AECOM
257 West Genesee Street, Suite 400
Buffalo, NY 14202

716.856.5636 tel
716.856.2545 fax

Data Validation Report

Project:	Portland Harbor Pre-Remedial Design Investigation and Baseline Sampling
Laboratory:	Test America, West Sacramento, California
Laboratory Group:	580-79257-2
Analyses/Method:	Clean Water Act - Dioxins and Furans / CWA1613B
Validation Level:	Stage 2A (Stage 4 for PDI-RB-SS-180731 and PDI-SC-S062-4TO6)
AECOM Project Number:	60566335.2.12

Prepared by:	Peter Fairbanks/AECOM	Completed on: 09/13/2018
Reviewed by:	George Kisluk/AECOM	File Name: 580-79257-2 Stage 2A -4 DVR

SUMMARY

The samples listed below were collected by AECOM in Portland Harbor in Portland, OR on July 31 and August 1, 2018.

Sample ID	Matrix/Sample Type
PDI-RB-SS-180731	Equipment Blank
PDI-SC-S023-0TO2	Sediment
PDI-SC-S023-2TO3.9	Sediment
PDI-SC-S023-3.9TO5.3	Sediment
PDI-SC-S023-5.3TO7.2	Sediment
PDI-SC-S023-7.2TO8.8	Sediment
PDI-SC-S031-0TO2	Sediment
PDI-SC-S031-2TO4	Sediment
PDI-SC-S031-4TO5.5	Sediment
PDI-SC-S031-5.5TO7	Sediment
PDI-SC-S031-7TO9.2	Sediment
PDI-SC-S038-0TO2	Sediment
PDI-SC-S038-2TO3.4	Sediment
PDI-SC-S038-3.4TO5.4	Sediment
PDI-SC-S038-5.4TO7.2	Sediment
PDI-SC-S062-0TO2	Sediment
PDI-SC-S062-2TO4	Sediment
PDI-SC-S062-6TO7.7	Sediment
PDI-SC-S085-0TO2	Sediment

Sample ID	Matrix/Sample Type
PDI-SC-S085-2TO4	Sediment
PDI-SC-S085-4TO6.4	Sediment
PDI-SC-S085-4TO6.4D	Field Duplicate of PDI-SC-S085-4TO6.4
PDI-RB-SS-180731	Equipment Blank
PDI-SC-S062-4TO6	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
✓	Mass resolution/ window defining mix (WDM)/isomer specificity check (ISC) results
✓	Initial calibration/continuing calibration verification
✗	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.

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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.

Mass Resolution/ WDM/ISC Results

The data were reviewed to ensure that:

- the perfluorokerosene (PFK) molecular leak was performed at the correct frequency (at the beginning and end of a 12-hour shift) and the mass resolution was at a resolving power of > 10,000;
- the window defining mix (WDM) containing the first and last eluting isomers in each homologous series was analyzed at the correct frequency;
- the isomer specificity check (ISC) standard criteria were met for the chromatographic resolution of 2,3,7,8-TCDD on the DB-5 column and of 2,3,7,8-TCDF on the DB-225 column.

All method QC acceptance 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 (as summarized by the laboratory);
- the initial calibration percent relative standard deviation (%RSD) method acceptance criteria were met for all native and labeled compounds;
- and the calibration verification standard (CCV) method acceptance criteria were met.

It should be noted that according to Section 17.1 of the laboratory's SOP, the following method modification was noted:

Section 10.2.4 of Method 1613 reads "The absolute retention time of 13C-1,2,3,4-TCDD shall exceed 25.0 minutes on the DB-5 column, and the retention time of 13C-1,2,3,4-TCDD shall exceed 15.0 minutes on the DB-225 column; otherwise the GC temperature program shall be adjusted and this test repeated until the above-stated minimum retention time criteria are met."

Our retention time on both columns deviate from the above method, but using section 1.5 of Method 1613 "the analyst is permitted to modify the method to lower the cost of measurements, provided that all performance criteria in this method are met," we have modified the GC program

to provide a shorter runtime while still meeting method performance criteria thus lowering the cost of analysis.

No data validation actions were taken on this basis.

Professional judgment was used to take no action in instances where the labeled compounds were not within the relative retention time (RRT) criterion since the required RRT criteria were met for all native compounds.

Additionally, professional judgment was used to take no action in instances where the recovery standard RT in the CCV was not within ± 15 seconds of the RT in the mid-level standard of the associated ICAL. These nonconformances result from routine column maintenance. A WDM is analyzed daily prior to sample analysis and retention times are adjusted accordingly; thus, data are not adversely impacted.

Laboratory Blanks/Equipment Blanks

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

Target compounds were detected in the method blanks and 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	RDL	Units
PDI-RB-SS-180731	1,2,3,4,6,7,8-HpCDD	1.1	0.20	pg/L
	1,2,3,4,6,7,8-HpCDF	0.60	0.23	pg/L
	1,2,3,4,7,8-HxCDD	1.2	0.26	pg/L
	1,2,3,7,8,9-HxCDF	0.35	0.16	pg/L
	OCDD	11	0.18	pg/L
	OCDF	1.9	0.23	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 ≥ the blank result and ≤ 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.

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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 ≤ 50% [if one or both results were greater than five times the quantitation limit (QL)] for solid matrices and ≤ 30% [if one or both results were greater than five times the QL] for aqueous matrices.

All field duplicate precision criteria were met.

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.

PFK Lock Mass

The laboratory confirmed that a lock mass is monitored for each instrument descriptor.

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", “J+”, and “J-“ qualifiers in instances where sample results were qualified for multiple quality control nonconformances.

Quantitation

Sample results detected at concentrations greater than the highest calibration standard, qualified by the laboratory with an “E”, require secondary dilutions in order to bring the concentrations down within the linear range of calibration, per Method 1613B. This was not done by the laboratory.

It should be noted that according to Section 12.1.7 of the laboratory’s SOP, unless the affected peak saturates the instrument detector, secondary dilutions are not performed. Furthermore, *“Historic data indicates that for the isotope dilution method, dilution and re-injection will not produce significantly different results from those reported with the “E” qualifier.”*

Despite the laboratory’s SOP, NFG guidance stipulates that if a sample is not properly diluted to bring the results within the linear range of calibration, then the results are qualified “J”.

Qualified sample results are summarized in Table 1.

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).

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 "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.

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. 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: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	RDL	Units	Validation Qualifiers	Validation Reason
PDI-RB-SS-180731	WQ	1,2,3,4,6,7,8-HpCDD	1.1	0.20	pg/L	JN	bl,k
PDI-RB-SS-180731	WQ	1,2,3,4,6,7,8-HpCDF	0.60	0.23	pg/L	JN	k
PDI-RB-SS-180731	WQ	1,2,3,7,8,9-HxCDF	0.35	0.16	pg/L	JN	k
PDI-RB-SS-180731	WQ	OCDD	11	0.18	pg/L	J+	bl
PDI-RB-SS-180731	WQ	OCDF	1.9	0.23	pg/L	J+	bl
PDI-SC-S023-0TO2	SE	1,2,3,4,7,8-HxCDD	0.00095	0.00021	ug/kg	JN	k
PDI-SC-S023-0TO2	SE	1,2,3,7,8,9-HxCDD	0.0024	0.00019	ug/kg	JN	k
PDI-SC-S023-0TO2	SE	2,3,7,8-TCDD	0.00033	0.000088	ug/kg	JN	k
PDI-SC-S023-2TO3.9	SE	OCDD	4.8	0.0025	ug/kg	J	q
PDI-SC-S023-3.9TO5.3	SE	2,3,7,8-TCDD	0.00012	0.000045	ug/kg	JN	k
PDI-SC-S023-5.3TO7.2	SE	2,3,7,8-TCDD	0.00023	0.000063	ug/kg	JN	k
PDI-SC-S023-7.2TO8.8	SE	1,2,3,7,8,9-HxCDD	0.00035	0.00022	ug/kg	JN	k
PDI-SC-S031-0TO2	SE	1,2,3,4,6,7,8-HpCDF	0.046	0.0010	ug/kg	JN	k
PDI-SC-S031-0TO2	SE	1,2,3,4,7,8,9-HpCDF	0.0025	0.0015	ug/kg	JN	k
PDI-SC-S031-2TO4	SE	1,2,3,7,8,9-HxCDD	0.0012	0.00017	ug/kg	JN	k
PDI-SC-S031-2TO4	SE	1,2,3,7,8-PeCDD	0.00038	0.00028	ug/kg	JN	k
PDI-SC-S031-4TO5.5	SE	1,2,3,6,7,8-HxCDD	0.00019	0.000051	ug/kg	JN	k
PDI-SC-S031-5.5TO7	SE	1,2,3,4,6,7,8-HpCDF	0.00053	0.00011	ug/kg	JN	k
PDI-SC-S031-5.5TO7	SE	OCDF	0.0010	0.00023	ug/kg	J+	bl
PDI-SC-S031-7TO9.2	SE	1,2,3,4,6,7,8-HpCDF	0.00018	0.000043	ug/kg	JN	bl,k
PDI-SC-S031-7TO9.2	SE	1,2,3,7,8,9-HxCDD	0.00013	0.000055	ug/kg	JN	k
PDI-SC-S031-7TO9.2	SE	OCDF	0.0011	0.00011	ug/kg	J+	bl
PDI-SC-S038-0TO2	SE	1,2,3,4,7,8-HxCDD	0.00027	0.00015	ug/kg	J+	bl
PDI-SC-S038-0TO2	SE	2,3,7,8-TCDF	0.00052	0.00035	ug/kg	JN	k
PDI-SC-S038-2TO3.4	SE	1,2,3,4,7,8-HxCDD	0.00014	0.000093	ug/kg	J+	bl
PDI-SC-S038-2TO3.4	SE	1,2,3,6,7,8-HxCDD	0.00012	0.000084	ug/kg	JN	k
PDI-SC-S038-2TO3.4	SE	1,2,3,7,8,9-HxCDD	0.00016	0.000082	ug/kg	JN	k
PDI-SC-S038-2TO3.4	SE	OCDF	0.0028	0.00024	ug/kg	J+	bl
PDI-SC-S038-3.4TO5.4	SE	1,2,3,4,6,7,8-HpCDF	0.00031	0.000053	ug/kg	JN	bl,k
PDI-SC-S038-3.4TO5.4	SE	1,2,3,4,7,8-HxCDD	0.00015	0.000048	ug/kg	J+	bl
PDI-SC-S038-3.4TO5.4	SE	1,2,3,6,7,8-HxCDD	0.000081	0.000044	ug/kg	JN	k
PDI-SC-S038-3.4TO5.4	SE	OCDF		0.00034	ug/kg	U	bl
PDI-SC-S038-5.4TO7.2	SE	1,2,3,4,6,7,8-HpCDF	0.00031	0.00010	ug/kg	JN	bl,k
PDI-SC-S038-5.4TO7.2	SE	1,2,3,4,7,8-HxCDD	0.00022	0.000067	ug/kg	J+	bl
PDI-SC-S038-5.4TO7.2	SE	1,2,3,7,8-PeCDF	0.00025	0.000069	ug/kg	JN	k
PDI-SC-S062-0TO2	SE	1,2,3,4,6,7,8-HpCDF	0.014	0.00040	ug/kg	JN	k
PDI-SC-S062-0TO2	SE	1,2,3,7,8,9-HxCDF	0.00037	0.00022	ug/kg	JN	k
PDI-SC-S062-2TO4	SE	1,2,3,7,8-PeCDD	0.00033	0.00014	ug/kg	JN	k

Sample ID	Matrix	Compound	Result	RDL	Units	Validation Qualifiers	Validation Reason
PDI-SC-S062-6TO7.7	SE	2,3,7,8-TCDD	0.00026	0.000066	ug/kg	JN	k
PDI-SC-S085-0TO2	SE	1,2,3,7,8-PeCDD	0.00048	0.00018	ug/kg	JN	k
PDI-SC-S085-0TO2	SE	2,3,4,6,7,8-HxCDF	0.0018	0.00037	ug/kg	JN	k
PDI-SC-S085-2TO4	SE	1,2,3,7,8,9-HxCDD	0.00016	0.000099	ug/kg	JN	k
PDI-SC-S085-2TO4	SE	2,3,4,7,8-PeCDF	0.00020	0.00013	ug/kg	JN	k
PDI-SC-S085-2TO4	SE	2,3,7,8-TCDD	0.00035	0.000087	ug/kg	JN	k
PDI-SC-S085-2TO4	SE	OCDF	0.0022	0.00015	ug/kg	J+	bl
PDI-SC-S085-4TO6.4	SE	1,2,3,6,7,8-HxCDF	0.00021	0.000079	ug/kg	JN	k
PDI-SC-S085-4TO6.4	SE	OCDF	0.0014	0.00012	ug/kg	JN	bl,k
PDI-SC-S085-4TO6.4D	SE	1,2,3,4,6,7,8-HpCDF	0.00046	0.000069	ug/kg	J+	bl
PDI-SC-S085-4TO6.4D	SE	1,2,3,4,7,8-HxCDD	0.00024	0.000050	ug/kg	J+	bl
PDI-SC-S085-4TO6.4D	SE	1,2,3,7,8,9-HxCDD	0.00021	0.000047	ug/kg	JN	k
PDI-SC-S085-4TO6.4D	SE	1,2,3,7,8-PeCDF	0.00011	0.000051	ug/kg	JN	k
PDI-SC-S085-4TO6.4D	SE	OCDF	0.0010	0.00011	ug/kg	JN	bl,k
PDI-RB-SS-180731	WQ	1,2,3,4,6,7,8-HpCDD	1.1	0.20	pg/L	JN	bl,k
PDI-RB-SS-180731	WQ	1,2,3,4,6,7,8-HpCDF	0.60	0.23	pg/L	JN	k
PDI-RB-SS-180731	WQ	1,2,3,7,8,9-HxCDF	0.35	0.16	pg/L	JN	k
PDI-RB-SS-180731	WQ	OCDD	11	0.18	pg/L	J+	bl
PDI-RB-SS-180731	WQ	OCDF	1.9	0.23	pg/L	J+	bl
PDI-SC-S062-4TO6	SE	1,2,3,6,7,8-HxCDF	0.0014	0.00018	ug/kg	JN	k
PDI-SC-S062-4TO6	SE	1,2,3,7,8,9-HxCDF	0.00018	0.00013	ug/kg	JN	k
PDI-SC-S062-4TO6	SE	2,3,7,8-TCDD	0.00030	0.000074	ug/kg	JN	k

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