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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
Service Request:	WG65907
Analyses/Method	Polybrominated Diphenylether (PBDE) by EPA Method 1614A - High Resolution Gas Chromatography (HRGC) High Resolution Mass Spectrometry (HRMS)
Validation Level:	Stage 2A
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Reviewed by:	File Name: WG65907_BDE_DVR

SUMMARY

The sample listed below was collected by AECOM in Portland Harbor in Portland, OR on August 22-24, 2018.

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

Data validation activities were conducted with reference to:

- EPA Method 1614A: Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS (USEPA, May 2010),
- USEPA Contract Laboratory Program (CLP) National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016),
- Quality Assurance Project Plan (QAPP), Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling, Portland Harbor Superfund Site (March 2018), and
- Laboratory Standard Operating Procedure

The National Functional Guidelines (NFG) were modified to accommodate the non-CLP methodologies. In the absence of method-specific information, laboratory quality control (QC) limits, project-specific requirements and/or Geosyntec 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
✓	Laboratory Duplicate
✓	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 following issues were noted with the COC, however they did not result in sample qualification.

  Incorrect error correction was observed on the COC, instead of a single strike-through dated and initialed by the person making the correction.

  The client sample identifications (IDs) listed on the COC did not agree with client sample IDs listed in the laboratory report. In an email correspondence, included in the report, the

client requested the laboratory correct the sample IDs from those listed on the COC to those listed in the laboratory report to match the sample IDs in the QAPP.

- 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.

  It was noted that several client samples were listed on the COC and received and logged in by the laboratory but the analytical results were not included in this 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 blanks are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL).

Target compounds were detected in the method blank associated with the samples in this data set.
Note, the laboratory does not qualify sample results "B" associated with method blank contamination.

The NFG guidance for high resolution methods stipulates that a conservative approach should be taken with regard to qualification of chlorinated biphenyl congeners due to the toxicity of these compounds and the reporting of false negative results should be avoided. Therefore, based on professional judgment a conservative approach to avoid reporting false negatives was taken for the brominated diphenyl congeners. As allowed in the NFG, a blank action limit (BAL) was determined as 5 times the method blank result and the data were assessed as described below:

- When the sample result was < the method blank result, the sample result was qualified as non-detect (U) at the sample result.
- When the sample result was $\geq$ the method blank result $\leq$ 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.

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.

Ongoing Precision and Recovery (OPR)

The OPR percent recoveries (%Rs) and/or relative percent differences (RPDs) were reviewed for conformance with the method QC acceptance criteria. All method QC acceptance criteria were met.

Laboratory Duplicate Results

A laboratory duplicate was 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.

Estimated Maximum Possible Concentrations (EMPCs)

The laboratory qualified the sample results with a "k" to indicate that the PBDE peak was detected but did not meet quantification criteria, these results should be considered Estimated Maximum Possible Concentrations (EMPCs). 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 replaced with the conventional overall "J", "J+", and "J-" qualifiers in instances where sample 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: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

Table 1 – Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	RDL	QL	Units	Validation Qualifier	Validation Reason
PDI-TF-SMB053	TF	PBDE-12/13	11.3	0.133	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-126	20.2	7.7	13.3	pg/g	J+	bl
PDI-TF-SMB053	TF	PBDE-128	7.25	1.67	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-138/166	0.997	0.539	13.3	pg/g	JN	k,bl
PDI-TF-SMB053	TF	PBDE-140	8.68	0.332	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-181	0.883	0.228	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-203	2.27	0.215	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-207	3.02	1.23	13.3	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-208	14.7	1.43	13.3	pg/g	JN	k,bl
PDI-TF-SMB053	TF	PBDE-35	10.6	4.58	15.9	pg/g	JN	k
PDI-TF-SMB053	TF	PBDE-79	73.2	0.133	13.3	pg/g	JN	k,bl

TF – tissue, fish
pg/g –picograms per gram

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
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