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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	SGS AXYS, Sydney, British Columbia Canada
Service Request:	WG65436
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A (SGS AYXS Method MLA-10)
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 September 10th and 11th, 2018.

Sample ID	Matrix/Sample Type
PDI-TF-SMB064	Fish Tissue
PDI-TF-SMB013	Fish Tissue
PDI-TF-SMB061	Fish Tissue
PDI-TF-SMB004	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),
- EPA Method 1668B: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (USEPA, November 2008),
- EPA Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (USEPA, April 2010),
- *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
	Duplicate
✓	Ongoing precision and recovery results
NA	Field duplicate results
✓	Labeled compounds and labeled clean-up standard recoveries
✗	Laboratory Duplicate
✗	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. The samples were shipped on dry ice and then stored at -20°C in the dark prior to extraction and analysis. The method QC acceptance criteria were met.

Laboratory Blanks/Equipment Blanks

Method and equipment rinsate 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. An equipment blank was not submitted with this data set.

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of PCB congeners 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 $\geq$ the method 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, sample result was not qualified.

Qualified sample results are summarized in Table 1. Method blank detected compounds are summarized in Attachment A, Table A-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. The method QC acceptance criteria were met.

Field Duplicate Results

A field duplicate was not submitted with this data set.

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 method QC acceptance criteria were met.

Laboratory Duplicate Analysis

A laboratory duplicate was performed on sample PDI-TF-SMB004.

Professional judgement was applied to use a relative percent difference criterion of <40% for results greater than five times the quantitation limit. The laboratory duplicate results satisfied the evaluation criterion.

Sample Results/Reporting Issues

The 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.

Compound Identification

The data were reviewed to evaluate whether

- the retention time, relative retention time (RRT), ion abundance ratios, SIM ion co-maximization, and signal to noise (S/N) ratio method acceptance criteria were met for compound identification.

Samples were qualified as follows:

Actions: (Based on NFG 2016 guidance and AECOM professional judgment)

Criteria	Actions
RRT falls outside of method limits and RT falls outside of window defining mix windows	If there is no peak, consider the analyte as nondetect (U) at the reported EDL for World Health Organization (WHO) Toxic Congeners. Non-WHO Toxic congeners are considered ND at the PQL.[2]
RRT falls outside of method limits and RT falls outside of window defining mix windows	Qualify associated positive results as estimated (J).[1]
S/N criteria not met	Consider the analyte as nondetect (U) at the reported EDL for WHO Toxic Congeners.[2]
Ion co-maximization and/or ion abundance ratios are outside of QC limits for a PCB congener	Report result as an EMPC and qualify as estimated (JN).[1]
Ion co-maximization and/or ion abundance ratios are outside QC limits for a Labeled compound	Qualify associated positive and nondetect results as estimated (J/UJ).[1]
[1]Based on AECOM professional judgment. [2]Based on NFG 2016 guidance.	

Qualified sample results are summarized in Table 1.

The RRTs for the PCB-79 and/or PCB-36 for samples PDI-TF-SMB064, PDI-TF-SMB013, and PDI-TF-SMB004 were slightly outside the RRT controls limits. However, the compounds are determined to present based on the comparison of chromatographic patterns between the samples and calibration data; results were qualified "JN" based on RRT exceedances. Nonconformances are summarized in Attachment A, Table A-2.

The laboratory qualified the sample results with a "K" to indicate that the ion abundance ratio was outside of the QC acceptance limits; 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.

The laboratory reported co-eluting results as one result. For example, PCB 12 and 13 for sample PDI-TF-SM064 is reported singularly as PCB-12 at a concentration of 5.19 pg/g with a laboratory flag of C on the data sheet. PCB 13 is reported with just a laboratory flag of C12 on the data sheet, and in the data validation assistant (DVA) is reported as PCB 12/13 at a concentration of 5.19 pg/g with a laboratory flag of C.

Lipids

The percent lipids data were reviewed. The PCB results were reported on a wet basis and not adjusted for percent lipids; an approach consistent with Method 1668A. Data were not qualified on the basis of percent lipids.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. The 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-SMB004	TA	PCB-123	364	48.6	pg/g	JN	k
PDI-TF-SMB004	TA	PCB-204	1.45	0.297	pg/g	JN	k
PDI-TF-SMB004	TA	PCB-36	533	11.2	pg/g	JN	q
PDI-TF-SMB004	TA	PCB-79	114	38.8	pg/g	JN	q
PDI-TF-SMB013	TA	PCB-123	97.5	15.0	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-126	20.2	18.5	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-145	2.06	0.412	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-2	1.07	0.401	pg/g	J+	bl
PDI-TF-SMB013	TA	PCB-204	1.26	0.279	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-24	4.05	0.228	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-3	2.92	0.371	pg/g	J+	bl
PDI-TF-SMB013	TA	PCB-7	3.44	2.68	pg/g	JN	k
PDI-TF-SMB013	TA	PCB-79	49.2	11.6	pg/g	JN	q
PDI-TF-SMB013	TA	PCB-9	5.11	2.65	pg/g	JN	k
PDI-TF-SMB061	TA	PCB-123	129	33.4	pg/g	JN	k
PDI-TF-SMB061	TA	PCB-126	54.3	38.2	pg/g	JN	k
PDI-TF-SMB061	TA	PCB-145	3.85	0.471	pg/g	JN	k
PDI-TF-SMB061	TA	PCB-79	84.2	7.42	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-123	204	22.8	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-126	44.6	24.8	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-35	2.38	1.91	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-38	3.38	1.92	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-57	12.3	3.95	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-7	7.70	3.67	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-79	87.6	3.41	pg/g	JN	q
PDI-TF-SMB064	TA	PCB-81	7.78	3.89	pg/g	JN	k
PDI-TF-SMB064	TA	PCB-9	9.84	3.64	pg/g	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 – Laboratory Blanks

Blank ID	Compound	Result	QL	Unit	BAL	Associated Samples
WG65436-101	PCB-1	0.348	2.91	pg/g	1.74	PDI-TF-SMB064 PDI-TF-SMB013 PDI-TF-SMB061 PDI-TF-SMB004
	PCB-105	1.11	2.91	pg/g	5.55	
	PCB-110/115	2.46	2.91	pg/g	12.3	
	PCB-118	2.73	2.91	pg/g	13.65	
	PCB-128/166	0.625	2.91	pg/g	3.125	
	PCB-129/138/160/163	2.62	2.91	pg/g	13.1	
	PCB-135/151/154	0.740	2.91	pg/g	3.7	
	PCB-146	0.715	2.91	pg/g	3.575	
	PCB-147/149	0.926	2.91	pg/g	4.63	
	PCB-153/168	2.92	2.91	pg/g	14.6	
	PCB-17	0.410	2.91	pg/g	2.05	
	PCB-170	0.387	2.91	pg/g	1.935	
	PCB-18/30	1.24	2.91	pg/g	6.2	
	PCB-180/193	1.60	2.91	pg/g	8	
	PCB-183/185	0.454	2.91	pg/g	2.27	
	PCB-187	1.58	2.91	pg/g	7.9	
	PCB-196	0.403	2.91	pg/g	2.015	
	PCB-2	0.239	2.91	pg/g	1.195	
	PCB-20/28	1.98	2.91	pg/g	9.9	
	PCB-203	0.407	2.91	pg/g	2.035	
	PCB-21/33	0.727	2.91	pg/g	3.635	
	PCB-3	0.671	2.91	pg/g	3.355	
	PCB-31	1.16	2.91	pg/g	5.8	
	PCB-32	0.576	2.91	pg/g	2.88	
	PCB-40/41/71	0.949	2.91	pg/g	4.745	
	PCB-42	0.494	2.91	pg/g	2.47	
	PCB-44/47/65	3.26	2.91	pg/g	16.3	
	PCB-49/69	1.19	2.91	pg/g	5.95	
	PCB-52	2.40	2.91	pg/g	12	
	PCB-56	0.647	2.91	pg/g	3.235	
	PCB-60	0.569	2.91	pg/g	2.845	
	PCB-61/70/74/76	3.03	2.91	pg/g	15.15	
	PCB-64	0.975	2.91	pg/g	4.875	
	PCB-66	2.20	2.91	pg/g	11	
	PCB-83/99	2.01	2.91	pg/g	10.05	
	PCB-84	0.549	2.91	pg/g	2.745	
	PCB-85/116/117	1.05	2.91	pg/g	5.25	
	PCB-86/87/97/108/119/125	1.82	2.91	pg/g	9.1	
	PCB-90/101/113	2.40	2.91	pg/g	12	
	PCB-93/95/98/100/102	1.31	2.91	pg/g	6.55	

Table A-2– Relative Retention Time Exceedances

Sample ID	PCB Congener	RRT	Lower Limit	Upper Limit
PDI-TF-SMB064	PCB-79	0.968	0.969	0.972
PDI-TF-SMB013	PCB-79	0.968	0.969	0.976
PDI-TF-SMB004	PCB-36	0.935	0.930	0.934
	PCB-79	0.968	0.969	0.972

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