AECOM
257 West Genesee Street, Suite 400
Buffalo, NY 14202

716.856.5636 tel
716.856.2545 fax

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS AXYS Sidney,BC,Canada
Service Request:	WG65146
Analyses/Method:	Organochlorine Pesticides by HRGC/HRMS / E1699
Validation Level:	Stage 2A
AECOM Project Number:	60566335.2.12
Prepared by:	Ann Marie Kropovitch/AECOM
Completed on:	12/10/2018
Reviewed by:	George Kisluk/AECOM
File Name:	WG65146-Pest DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 16-18, 2018.

Sample ID	Matrix/Sample Type
PDI-TF-SMB046	Fish Tissue
PDI-TF-SMB049	Fish Tissue
PDI-TF-SMB052	Fish Tissue
PDI-TF-SMB057	Fish Tissue
PDI-TF-SMB067	Fish Tissue
PDI-TF-SMB075	Fish Tissue
PDI-TF-SMB077	Fish Tissue
PDI-TF-SMB079	Fish Tissue
PDI-TF-SMB085	Fish Tissue
PDI-TF-SMB086	Fish Tissue
PDI-TF-SMB087	Fish Tissue
PDI-TF-SMB089	Fish Tissue
PDI-TF-SMB090	Fish Tissue
PDI-TF-SMB091	Fish Tissue
PDI-TF-SMB092	Fish Tissue
PDI-TF-SMB093	Fish Tissue
PDI-TF-SMB099	Fish Tissue
PDI-TF-SMB101	Fish Tissue
PDI-TF-SMB103	Fish Tissue
PDI-TF-SMB105	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
✓	Laboratory blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Ongoing precision and recovery results
NA	Field duplicate results
✗	Labeled compounds and labeled 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 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

Method blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL).

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of pesticides due to the toxicity of these compounds 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. As allowed in the NFG, a blank action limit (BAL) was determined as 5 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 ≥ the method blank result and ≤ the BAL, the sample result was qualified as estimated and potentially biased high (J+).

When the sample result was > the BAL, sample result was not qualified.

All sample results were > the BAL, therefore no sample results were qualified.

Ongoing Precision and Recovery

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

Field Duplicate Results

A field duplicate was not submitted for this sample delivery group (SDG).

Labeled Compounds and Labeled Clean-up Standard Recoveries

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

The percent recoveries fell outside of the QC acceptance limits for the labeled compounds listed for the following sample:

PDI-TF-SMB090

Nonconformances are summarized in Attachment A in Table A-1. Samples were qualified as follows:

Actions: (Based on NFG 2016)

Criteria	Actions	
	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
See Table 6 of method for method QC acceptance criteria[1] [2]The PCB congener 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.

Sample Results/Reporting Issues

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

The laboratory qualified the sample results with a "KJ" to indicate that the pesticide peak was detected but did not meet quantitication criteria; the result should be considered as an Estimated Maximum Possible Concentration (EMPC). These results were qualified as estimated and tentatively identified (JN). Qualified sample results are summarized in Table 1.

It should be noted that the "JN" qualifier was retained rather than replacement with the conventional overall "J", “J+”, and “J-“ qualifiers 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.

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-TF-SMB049	TA	Aldrin	0.017	0.0046	ug/kg	JN	k
PDI-TF-SMB075	TA	Aldrin	0.018	0.0046	ug/kg	JN	k
PDI-TF-SMB079	TA	Aldrin	0.013	0.0046	ug/kg	JN	k
PDI-TF-SMB087	TA	Aldrin	0.011	0.0046	ug/kg	JN	k
PDI-TF-SMB089	TA	Aldrin	0.014	0.0046	ug/kg	JN	k
PDI-TF-SMB090	TA	2,4-DDT	0.363	0.0158	ug/kg	J	lc
PDI-TF-SMB090	TA	4,4'-DDD	3.81	0.0140	ug/kg	J	lc
PDI-TF-SMB090	TA	4,4'-DDE	31.3	0.0085	ug/kg	J	lc
PDI-TF-SMB090	TA	4,4'-DDT	2.59	0.0176	ug/kg	J	lc
PDI-TF-SMB091	TA	Aldrin	0.007	0.0046	ug/kg	JN	k
PDI-TF-SMB093	TA	Aldrin	0.008	0.0046	ug/kg	JN	k
PDI-TF-SMB099	TA	Aldrin	0.010	0.0046	ug/kg	JN	k

Attachment A

Table A-1 - Labeled Compound and Labeled Clean-Up Standard Recoveries

Sample ID	Compound	% Recovery	Lower Limit	Upper Limit
PDI-TF-SMB090	4,4'-DDE	162	40	150
	4,4'-DDD	177	40	150
	2,4'-DDT	165	40	150
	4,4'-DDT	174	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
ma	Multiple analyses. Sample analyzed more than once, a value from another analysis should be used.
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