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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, Knoxville, Tennessee	
Service Request:	580-77187-3	
Analyses/Method:	Chlorinated Biphenyls by HRGC/HRMS / E1668A	
Validation Level:	Stage 4	
AECOM Project Number:	60566335.2.12	
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Reviewed by:	Elissa McDonagh/AECOM	File Name: 580-77187-3 DVR

SUMMARY

The sample listed below was received by TestAmerica on May 10, 2018.

Sample ID	Matrix/Sample Type
Puget Sound Sediment Reference Material	Performance Evaluation

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
- ✓ GC/MS performance checks
- ✓ Initial calibration/continuing calibration verification
- ✓ Laboratory blanks/equipment 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 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 laboratory noted that when the Puget Sound Sediment Reference Material was received at the Tacoma facility it arrived at a temperature of 25°C. No data validation actions were taken on this basis due to the persistence of PCBs.

GC/MS Performance Checks

The data were reviewed to ensure that

- the perfluorokerosene (PFK) molecular leak was performed at the correct frequency and method acceptance criteria were met;
- the method acceptance criteria were met in the Diluted combined 209 congener standard for the chromatographic resolution on the SPB-octyl column of the congener pairs PCB-34 and PCB-23, and PCB-187 and PCB-182;
- the method acceptance criteria were met in the Diluted combined 209 congener standard for the co-elution of the congener pair PCB-156 and PCB-157 within 2 seconds of the peak maximum on the SPB-octyl column; and

- the retention time for decachlorobiphenyl (PCB 209) was greater than 55 minutes as required by the method.

All method QC acceptance criteria were met.

As stipulated in the laboratory's SOP, the laboratory may use FC43 rather than PFK for monitoring the mass resolution. In cases where FC43 is used, the selected reference peaks do cover the mass range of the descriptors and all mass resolution 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 for all native toxics/level of chlorination (LOC) congeners and labeled toxics/LOC/window-defining congeners (as summarized by the laboratory);
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for all native toxic/LOC congeners, and labeled toxics/LOC/window-defining congeners; and that performance was technically acceptable in the absence of method criteria for additional congeners in the standards; and
- the calibration verification standard (VER) method acceptance criteria were met for all native toxic/LOC congeners, and labeled toxics/LOC/window-defining congeners, and that performance was technically acceptable in the absence of method criteria for additional congeners in the standards.

All 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). An equipment blank was not submitted in this data set.

Target compounds were detected in the method blank associated with the samples in this data set. Detected compounds are summarized in Attachment A in Table A-1.

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 blank result:

- When the sample results were < the blank result, the sample result was qualified as nondetect (U) at the sample result.
- When the sample result was $\geq$ the 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, the sample result was not qualified.

Qualification of the data was not required.

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

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

Field Duplicate Results

A field duplicate pair was not submitted with 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

During data validation, it was discovered that the ion ratio QC limits entered into the laboratory's CHROM data system were incorrect for PCB-5 and PCB-159. Additionally, it was discovered that the CHROM data system did not always provide the area for one of the two ions when manually assigned by the analyst and this resulted in the ion ratio being reported as 0. A database query was performed by the laboratory to determine which results were impacted by these errors. The laboratory updated the CHROM data system to correct for these issues and affected samples were reprocessed. For samples analyzed after the discovery of these issues, all lab reports will indicate the correct QC limits for the ion ratios for PCB 5 and PCB 159. As an additional precaution, the laboratory continues to monitor the sample results in order to ensure all peak areas are being provided by the CHROM data system and the incidence of missing area results no longer exists.

It should be noted, that sample or standard results were not reprocessed for the following instances since the sample concentration or final reported result were not impacted.

- The PCB congener detected in a sample was determined to be found at a concentration that was less than the EDL. Consequently, the result is reported as not detected.
- The PCB congener was calculated and reported correctly in spite of the incorrect QC limit noted in the CHROM data system.

For the scenarios listed above, the ion ratio QC limits reported in the laboratory report will not reflect the corrected change to the CHROM data system; however, all sample results have been reported correctly.

All sample results detected at concentrations less than the lowest calibration standard 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 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.

Samples were qualified as follows:

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

Criteria[1]	Actions[2]
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 WHO Toxics congeners. Non-WHO Toxic congeners are considered ND at the ML.
S/N criteria not met	Consider the analyte as nondetect (U) at the reported EDL for WHO Toxics congeners
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.	

All QC acceptance criteria were met with the following exceptions. As described in the table above, 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 sample results with a "q" 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.

Percent Solids Content

The percent solids data were reviewed since the amount of moisture in a solid sample may have an impact on data representativeness. Due to the extremely low solubility of PCB congeners in water, these analytes should be contained in the solid phase. Consequently, the NFG guidance does not stipulate a percent solids criterion. If applicable, EPA Regional guidance is used when assessing percent solids content. In the absence of EPA Regional guidance, AECOM uses 30% solids (from the NFG semivolatile guidance) as a benchmark to evaluate the percent solids content and professional judgment is used to determine the necessity to qualify data. Qualification on this basis was not required.

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

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
Puget Sound Sediment Reference Material	SE	PCB-24	0.0091	0.00089	ng/g	JN	k
Puget Sound Sediment Reference Material	SE	PCB-46	0.055	0.0054	ng/g	JN	k
Puget Sound Sediment Reference Material	SE	PCB-5	0.0051	0.0011	ng/g	JN	k
Puget Sound Sediment Reference Material	SE	PCB-58	0.0092	0.0029	ng/g	JN	k
Puget Sound Sediment Reference Material	SE	PCB-81	0.0037	0.0028	ng/g	JN	k

Attachment A

Nonconformance Summary Tables

Table A-1 – Lab Blanks

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
MB 140-20383/13-B	PCB-101	0.000331	0.000041	ng/g	0.001655	Puget Sound Sediment Reference Material
	PCB-105	0.000449	0.00015	ng/g	0.002245	
	PCB-107	0.000225	0.00016	ng/g	0.001125	
	PCB-109	0.000205	0.000040	ng/g	0.001025	
	PCB-11	0.00197	0.00046	ng/g	0.00985	
	PCB-113	0.000331	0.000041	ng/g	0.001655	
	PCB-116	0.000142	0.000038	ng/g	0.00071	
	PCB-117	0.000142	0.000038	ng/g	0.00071	
	PCB-118	0.000323	0.00015	ng/g	0.001615	
	PCB-119	0.000205	0.000040	ng/g	0.001025	
	PCB-121	0.000406	0.000033	ng/g	0.00203	
	PCB-125	0.000205	0.000040	ng/g	0.001025	
	PCB-129	0.00100	0.00011	ng/g	0.005	
	PCB-138	0.00100	0.00011	ng/g	0.005	
	PCB-147	0.000515	0.00012	ng/g	0.002575	
	PCB-149	0.000515	0.00012	ng/g	0.002575	
	PCB-153	0.000399	0.000094	ng/g	0.001995	
	PCB-156	0.000420	0.00011	ng/g	0.0021	
	PCB-157	0.000420	0.00011	ng/g	0.0021	
	PCB-160	0.00100	0.00011	ng/g	0.005	
	PCB-163	0.00100	0.00011	ng/g	0.005	
	PCB-168	0.000399	0.000094	ng/g	0.001995	
	PCB-17	0.000260	0.000054	ng/g	0.0013	
	PCB-170	0.000146	0.000041	ng/g	0.00073	
	PCB-18	0.000397	0.000048	ng/g	0.001985	
	PCB-183	0.000581	0.000036	ng/g	0.002905	
	PCB-185	0.000581	0.000036	ng/g	0.002905	
	PCB-186	0.000143	0.000029	ng/g	0.000715	
	PCB-189	0.000254	0.000086	ng/g	0.00127	
	PCB-194	0.000108	0.000050	ng/g	0.00054	
	PCB-20	0.000907	0.00032	ng/g	0.004535	
	PCB-205	0.0000796	0.000038	ng/g	0.000398	
	PCB-21	0.000660	0.00030	ng/g	0.0033	
	PCB-26	0.000461	0.00032	ng/g	0.002305	
	PCB-28	0.000907	0.00032	ng/g	0.004535	

Blank ID	Compound	Result	ML	Units	BAL	Associated Samples
	PCB-29	0.000461	0.00032	ng/g	0.002305	
	PCB-3	0.000760	0.00024	ng/g	0.0038	
	PCB-30	0.000397	0.000048	ng/g	0.001985	
	PCB-31	0.000809	0.00029	ng/g	0.004045	
	PCB-33	0.000660	0.00030	ng/g	0.0033	
	PCB-44	0.0100	0.00032	ng/g	0.05	
	PCB-45	0.00305	0.00038	ng/g	0.01525	
	PCB-47	0.0100	0.00032	ng/g	0.05	
	PCB-5	0.000549	0.00051	ng/g	0.002745	
	PCB-51	0.00305	0.00038	ng/g	0.01525	
	PCB-61	0.00102	0.00024	ng/g	0.0051	
	PCB-65	0.0100	0.00032	ng/g	0.05	
	PCB-68	0.00201	0.00022	ng/g	0.01005	
	PCB-70	0.00102	0.00024	ng/g	0.0051	
	PCB-74	0.00102	0.00024	ng/g	0.0051	
	PCB-76	0.00102	0.00024	ng/g	0.0051	
	PCB-85	0.000142	0.000038	ng/g	0.00071	
	PCB-86	0.000205	0.000040	ng/g	0.001025	
	PCB-87	0.000205	0.000040	ng/g	0.001025	
	PCB-90	0.000331	0.000041	ng/g	0.001655	
	PCB-97	0.000205	0.000040	ng/g	0.001025	

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
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