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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:	WG65907-PCB
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A
Validation Level:	Stage 2A
AECOM Project Number:	60566335.2.12
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Reviewed by:	George Kisluk/AECOM	File Name: WG65907-PCB DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on August 22, 2018.

Sample ID	Matrix/Sample Type
PDI-TF-SMB053	Fish Tissue

Data validation activities were conducted with reference to:

- *EPA Method 1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS* (USEPA, August 2003),
- *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
- NA   Matrix spike (MS) and/or matrix spike duplicate (MSD) results
- ✓   Ongoing precision and recovery results

NA   Matrix duplicate (MD) 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.

As noted in the data validation report associated with laboratory group WG65252-PCB, a laboratory accident during initial extraction of sample PDI-TF-SMB053. Hence, this sample required re-extraction/re-analysis and the PCB results are reported in this data validation report.

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). Target compounds were detected in the laboratory method blank associated with the sample in this data set. Note, the laboratory does not qualify sample results "B" associated with method blank contamination. All PCB congener results in the sample were greater than the blank action limit (BAL) of 5 times the method blank results. No data validation actions were taken on this basis.

MS/MSD Results

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

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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.

MD Duplicate Results

The MD not performed on a sample in this data set. No data validation actions were taken on this basis.

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. All method QC acceptance criteria were met.

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.

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

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection limit qualifier…..To account for lab background levels and variability in instrument response, detection limits are reported no lower than 0.5 pg absolute (i.e., 0.073 pg/g on a 10g sample size and 1.45 extract splitting factor).

Compound Quantitation and Identification

Samples exhibiting lock-mass ion interference are qualified with a "G" by the laboratory. Professional judgement was used to qualify affected results "J" or "UJ".

Qualified sample results are summarized in Table 1.

Estimated Maximum Possible Concentrations (EMPCs)

The data were reviewed to identify sample results that were indicated by the laboratory to be EMPCs because of identification criteria not being met.

The laboratory qualified all 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" and "J+" qualifiers in instances where sample results were qualified for multiple quality control nonconformances.

<u>Percent Solids Content</u>

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.

<u>Dilutions</u>

Sample ID	Congener	Dilution Factor
PDI-TF-SMB053	PCB-118, PCB-170 to PCB-209	10

Note, the undiluted analysis of sample PDI-TF-SMB053 exhibited matrix interference for PCB-186 and PCB-192. The results for these two congeners were reported from the DF10 analysis by the laboratory as non-detect (U), hence, the RDLs are elevated. Using professional judgement, the non-detect results were qualified as estimated (UJ).

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: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

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Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-TF-SMB053	TF	PCB-126	33.3	20.9	0.332	pg/g	JN	k
PDI-TF-SMB053	TF	PCB-127	20.8	19.9	0.358	pg/g	JN	k
PDI-TF-SMB053	TF	PCB-184	18.1	0.489	3.32	pg/g	JN	k
PDI-TF-SMB053	TF	PCB-186		0.541	3.32	pg/g	UJ	d
PDI-TF-SMB053	TF	PCB-192		0.602	4.24	pg/g	UJ	d
PDI-TF-SMB053	TF	PCB-204	2.29	0.492	3.32	pg/g	JN	k
PDI-TF-SMB053	TF	PCB-66	13600	6.02	0.332	pg/g	J	v
PDI-TF-SMB053	TF	PCB-86/87/97/108/119/125	6660	1.62	3.18	pg/g	J	v

Attachment A

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 B

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
cr	Chromatographic resolution
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
q	Quantitation issue
r	Dual column RPD
rt	SIM ions not within + 2 seconds, or not within relative retention time (RRT) windowQuantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
v	Compound identification issue
x	Percent solids
y	Serial dilution results
z	ICS results