-SMB115	Fish Tissue
PDI-TF-SMB116	Fish Tissue
PDI-TF-SMB118	Fish Tissue
PDI-TF-SMB121	Fish Tissue
PDI-TF-SMB122	Fish Tissue
PDI-TF-SMB123	Fish Tissue
PDI-TF-SMB124	Fish Tissue
PDI-TF-SMB125	Fish Tissue
PDI-TF-SMB126	Fish Tissue
PDI-TF-SMB127	Fish Tissue
PDI-TF-SMB131	Fish Tissue
PDI-TF-SMB134	Fish Tissue
PDI-TF-SMB135	Fish Tissue

Data validation activities were conducted with reference to:

- *EPA Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (USEPA, 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 standard operating procedure (SOP) and 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 resolution/ chromatographic resolution/ column breakdown check results
✓	Initial calibration/continuing calibration verification
✓	Laboratory blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✗	Ongoing precision and recovery (OPR) and certified reference material (CRM) results
✓	Matrix duplicate (MD) results
✓	Labeled compound and clean-up standard 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 Resolution/ Chromatographic Resolution/ Column Breakdown Check 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 chromatographic resolution check was performed at the correct frequency between 4,4'-DDD and 2,4'-DDT and at a resolution of ≤ 35%;
- the column breakdown check was performed at the correct frequency for 4,4'-DDT and a breakdown of ≤15%.

All method QC acceptance criteria were met.

Initial Calibration/Continuing Calibration Verification

The data were reviewed to ensure that:

- the absolute and relative retention time, signal/noise (S/N), and ion abundance ratio method acceptance criteria were met (as summarized by the laboratory);
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for all native and labeled compounds;
- The calibration verification standard (CCV) method acceptance criteria were met.

It should be noted that according to the laboratory's SOP, the following method modifications were noted:

- Alternate mass ions were used for cis + trans-Chlordane, cis + trans-Nonachlor, and corresponding ^{13}C-labelled standards. In addition, primary mass ion and secondary mass ion ratios (m1:m2, per Method 1699) were reversed (i.e., m2:m1) for these compounds/standards. The theoretical ratio criterion for these compounds were evaluated accordingly.
- The m1:m2 ratio for dieldrin and ^{13}C-dieldrin (per Method 1699) was reversed (i.e., m2:m1).

Method 1699 states: *"Other quantitation references and procedures may be used provided that the results produced are as accurate as results produced by the quantitation references and procedures described in Section 10.4".* The above referenced modifications were applied to all sample and QC analyses. Therefore, no data validation actions were taken on this basis.

Laboratory Blanks

Laboratory method blanks results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL).

Target compounds were detected in the method blanks associated with the samples in this data set. Note, the laboratory does not qualify sample results "B" associated with method blank contamination. Since the sample concentrations were greater than 5 times the blank results, no data validation actions were taken on this basis.

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 and CRM Results

The OPR and CRM %Rs and/or RPDs were reviewed for conformance with the method/reference QC acceptance criteria.

Nonconformances are summarized in Attachment A in Table A-1. 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
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 5 in method 1699 for acceptance criteria

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

Note, the extract for the CRM went to dryness prior to florisil cleanup. This may have affected the recovery of DDT in the CRM only (isolated instance). Since the CRM ^{13}C-labelled standards and the associated OPR recoveries were within QC limits, the CRM outliers did not warrant rejection of the sample data. Instead, the DDT results were conditionally qualified. Qualified sample results are summarized in Table A-1.

MD Results

MD RPDs were reviewed for conformance with the laboratory QC acceptance criteria of $\leq$ 40% [if one or both results were greater than five times the quantitation limit (QL)] for solid matrices. All method QC acceptance criteria were met.

Labeled Compound and Clean-up Standard Recoveries

The labeled compounds and labeled clean-up standard %Rs were reviewed for conformance with the QC acceptance criteria. All method QC acceptance criteria were met.

As noted in the laboratory's case narrative, the laboratory inadvertently double-spike samples PDI-TF-SMB121 and PDI-TF-SMB134 with ^{13}C-labelled recovery standard. The appropriate factor was applied to the surrogate recoveries by the laboratory to account for the spiking variance. This variance does not affect pesticide quantification for these samples. 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 or sample detection limit (SDL) are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

As stated in the laboratory's case narrative, the following reporting issue was noted:

> *To account for instrument variability and lab background levels, it is SGS AXYS policy to report detection limits no lower than Minimum Reporting Limits described in MSU-028. The reported limit (RL) for each target analyte is the greater of the SDL and the minimum reporting limit in MSU-028.*

This is also noted in the laboratory's SOP.

PFK Lock Mass

The laboratory confirmed that a lock mass is monitored for each instrument descriptor. All QC

Compound Identification

The data were reviewed to ensure that:

- the retention time, relative retention time, ion abundance ratios, SIM ion co-maximization, and S/N method acceptance criteria were met for compound identification; and
- the quantitative determination of pesticides were not affected by the presence of matrix interferences detected above the 3:1 S/N ratio limit.

All QC acceptance criteria were met with the following exceptions. Sample results which don't meet all of the method stipulated qualitative identification criteria are considered to be Estimated Maximum Possible Concentrations (EMPCs). Details concerning sample results in this data set which did not meet these identification criteria are noted below along with any data qualifications, as applicable.

The laboratory qualified all EMPC sample results with a "K" laboratory qualifier to indicate that the ion ratio criterion was not met. All ion ratios were verified and affected sample results which did not meet the ion ratio criteria were qualified as estimated and tentatively identified (JN). Qualified sample results are shown in Table 1.

It should be noted that the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where sample results were qualified for multiple quality control nonconformances.

Percent Solids Content

Since the sample matrix was fish tissue, all sample results have been reported on a “wet weight” basis.

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

Dilutions

Sample ID	Compound	Dilution Factor
PDI-TF-SMD118	4,4’-DDE	2.5
PDI-TF-SMD121	4,4’-DDE	2.5

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	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-TF-SMB063	TF	2,4-DDT	0.649	0.0083	0.0060	ug/kg	J	l
PDI-TF-SMB063	TF	4,4'-DDT	3.97	0.0098	0.0062	ug/kg	J	l
PDI-TF-SMB063	TF	cis-Nonachlor	1.44	0.0079	0.0183	ug/kg	J	l
PDI-TF-SMB073	TF	2,4-DDT	0.334	0.0193	0.0062	ug/kg	J	l
PDI-TF-SMB073	TF	4,4'-DDT	3.36	0.0235	0.0064	ug/kg	J	l
PDI-TF-SMB073	TF	Aldrin	0.007	0.0041	0.0088	ug/kg	JN	k
PDI-TF-SMB073	TF	cis-Nonachlor	2.94	0.0135	0.0191	ug/kg	J	l
PDI-TF-SMB114	TF	2,4-DDT	0.130	0.0267	0.0067	ug/kg	J	l
PDI-TF-SMB114	TF	4,4'-DDT	1.96	0.0323	0.0069	ug/kg	J	l
PDI-TF-SMB114	TF	Aldrin	0.006	0.0045	0.0095	ug/kg	JN	k
PDI-TF-SMB114	TF	cis-Nonachlor	1.26	0.0045	0.0205	ug/kg	J	l
PDI-TF-SMB115	TF	2,4-DDT	0.201	0.0151	0.0065	ug/kg	JN	k,l
PDI-TF-SMB115	TF	4,4'-DDT	1.39	0.0201	0.0067	ug/kg	J	l
PDI-TF-SMB115	TF	cis-Nonachlor	1.19	0.0051	0.0199	ug/kg	J	l
PDI-TF-SMB116	TF	2,4-DDT	0.067	0.0067	0.0064	ug/kg	J	l
PDI-TF-SMB116	TF	4,4'-DDT	0.911	0.0075	0.0067	ug/kg	J	l
PDI-TF-SMB116	TF	cis-Nonachlor	0.821	0.0043	0.0198	ug/kg	J	l
PDI-TF-SMB118	TF	2,4-DDT	0.242	0.0081	0.0072	ug/kg	J	l
PDI-TF-SMB118	TF	4,4'-DDT	4.01	0.0093	0.0074	ug/kg	J	l
PDI-TF-SMB118	TF	Aldrin	0.011	0.0048	0.0102	ug/kg	JN	k
PDI-TF-SMB118	TF	cis-Nonachlor	5.87	0.0143	0.0221	ug/kg	J	l
PDI-TF-SMB121	TF	2,4-DDT	0.487	0.0122	0.0066	ug/kg	J	l
PDI-TF-SMB121	TF	4,4'-DDT	4.83	0.0144	0.0068	ug/kg	J	l
PDI-TF-SMB121	TF	cis-Nonachlor	2.72	0.0090	0.0202	ug/kg	J	l
PDI-TF-SMB122	TF	2,4-DDT	0.162	0.0076	0.0061	ug/kg	J	l
PDI-TF-SMB122	TF	4,4'-DDT	1.55	0.0093	0.0063	ug/kg	J	l
PDI-TF-SMB122	TF	cis-Nonachlor	0.786	0.0041	0.0187	ug/kg	J	l
PDI-TF-SMB123	TF	2,4-DDT	0.205	0.0097	0.0066	ug/kg	J	l
PDI-TF-SMB123	TF	4,4'-DDT	1.29	0.0111	0.0068	ug/kg	J	l
PDI-TF-SMB123	TF	cis-Nonachlor	0.817	0.0044	0.0201	ug/kg	J	l
PDI-TF-SMB124	TF	2,4-DDT	0.204	0.0152	0.0066	ug/kg	J	l
PDI-TF-SMB124	TF	4,4'-DDT	2.05	0.0184	0.0068	ug/kg	J	l
PDI-TF-SMB124	TF	cis-Nonachlor	0.823	0.0044	0.0202	ug/kg	J	l
PDI-TF-SMB125	TF	2,4-DDT	0.079	0.0222	0.0063	ug/kg	J	l
PDI-TF-SMB125	TF	4,4'-DDT	0.921	0.0269	0.0065	ug/kg	J	l
PDI-TF-SMB125	TF	cis-Nonachlor	0.439	0.0042	0.0192	ug/kg	J	l
PDI-TF-SMB126	TF	2,4-DDT	0.312	0.0119	0.0065	ug/kg	J	l
PDI-TF-SMB126	TF	4,4'-DDT	4.08	0.0143	0.0068	ug/kg	J	l

Sample ID	Matrix	Compound	Result	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-TF-SMB126	TF	cis-Nonachlor	2.10	0.0069	0.0201	ug/kg	J	l
PDI-TF-SMB127	TF	2,4-DDT	0.104	0.0084	0.0071	ug/kg	J	l
PDI-TF-SMB127	TF	4,4'-DDT	1.67	0.0096	0.0074	ug/kg	J	l
PDI-TF-SMB127	TF	cis-Nonachlor	0.884	0.0047	0.0218	ug/kg	J	l
PDI-TF-SMB131	TF	2,4-DDT	0.389	0.0126	0.0074	ug/kg	J	l
PDI-TF-SMB131	TF	4,4'-DDT	2.66	0.0158	0.0077	ug/kg	J	l
PDI-TF-SMB131	TF	cis-Nonachlor	2.04	0.0075	0.0227	ug/kg	J	l
PDI-TF-SMB134	TF	2,4-DDT	0.147	0.0096	0.0073	ug/kg	J	l
PDI-TF-SMB134	TF	4,4'-DDT	1.64	0.0109	0.0075	ug/kg	J	l
PDI-TF-SMB134	TF	cis-Nonachlor	0.787	0.0051	0.0224	ug/kg	J	l
PDI-TF-SMB135	TF	2,4-DDT	0.232	0.0101	0.0067	ug/kg	J	l
PDI-TF-SMB135	TF	4,4'-DDT	3.17	0.0124	0.0069	ug/kg	J	l
PDI-TF-SMB135	TF	cis-Nonachlor	1.21	0.0065	0.0205	ug/kg	J	l

Attachment A

Nonconformance Summary Tables

Table A-1 – Certified Reference Material Recoveries

Compound	% Recovery	Lower Limit	Upper Limit
cis-Nonachlor	135	77	123
2,4'-DDT	0	70	130
4,4'-DDT	0	70	130
^{13}C-2,4'-DDT	118	40	150
^{13}C-4,4'-DDT	114	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
ma	Multiple analyses, sample analyzed more than once, a value from another analysis should be used
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
rt	SIM ions not within + 2 seconds or not within relative retention time (RRT) window
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results