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Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling	
Laboratory:	Test America, West Sacramento, California	
Laboratory Group:	580-81511-2	
Analyses/Method:	Clean Water Act - Dioxins and Furans / CWA1613B	
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 October 31, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-ST-1810	Equipment Blank
PDI-ST-T06A-1810	Sediment
PDI-ST-T06B-1810	Sediment
PDI-ST-T07A-1810	Sediment
PDI-ST-T07B-1810	Sediment

Data validation activities were conducted with reference to:

- EPA Method 1613B: *Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS (October 1994),*
- *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
✓	Ongoing precision and recovery (OPR) results
NA	Field duplicate results
✓	Labeled compound and 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. All method QC acceptance criteria were met.

Laboratory Blanks/Equipment Blanks

Laboratory method blanks and equipment blank results are evaluated as to whether there are contaminants detected above the estimated detection limit (EDL).

Target compounds were detected in the method blanks and the equipment blank associated with the samples in this data set. The equipment blank contamination, after laboratory method blank actions were applied, is summarized below for informational purposes only. Equipment blank contamination was not used to qualify field samples.

Blank ID	Compound	Result	EDL	Units
PDI-RB-ST-1810	1,2,3,4,6,7,8-HpCDF	3.1	0.98	pg/L

The NFG guidance stipulates that a conservative approach should be taken with regards to qualification of PCDD/PCDFs 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, 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.

OPR Results

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

Field duplicate RPDs were reviewed for conformance with the AECOM QC acceptance criteria of $\leq$ 50% [if one or both results were greater than five times the quantitation limit (QL)] for solid matrices and $\leq$ 30% [if one or both results were greater than five times the QL] for aqueous matrices.

No field duplicate was collected on a sample reported with this laboratory group. Precision was assessed using the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results.

Labeled Compound and 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 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; and
- the quantitative determination of PCDFs were not affected by the presence of polychlorinated diphenyl ether (PCDPE) interferences detected above the 2.5:1 S/N ratio limit.

All QC acceptance criteria were met with the following exceptions. 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 EMPC 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.

It should be noted that the "JN" qualifier was retained rather than replacement with the conventional overall "J" qualifier in instances where sample results were qualified for multiple quality control nonconformances.

Second Column Confirmation (2,3,7,8-TCDF)

The sample data were reviewed to ensure that results for 2,3,7,8-TCDF when analyzed on a DB-5 (or equivalent) column were confirmed on a second column (i.e., DB-225 or equivalent) when isomer specificity is not achieved. All sample results requiring confirmation were confirmed and results were reported from the confirmation column.

It should be noted that according to Section 11.3.5 of the laboratory's SOP, *"Any sample which 2,3,7,8-TCDF is identified above the lower calibration limit must be confirmed on a DB-225 column, SP-2331, or equivalent GC column."* This suggests that 2,3,7,8-TCDF results detected below the lower calibration limit (i.e., "J" values) are not confirmed on a secondary column by the laboratory. Professional judgment was used to take no action in instances where 2,3,7,8-TCDF was detected as "J" values on the primary column (i.e., DB-5).

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 dioxins and furans 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. Samples exhibiting percent solids <30% are qualified "J" or "UJ". Qualified sample results are shown in Table 1.

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: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-RB-ST-1810	WQ	1,2,3,4,6,7,8-HpCDF	3.1	0.98	0.98	pg/L	JN	bl,k
PDI-RB-ST-1810	WQ	1,2,3,4,7,8,9-HpCDF		4.4	1.3	pg/L	U	bl
PDI-RB-ST-1810	WQ	OCDD		15	2.3	pg/L	U	bl
PDI-RB-ST-1810	WQ	OCDF		6.8	2.0	pg/L	U	bl
PDI-ST-T06A-1810	SE	1,2,3,4,6,7,8-HpCDD	0.11	0.0028	0.0028	ug/kg	J	x
PDI-ST-T06A-1810	SE	1,2,3,4,6,7,8-HpCDF	0.019	0.00099	0.00099	ug/kg	J	x
PDI-ST-T06A-1810	SE	1,2,3,4,7,8,9-HpCDF		0.0012	0.0012	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,4,7,8-HxCDD		0.00076	0.00076	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,4,7,8-HxCDF		0.0011	0.0011	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,6,7,8-HxCDD	0.0064	0.00071	0.00071	ug/kg	J	x
PDI-ST-T06A-1810	SE	1,2,3,6,7,8-HxCDF		0.0011	0.0011	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,7,8,9-HxCDD	0.0038	0.00065	0.00065	ug/kg	J	x
PDI-ST-T06A-1810	SE	1,2,3,7,8,9-HxCDF		0.00060	0.00060	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,7,8-PeCDD		0.00061	0.00061	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	1,2,3,7,8-PeCDF		0.00048	0.00048	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	2,3,4,6,7,8-HxCDF		0.00072	0.00072	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	2,3,4,7,8-PeCDF		0.00053	0.00053	ug/kg	UJ	x
PDI-ST-T06A-1810	SE	2,3,7,8-TCDD	0.00063	0.00052	0.00052	ug/kg	J	x
PDI-ST-T06A-1810	SE	2,3,7,8-TCDF	0.0015	0.00034	0.00034	ug/kg	JN	k,x
PDI-ST-T06A-1810	SE	OCDD	0.94	0.0033	0.0033	ug/kg	J	x
PDI-ST-T06A-1810	SE	OCDF	0.061	0.0021	0.0021	ug/kg	J	x
PDI-ST-T06B-1810	SE	1,2,3,4,6,7,8-HpCDD	0.081	0.0021	0.0021	ug/kg	J	x
PDI-ST-T06B-1810	SE	1,2,3,4,6,7,8-HpCDF	0.017	0.00084	0.00084	ug/kg	JN	k,x
PDI-ST-T06B-1810	SE	1,2,3,4,7,8,9-HpCDF		0.00096	0.00096	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	1,2,3,4,7,8-HxCDD	0.0014	0.00087	0.00087	ug/kg	J	x
PDI-ST-T06B-1810	SE	1,2,3,4,7,8-HxCDF		0.0013	0.0013	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	1,2,3,6,7,8-HxCDD	0.0052	0.00078	0.00078	ug/kg	J	x
PDI-ST-T06B-1810	SE	1,2,3,6,7,8-HxCDF		0.0012	0.0012	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	1,2,3,7,8,9-HxCDD	0.0034	0.00073	0.00073	ug/kg	JN	k,x
PDI-ST-T06B-1810	SE	1,2,3,7,8,9-HxCDF		0.00065	0.00065	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	1,2,3,7,8-PeCDD		0.00053	0.00053	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	1,2,3,7,8-PeCDF		0.00042	0.00042	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	2,3,4,6,7,8-HxCDF		0.00076	0.00076	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	2,3,4,7,8-PeCDF	0.00092	0.00047	0.00047	ug/kg	J	x
PDI-ST-T06B-1810	SE	2,3,7,8-TCDD		0.00045	0.00045	ug/kg	UJ	x
PDI-ST-T06B-1810	SE	2,3,7,8-TCDF	0.0013	0.00024	0.00024	ug/kg	JN	k,x
PDI-ST-T06B-1810	SE	OCDD	0.65	0.0019	0.0019	ug/kg	J	x
PDI-ST-T06B-1810	SE	OCDF	0.045	0.00074	0.00074	ug/kg	J	x

Sample ID	Matrix	Compound	Result	RDL	EDL	Units	Validation Qualifiers	Validation Reason
PDI-ST-T07A-1810	SE	1,2,3,4,6,7,8-HpCDD	0.36	0.0098	0.0098	ug/kg	J	x
PDI-ST-T07A-1810	SE	1,2,3,4,6,7,8-HpCDF	0.060	0.0039	0.0039	ug/kg	J	x
PDI-ST-T07A-1810	SE	1,2,3,4,7,8,9-HpCDF		0.0043	0.0043	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,4,7,8-HxCDD		0.0031	0.0031	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,4,7,8-HxCDF		0.0047	0.0047	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,6,7,8-HxCDD	0.0098	0.0027	0.0027	ug/kg	J	x
PDI-ST-T07A-1810	SE	1,2,3,6,7,8-HxCDF		0.0044	0.0044	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,7,8,9-HxCDD	0.0088	0.0026	0.0026	ug/kg	JN	k,x
PDI-ST-T07A-1810	SE	1,2,3,7,8,9-HxCDF		0.0025	0.0025	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,7,8-PeCDD		0.0032	0.0032	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	1,2,3,7,8-PeCDF		0.0026	0.0026	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	2,3,4,6,7,8-HxCDF		0.0028	0.0028	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	2,3,4,7,8-PeCDF		0.0030	0.0030	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	2,3,7,8-TCDD		0.0023	0.0023	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	2,3,7,8-TCDF		0.0017	0.0017	ug/kg	UJ	x
PDI-ST-T07A-1810	SE	OCDD	5.4	0.0081	0.0081	ug/kg	J	x
PDI-ST-T07A-1810	SE	OCDF	0.65	0.0039	0.0039	ug/kg	J	x
PDI-ST-T07B-1810	SE	1,2,3,4,6,7,8-HpCDD	0.13	0.0025	0.0025	ug/kg	J	x
PDI-ST-T07B-1810	SE	1,2,3,4,6,7,8-HpCDF	0.029	0.0011	0.0011	ug/kg	JN	k,x
PDI-ST-T07B-1810	SE	1,2,3,4,7,8,9-HpCDF	0.0022	0.0012	0.0012	ug/kg	J	x
PDI-ST-T07B-1810	SE	1,2,3,4,7,8-HxCDD		0.00097	0.00097	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	1,2,3,4,7,8-HxCDF		0.0013	0.0013	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	1,2,3,6,7,8-HxCDD	0.0079	0.00097	0.00097	ug/kg	J	x
PDI-ST-T07B-1810	SE	1,2,3,6,7,8-HxCDF		0.0010	0.0010	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	1,2,3,7,8,9-HxCDD	0.0044	0.00086	0.00086	ug/kg	J	x
PDI-ST-T07B-1810	SE	1,2,3,7,8,9-HxCDF		0.00062	0.00062	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	1,2,3,7,8-PeCDD		0.00079	0.00079	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	1,2,3,7,8-PeCDF		0.00070	0.00070	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	2,3,4,6,7,8-HxCDF	0.0013	0.00068	0.00068	ug/kg	J	x
PDI-ST-T07B-1810	SE	2,3,4,7,8-PeCDF		0.00085	0.00085	ug/kg	UJ	x
PDI-ST-T07B-1810	SE	2,3,7,8-TCDD	0.0011	0.00058	0.00058	ug/kg	JN	k,x
PDI-ST-T07B-1810	SE	2,3,7,8-TCDF	0.0019	0.00043	0.00043	ug/kg	J	x
PDI-ST-T07B-1810	SE	OCDD	1.0	0.0021	0.0021	ug/kg	J	x
PDI-ST-T07B-1810	SE	OCDF	0.080	0.00089	0.00089	ug/kg	J	x

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
ma	Multiple analyses, sample analyzed more than once, a value from another analysis should be used
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	Low % solids
y	Serial dilution results
z	ICS results