



LABORATORY DATA CONSULTANTS, INC.

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Windward Environmental, LLC
200 West Mercer Street, Suite 401
Seattle, WA 98119
ATTN: Amara Vandervort
amarav@windwardenv.com

November 10, 2021

SUBJECT: Duwamish AOC4 - Data Validation

Dear Ms. Vandervort,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on October 12 and 14, 2021. Attachment 1 is a summary of the samples that were reviewed for analysis.

LDC Project #52274:

SDG #

21F0442, 21G0213
21G0108, 21G0127
21G0138

Fraction

Polychlorinated Dioxins/Dibenzofurans

The data validation was performed under Stage 2B & 4 guidelines. The analysis was validated using the following documents, as applicable to method:

- Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020)
- USEPA National Functional Guidelines (NFG) for Organic Superfund Methods Data Review (January 2017)
- USEPA National Functional Guidelines (NFG) for High Resolution Superfund Methods Data Review (April 2016)

Please feel free to contact us if you have any questions.

Sincerely,

Pei Geng
Project Manager/Senior Chemist
pgeng@lab-data.com

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Duwamish AOC4

LDC Report Date: November 4, 2021

Parameters: Polychlorinated Dioxins/Dibenzofurans

Validation Level: Stage 2B

Laboratory: Analytical Resources, Inc., Tukwila, WA

Sample Delivery Group (SDG): 21F0442

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
LDW21-SC564A	21F0442-01	Sediment	06/29/21
LDW21-SC564B	21F0442-02	Sediment	06/29/21
LDW21-SC564C	21F0442-03	Sediment	06/29/21
LDW21-SC564D	21F0442-04	Sediment	06/29/21
LDW21-SC564E	21F0442-05	Sediment	06/29/21
LDW21-SC564G	21F0442-06	Sediment	06/29/21
LDW21-SC564I	21F0442-07	Sediment	06/29/21
LDW21-SC564IRE	21F0442-07RE	Sediment	06/29/21
LDW21-SC564K	21F0442-08	Sediment	06/29/21
LDW21-SC564ADUP	21F0442-01DUP	Sediment	06/29/21

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020) and a modified outline of the USEPA National Functional Guidelines (NFG) for Organic Superfund Methods Data Review (January 2017). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) Method 1613B

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered not detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and less than or equal to 35.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were within the QC limits for all analytes and labeled compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration results were within the QC limits for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BJG0187-BLK1	07/08/21	OCDD	0.865 ug/Kg	LDW21-SC564A LDW21-SC564B LDW21-SC564C LDW21-SC564D LDW21-SC564E LDW21-SC564G LDW21-SC564I LDW21-SC564K
BJH0186-BLK1	08/09/21	1,2,3,4,6,7,8-HpCDD OCDD Total PeCDF Total HxCDD Total HpCDD	0.228 ug/Kg 1.55 ug/Kg 0.0476 ug/Kg 0.180 ug/Kg 0.103 ug/Kg	LDW21-SC564IRE

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater than the concentrations found in the associated laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicate Sample Analysis

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
LDW21-SC564ADUP (LDW21-SC564A LDW21-SC564ADUP)	OCDF	37.1 (≤25)	J (all detects)	A

VIII. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

SRM ID (Associated Samples)	Analyte	%R (Limits)	Flag*	A or P
BJG0187-SRM1 (LDW21-SC564A LDW21-SC564B LDW21-SC564C LDW21-SC564D LDW21-SC564E LDW21-SC564G LDW21-SC564I LDW21-SC564K LDW21-SC564ADUP)	2,3,7,8-TCDF	0 (50-150)	J (all detects) UJ (all non-detects)	A
	1,2,3,7,8,9-HxCDF	0 (50-150)	J (all detects) UJ (all non-detects)	

For BJG0187-SRM1, although the percent recoveries were severely low for 2,3,7,8-TCDF and 1,2,3,7,8,9-HxCDF due to the laboratory using a smaller amount, the associated sample results were qualified as estimated (J/UJ) since the LCS recoveries were within the QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Affected Analyte	Flag	A or P
LDW21-SC564I	13C-2,3,7,8-TCDF	13.2 (24-169)	2,3,7,8-TCDF	J (all detects) UJ (all non-detects)	P
	13C-2,3,7,8-TCDD	23.1 (25-164)	2,3,7,8-TCDD	J (all detects) UJ (all non-detects)	

XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria with the following exceptions:

Sample	Analyte	Flag	A or P
All samples in SDG 21F0442	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A
All samples in SDG 21F0442	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U	A

Raw data were not reviewed for Stage 2B validation.

XII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

XIII. System Performance

Raw data were not reviewed for Stage 2B validation.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

In the case where more than one result was reported for an individual sample, the least technically acceptable results were deemed not reportable as follows:

Sample	Analyte	Reason	Flag	A or P
LDW21-SC564I	All analytes	Internal standard outside of limits.	Not reportable	-

Due to DUP RPD, SRM %R, and results reported by the laboratory as EMPCs, data were qualified as estimated in ten samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 21F0442

Sample	Analyte	Flag	A or P	Reason
LDW21-SC564A LDW21-SC564ADUP	OCDF	J (all detects)	A	Duplicate sample analysis (RPD)
LDW21-SC564A LDW21-SC564B LDW21-SC564C LDW21-SC564D LDW21-SC564E LDW21-SC564G LDW21-SC564K LDW21-SC564ADUP	2,3,7,8-TCDF 1,2,3,7,8,9-HxCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Standard reference materials (%R)
LDW21-SC564A LDW21-SC564B LDW21-SC564C LDW21-SC564D LDW21-SC564E LDW21-SC564G LDW21-SC564IRE LDW21-SC564K LDW21-SC564ADUP	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A	Target analyte quantitation (EMPC)
LDW21-SC564A LDW21-SC564B LDW21-SC564C LDW21-SC564D LDW21-SC564E LDW21-SC564G LDW21-SC564IRE LDW21-SC564K LDW21-SC564ADUP	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U	A	Target analyte quantitation (EMPC)
LDW21-SC564I	All analytes	Not reportable	-	Overall assessment of data

Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG 21F0442

No Sample Data Qualified in this SDG

Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG 21F0442

No Sample Data Qualified in this SDG

LDC #: 52274A21
 SDG #: 21F0442
 Laboratory: Analytical Resources, Inc., Tukwila, WA

VALIDATION COMPLETENESS WORKSHEET
 Stage 2B

Date: 10/25/21
 Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A	
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	$\pm SD \leq 20/35\%$, $1\sigma \leq QC$ limits
IV.	Continuing calibration	A	$CV \leq QC$ limits
V.	Laboratory Blanks	N	
VI.	Field blanks	N	
VII.	Matrix spike/Matrix spike duplicates / DNP	N/SW	
VIII.	Laboratory control samples / SPN	A/SW	LC9
IX.	Field duplicates	N	
X.	Internal standards	N	
XI.	Target analyte quantitation	N	
XII.	Target analyte identification	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	N	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	LDW21-SC564A	21F0442-01	Sediment	06/29/21
2	LDW21-SC564B	21F0442-02	Sediment	06/29/21
3	LDW21-SC564C	21F0442-03	Sediment	06/29/21
4	LDW21-SC564D	21F0442-04	Sediment	06/29/21
5	LDW21-SC564E	21F0442-05	Sediment	06/29/21
6	LDW21-SC564G	21F0442-06	Sediment	06/29/21
7	LDW21-SC564I	21F0442-07	Sediment	06/29/21
8	LDW21-SC564IRE	21F0442-07RE	Sediment	06/29/21
9	LDW21-SC564K	21F0442-08	Sediment	06/29/21
10	LDW21-SC564ADUP	21F0442-01DUP	Sediment	06/29/21
11				

Notes:

BJE0187BK				
BJE0186				
BJH0186BK				

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes: _____

VALIDATION FINDINGS WORKSHEET
Blanks

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Were all samples associated with a method blank?
- Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y N N/A Was the method blank contaminated?

Blank extraction date: 7/8/21 Blank analysis date: 7/9/21 Associated samples: 1-7.9
Conc. units: 15 fg

Compound	Blank ID	Sample Identification							
BLK	0187-BK-1								
<u>F</u>	<u>0.865</u>								

Blank extraction date: 8/9/21 Blank analysis date: 8/11/21
Conc. units: 15 fg Associated Samples: 8

Compound	Blank ID	Sample Identification							
BLK	0186-BK-1								
<u>F</u>	<u>0.228</u>								
<u>F</u>	<u>1.55</u>								
<u>W</u>	<u>0.0476</u>								
<u>T</u>	<u>0.180</u>								
<u>U</u>	<u>0.103</u>								

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Was a LCS required?
- N N/A Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?
- N N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

#	Date	Lab ID/Reference	Compound	LCS SRM %R (Limits)	LCS %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		18T-SRM1	H	0 (50-150)	()	()	1-T. 9-10	<input checked="" type="checkbox"/> N/A *
			N	0 ()	()	()	(lots + N/A)	<input checked="" type="checkbox"/> N/A
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** due to Lab using smaller amount. LCS is within QC limits*

VALIDATION FINDINGS WORKSHEET
Internal Standards

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y ~~N~~ ~~N/A~~ Are all internal standard recoveries were within the QC criteria?

Y ~~N~~ ~~N/A~~ Was the S/N ratio all internal standard peaks ≥ 10 ?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit)	Qualifications
		T (det+ND)	13C-H	13.2 (24-169)	✓ N/A (H, V)
			13C-A	23.1 (25-164)	↓ (A, R)
			37C-A	23.2 (35-197)	Normal (no assigned)

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported RLs

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y (N) N/A
 Y N (N/A)

Were the correct labeled compound, quantitation ions and relative response factors (RRF) used to quantitate the compound?
 Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	All compounds reported as estimated maximum possible concentration (EMPC) > RL		Jdets/A
		All	All compounds reported as estimated maximum possible concentration (EMPC) < RL		U/A

Comments: See sample calculation verification worksheet for recalculations

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Duwamish AOC4
LDC Report Date: November 4, 2021
Parameters: Polychlorinated Dioxins/Dibenzofurans
Validation Level: Stage 2B
Laboratory: Analytical Resources, Inc., Tukwila, WA
Sample Delivery Group (SDG): 21G0213

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
LDW21-IT655B	21G0213-01	Sediment	07/19/21
LDW21-IT655C	21G0213-02	Sediment	07/19/21
LDW21-IT655E	21G0213-03	Sediment	07/19/21
LDW21-IT663B	21G0213-06	Sediment	07/19/21
LDW21-IT663BDL	21G0213-06DL	Sediment	07/19/21
LDW21-IT663C	21G0213-07	Sediment	07/19/21
LDW21-IT663CDL	21G0213-07DL	Sediment	07/19/21
LDW21-IT663E	21G0213-08	Sediment	07/19/21

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020) and a modified outline of the USEPA National Functional Guidelines (NFG) for High Resolution Superfund Methods Data Review (April 2016). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) Method 1613B

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered not detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and less than or equal to 35.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were within the QC limits for all analytes and labeled compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration results were within the QC limits for all analytes and labeled compounds with the following exceptions:

Date	Analyte	Concentration (Limits)	Associated Samples	Affected Analyte	Flag	A or P
09/21/21	13C-1,2,3,4,6,7,8-HpCDF 13C-1,2,3,4,7,8,9-HpCDF	76.2 (78-129) 72.9 (77-129)	LDW21-IT655B LDW21-IT655C	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
09/22/21 (21092044)	13C-1,2,3,4,6,7,8-HpCDF 13C-1,2,3,4,7,8,9-HpCDF	77.3 (78-129) 72.5 (77-129)	LDW21-IT655E LDW21-IT663B LDW21-IT663C LDW21-IT663E	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

Date	Analyte	Concentration (Limits)	Associated Samples	Affected Analyte	Flag	A or P
09/22/21 (21092054)	13C-2,3,4,7,8-PeCDF 13C-1,2,3,4,7,8,9-HpCDF	76.4 (77-130) 76.0)77-129)	LDW21-IT663BDL LDW21-IT663CDL	2,3,4,7,8-PeCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) J (all detects)	P

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BJH0203-BLK	09/09/21	1,2,3,4,6,7,8-HpCDD OCDD Total HpCDD	0.247 ug/Kg 4.03 ug/Kg 0.266 ug/Kg	All samples in SDG 21G0213

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
LDW21-IT655B	1,2,3,4,6,7,8-HpCDD	2.19 ug/Kg	2.19U ug/Kg
LDW21-IT655C	1,2,3,4,6,7,8-HpCDD OCDD	1.27 ug/Kg 15.9 ug/Kg	1.27U ug/Kg 15.9U ug/Kg
LDW21-IT655E	1,2,3,4,6,7,8-HpCDD OCDD Total HpCDD	1.31 ug/Kg 15.4 ug/Kg 1.31 ug/Kg	1.31U ug/Kg 15.4U ug/Kg 1.31J ug/Kg

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

VIII. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Labeled Compounds

All percent recoveries (%R) for labeled compounds used to quantitate target analytes were within QC limits.

XI. Target Analyte Quantitation

All target analyte quantitation met validation criteria with the following exceptions:

Sample	Analyte	Flag	A or P
All samples in SDG 21G0213	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A
All samples in SDG 21G0213	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U (all non-detects)	A

Sample	Analyte	Finding	Criteria	Flag	A or P
LDW21-IT663BDL LDW21-IT663CDL	OCDD	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	A

Raw data were not reviewed for Stage 2B validation.

XII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

XIII. System Performance

Raw data were not reviewed for Stage 2B validation.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

In the case where more than one result was reported for an individual sample, the least technically acceptable results were deemed not reportable as follows:

Sample	Analyte	Reason	Flag	A or P
LDW21-IT663B LDW21-IT663C	1,2,3,4,6,7,8-HpCDD OCDD	Results exceeded calibration range.	Not reportable	-
LDW21-IT663BDL LDW21-IT663CDL	All analytes except 1,2,3,4,6,7,8-HpCDD OCDD	Results from undiluted analyses were more usable.	Not reportable	-

Due to continuing calibration concentration, analytes reported as EMPC, and exceeded calibration range, data were qualified as estimated or not detected in eight samples.

Due to laboratory blank contamination, data were qualified as estimated or not detected in three samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 21G0213**

Sample	Analyte	Flag	A or P	Reason
LDW21-IT655B LDW21-IT655C LDW21-IT655E LDW21-IT663E	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Continuing calibration (concentration)
LDW21-IT655B LDW21-IT655C LDW21-IT655E LDW21-IT663B LDW21-IT663BDL LDW21-IT663C LDW21-IT663CDL LDW21-IT663E	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A	Target analyte quantitation (EMPC)
LDW21-IT655B LDW21-IT655C LDW21-IT655E LDW21-IT663B LDW21-IT663BDL LDW21-IT663C LDW21-IT663CDL LDW21-IT663E	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U (all non-detects)	A	Target analyte quantitation (EMPC)
LDW21-IT663BDL LDW21-IT663CDL	OCDD	J (all detects)	A	Target Analyte Quantitation (exceeded calibration range)
LDW21-IT663B LDW21-IT663C	1,2,3,4,6,7,8-HpCDD OCDD	Not reportable	-	Overall assessment of data
LDW21-IT663BDL LDW21-IT663CDL	All analytes except 1,2,3,4,6,7,8-HpCDD OCDD	Not reportable	-	Overall assessment of data

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG 21G0213**

Sample	Analyte	Modified Final Concentration	A or P
LDW21-IT655B	1,2,3,4,6,7,8-HpCDD	2.19U ug/Kg	A
LDW21-IT655C	1,2,3,4,6,7,8-HpCDD OCDD	1.27U ug/Kg 15.9U ug/Kg	A
LDW21-IT655E	1,2,3,4,6,7,8-HpCDD OCDD Total HpCDD	1.31U ug/Kg 15.4U ug/Kg 1.31J ug/Kg	A

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary -
SDG 21G0213**

No Sample Data Qualified in this SDG

LDC #: 52274B21

VALIDATION COMPLETENESS WORKSHEET

SDG #: 21G0213

Stage 2B

Laboratory: Analytical Resources, Inc., Tukwila, WA

Date: 10/16/21

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A	
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	ISO = 20/35% . Key QC limits
IV.	Continuing calibration	SW	QC limits
V.	Laboratory Blanks	SW	
VI.	Field blanks	N	
VII.	Matrix spike/Matrix spike duplicates	N	es
VIII.	Laboratory control samples / SW	A	LCs
IX.	Field duplicates	N	
X.	Internal standards	A	
XI.	Target analyte quantitation	SW	
XII.	Target analyte identification	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	SW	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	LDW21-IT655B	21G0213-01	Sediment	07/19/21
2	LDW21-IT655C	21G0213-02	Sediment	07/19/21
3	LDW21-IT655E	21G0213-03	Sediment	07/19/21
4	LDW21-IT663B	21G0213-06	Sediment	07/19/21
5	LDW21-IT663BDL	21G0213-06DL	Sediment	07/19/21
6	LDW21-IT663C	21G0213-07	Sediment	07/19/21
7	LDW21-IT663CDL	21G0213-07DL	Sediment	07/19/21
8	LDW21-IT663E	21G0213-08	Sediment	07/19/21
9				
10				

Notes:

BNH0203				

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes: _____

VALIDATION FINDINGS WORKSHEET Continuing Calibration

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Was a routine calibration was performed at the beginning and end of each 12 hour period?
 Y N N/A Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled?
 Y N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: 20.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	9/17/21	21092022A	13C-0	72.1 (TT-129)		MB	✓ N/A
			13C-P	69.9 (TT-129)			↓
	9/17/21	21092031A	13C-0	76.2		1-2 (det B) + N (2)	↓
			13C-P	72.9			↓
	9/22/21	21092044	13C-0	77.3		3-4, 6, 8 (det B) + N (2)	↓
			13C-P	72.5			↓
	9/22/21	21092054	13C-HJ	76.4 (TT-130)		5, 7 (det B)	↓
			13C-P	76.0 (TT-129)			↓

PCDDs			PCDFs		
	Selected ions (m/z)	Ion Abundance Ratio		Selected ions (m/z)	Ion Abundance Ratio
Tetra-	M/M+2	0.65-0.89	Tetra-	M/M+2	0.65-0.89
Penta-	M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78
Hexa-	M+2/M+4	1.05-1.43	Hexa-	M+2/M+4	1.05-1.43
Hexa- ¹³ C-HxCDF (IS) only	M/M+2	0.43-0.59	Hexa- ¹³ C-HxCDF (IS) only	M/M+2	0.43-0.59
Hepta- ¹³ C-HpCDF (IS) only	M/M+2	0.37-0.51	Hepta- ¹³ C-HpCDF (IS) only	M/M+2	0.37-0.51
Hepta-	M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20
Octa-	M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02

VALIDATION FINDINGS WORKSHEET
Blanks

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Were all samples associated with a method blank?
- N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- N N/A Was the method blank contaminated?

Blank extraction date: 9/9/21 Blank analysis date: 9/21/21 Associated samples: All
 Conc. units: ng/g

Compound	Blank ID	Sample Identification							
		1	2	3					
	BHT0203-BH								
F	0.24T	2.19/U	1.27/U	1.31/U					
Q	4.03	20.5/U	15.9/U	15.4/U					
U	0.26			1.31/U					

Blank extraction date: _____ Blank analysis date: _____
 Conc. units: _____ Associated Samples: _____

Compound	Blank ID	Sample Identification							

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
 All contaminants within five times the method blank concentration were qualified as not detected, "U".

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported RLs

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y (N) N/A
Y N N/A

Were the correct labeled compound, quantitation ions and relative response factors (RRF) used to quantitate the compound?
Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	All compounds reported as estimated maximum possible concentration (EMPC) > RL		Jdets/A
		All	All compounds reported as estimated maximum possible concentration (EMPC) < RL		U/A
		5, 7	G exceeded calibration range		Jdets/A

Comments: See sample calculation verification worksheet for recalculations

VALIDATION FINDINGS WORKSHEET
Overall Assessment of Data

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

All available information pertaining to the data were reviewed using professional judgement to compliment the determination of the overall quality of the data.

Y N N/A Was the overall quality and usability of the data acceptable?

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		4, 6	F, CF > catenb range		N/A
		5, 7	F, CF All except F, CF		↓

Comments: _____

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Duwamish AOC4

LDC Report Date: November 4, 2021

Parameters: Polychlorinated Dioxins/Dibenzofurans

Validation Level: Stage 2B

Laboratory: Analytical Resources, Inc., Tukwila, WA

Sample Delivery Group (SDG): 21G0108

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
LDW21-IT654B	21G0108-02	Sediment	07/08/21
LDW21-IT654C	21G0108-03	Sediment	07/08/21
LDW21-IT654E	21G0108-04	Sediment	07/08/21
LDW21-IT649B	21G0108-06	Sediment	07/08/21
LDW21-IT649C	21G0108-07	Sediment	07/08/21
LDW21-IT649E	21G0108-08	Sediment	07/08/21
LDW21-IT669B	21G0108-09	Sediment	07/08/21
LDW21-IT669C	21G0108-10	Sediment	07/08/21
LDW21-IT669E	21G0108-11	Sediment	07/08/21
LDW21-IT654BDUP	21G0108-02DUP	Sediment	07/08/21

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020) and a modified outline of the USEPA National Functional Guidelines (NFG) for High Resolution Superfund Methods Data Review (April 2016). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) Method 1613B

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered not detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and less than or equal to 35.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were within the QC limits for all analytes and labeled compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration results were within the QC limits for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BJG0465-BLK1	08/05/21	OCDD	1.25 ug/Kg	All samples in SDG 21G0108

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated laboratory blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
LDW21-IT654E	OCDD	7.57 ug/Kg	7.57U ug/Kg

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicate Sample Analysis

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
LDW21-IT654BDUP (LDW21-IT654B LDW21-IT654BDUP)	1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDD OCDF OCDD	34.0 (≤25) 174 (≤25) 77.4 (≤25) 127 (≤25)	J (all detects) J (all detects) J (all detects) J (all detects)	A

VIII. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

SRM ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
BJG0465-SRM1	2,3,7,8-TCDD	0 (50-150)	All samples in SDG 21G0108	J (all detects) UJ (all non-detects)	A

For BJG0465-SRM1, although the percent recoveries were severely low for 2,3,7,8-TCDD due to the laboratory using a smaller amount, the associated sample results were qualified as estimated (J/UJ) since the LCS recoveries were within the QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Labeled Compounds

All percent recoveries (%R) for labeled compounds used to quantitate target analytes were within QC limits.

XI. Target Analyte Quantitation

All target analyte quantitation met validation criteria with the following exceptions:

Sample	Analyte	Flag	A or P
All samples in SDG 21G0108	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A
All samples in SDG 21G0108	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U (all non-detects)	A
All samples in SDG 21G0108	All results flagged "X" by the laboratory due to chlorinated diphenyl ether (CDPE) interference.	J (all detects)	A

Sample	Analyte	Finding	Criteria	Flag	A or P
LDW21-IT669C	OCDD	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

Raw data were not reviewed for Stage 2B validation.

XII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

XIII. System Performance

Raw data were not reviewed for Stage 2B validation.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to DUP RPD, standard reference material %R, analytes reported as EMPC, CDPE

interference, and exceeded calibration range, data were qualified as estimated or not detected in ten samples.

Due to laboratory blank contamination, data were qualified as not detected in one sample.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 21G0108**

Sample	Analyte	Flag	A or P	Reason
LDW21-IT654B LDW21-IT654BDUP	1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDD OCDF OCDD	J (all detects) J (all detects) J (all detects) J (all detects)	A	Duplicate sample analysis (RPD)
LDW21-IT654B LDW21-IT654C LDW21-IT654E LDW21-IT649B LDW21-IT649C LDW21-IT649E LDW21-IT669B LDW21-IT669C LDW21-IT669E LDW21-IT654BDUP	2,3,7,8-TCDD	J (all detects) UJ (all non-detects)	A	Standard reference material (%R)
LDW21-IT654B LDW21-IT654C LDW21-IT654E LDW21-IT649B LDW21-IT649C LDW21-IT649E LDW21-IT669B LDW21-IT669C LDW21-IT669E LDW21-IT654BDUP	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A	Target analyte quantitation (EMPC)
LDW21-IT654B LDW21-IT654C LDW21-IT654E LDW21-IT649B LDW21-IT649C LDW21-IT649E LDW21-IT669B LDW21-IT669C LDW21-IT669E LDW21-IT654BDUP	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U (all non-detects)	A	Target analyte quantitation (EMPC)
LDW21-IT654B LDW21-IT654C LDW21-IT654E LDW21-IT649B LDW21-IT649C LDW21-IT649E LDW21-IT669B LDW21-IT669C LDW21-IT669E LDW21-IT654BDUP	All results flagged "X" by the laboratory due to chlorinated diphenyl ether (CDPE) interference.	J (all detects)	A	Target analyte quantitation (CDPE)
LDW21-IT669C	OCDD	J (all detects)	P	Target analyte quantitation (exceeded calibration range)

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification
Summary - SDG 21G0108**

Sample	Analyte	Modified Final Concentration	A or P
LDW21-IT654E	OCDD	7.57U ug/Kg	A

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary -
SDG 21G0108**

No Sample Data Qualified in this SDG

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A	
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	RSD = 20/35% 10% ≤ RCLimits
IV.	Continuing calibration	A	10% ≤ RCLimits
V.	Laboratory Blanks	N	
VI.	Field blanks	N	
VII.	Matrix spike/Matrix spike duplicates	N/SW	CS
VIII.	Laboratory control samples	A/SW	CS
IX.	Field duplicates	N	
X.	Internal standards	A	
XI.	Target analyte quantitation	N	
XII.	Target analyte identification	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	LDW21-IT654B	21G0108-02	Sediment	07/08/21
2	LDW21-IT654C	21G0108-03	Sediment	07/08/21
3	LDW21-IT654E	21G0108-04	Sediment	07/08/21
4	LDW21-IT649B	21G0108-06	Sediment	07/08/21
5	LDW21-IT649C	21G0108-07	Sediment	07/08/21
6	LDW21-IT649E	21G0108-08	Sediment	07/08/21
7	LDW21-IT669B	21G0108-09	Sediment	07/08/21
8	LDW21-IT669C	21G0108-10	Sediment	07/08/21
9	LDW21-IT669E	21G0108-11	Sediment	07/08/21
10	LDW21-IT654BDUP	21G0108-02DUP	Sediment	07/08/21
11				

Notes:

BLANKS - [Signature]					

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes: _____

VALIDATION FINDINGS WORKSHEET

Blanks

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Were all samples associated with a method blank?
- Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y N N/A Was the method blank contaminated?

Blank extraction date: 8/5/21 Blank analysis date: 8/27/21 Associated samples: All
 Conc. units: ng/kg

Compound	Blank ID	Sample Identification							
<u>G</u>	<u>BJE0465-BK1</u> <u>1.25</u>	<u>3</u>	<u>7.5F/U</u>						

Blank extraction date: _____ Blank analysis date: _____
 Conc. units: _____ Associated Samples: _____

Compound	Blank ID	Sample Identification							

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
 All contaminants within five times the method blank concentration were qualified as not detected, "U".

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported RLs

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A
 Y N N/A

Were the correct labeled compound, quantitation ions and relative response factors (RRF) used to quantitate the compound?
 Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	All compounds reported as estimated maximum possible concentration (EMPC) < RL		U/A
		All	All compounds flagged "X" due to chlorinated diphenyl ether interference		Jdets/A
		All	All compounds reported as estimated maximum possible concentration (EMPC) > RL		Jdets/A
		8	G exceeded calibration range		Jdets/P

Comments: See sample calculation verification worksheet for recalculations

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Duwamish AOC4

LDC Report Date: November 4, 2021

Parameters: Polychlorinated Dioxins/Dibenzofurans

Validation Level: Stage 2B

Laboratory: Analytical Resources, Inc., Tukwila, WA

Sample Delivery Group (SDG): 21G0127

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
LDW21-SS668	21G0127-01	Sediment	07/12/21
LDW21-SS667	21G0127-02	Sediment	07/12/21
LDW21-SS651	21G0127-03	Sediment	07/12/21
LDW21-SS633	21G0127-05	Sediment	07/12/21
LDW21-SS645	21G0127-08	Sediment	07/12/21
LDW21-SS656	21G0127-10	Sediment	07/12/21
LDW21-SS647	21G0127-11	Sediment	07/12/21
LDW21-SS634	21G0127-12	Sediment	07/12/21
LDW21-SS668DUP	21G0127-01DUP	Sediment	07/12/21
LDW21-SS647DUP	21G0127-11DUP	Sediment	07/12/21

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020) and a modified outline of the USEPA National Functional Guidelines (NFG) for Organic Superfund Methods Data Review (January 2017). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) Method 1613B

All sample results were subjected to Stage 2B data validation, which comprises an evaluation of quality control (QC) summary results.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered not detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and less than or equal to 35.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were within the QC limits for all analytes and labeled compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration results were within the QC limits for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BJH0186-BLK	08/09/21	1,2,3,4,6,7,8-HpCDD OCDD Total PeCDF Total HxCDD Total HpCDD	0.228 ug/Kg 1.55 ug/Kg 0.0476 ug/Kg 0.180 ug/Kg 0.103 ug/Kg	LDW21-SS668 LDW21-SS667 LDW21-SS651 LDW21-SS633 LDW21-SS645 LDW21-SS656
BJH0645-BLK	08/26/21	OCDD	3.88 ug/Kg	LDW21-SS647 LDW21-SS634

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater than the concentrations found in the associated laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicate Sample Analysis

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
LDW21-SS668DUP (LDW21-SS668 LDW21-SS668DUP)	1,2,3,4,6,7,8-HpCDD OCDF OCDD	53.0 (≤25) 22.8 (≤25) 44.2 (≤25)	J (all detects)	A

VIII. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Internal Standards

All internal standard areas and retention times were within QC limits.

XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria with the following exceptions:

Sample	Analyte	Flag	A or P
All samples in SDG 21G0127	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A
All samples in SDG 21G0127	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U	A

Raw data were not reviewed for Stage 2B validation.

XII. Target Analyte Identification

Raw data were not reviewed for Stage 2B validation.

XIII. System Performance

Raw data were not reviewed for Stage 2B validation.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to DUP RPD and results reported by the laboratory as EMPCs, data were qualified as estimated in ten samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 21G0127**

Sample	Analyte	Flag	A or P	Reason
LDW21-SS668 LDW21-SS668DUP	1,2,3,4,6,7,8-HpCDD OCDF OCDD	J (all detects)	A	Duplicate sample analysis (RPD)
LDW21-SS668 LDW21-SS667 LDW21-SS651 LDW21-SS633 LDW21-SS645 LDW21-SS656 LDW21-SS647 LDW21-SS634 LDW21-SS668DUP LDW21-SS647DUP	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	J (all detects)	A	Target analyte quantitation (EMPC)
LDW21-SS668 LDW21-SS667 LDW21-SS651 LDW21-SS633 LDW21-SS645 LDW21-SS656 LDW21-SS647 LDW21-SS634 LDW21-SS668DUP LDW21-SS647DUP	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	U	A	Target analyte quantitation (EMPC)

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG 21G0127**

No Sample Data Qualified in this SDG

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG 21G0127**

No Sample Data Qualified in this SDG

LDC #: 52274D21

VALIDATION COMPLETENESS WORKSHEET

SDG #: 21G0127

Stage 2B

Laboratory: Analytical Resources, Inc., Tukwila, WA

Date: 10/6/21

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A	
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	#20 = 20/25/0 10% ≤ QC Limits
IV.	Continuing calibration	A	10% ≤ QC Limits
V.	Laboratory Blanks	N	
VI.	Field blanks	N	
VII.	Matrix spike/Matrix spike duplicates / DUP	N/SW	
VIII.	Laboratory control samples / SEM	A	LCS
IX.	Field duplicates	N	
X.	Internal standards	A	
XI.	Target analyte quantitation	SN	
XII.	Target analyte identification	N	
XIII.	System performance	N	
XIV.	Overall assessment of data	A	

Note: A = Acceptable
N = Not provided/applicable
SW = See worksheet

ND = No compounds detected
R = Rinsate
FB = Field blank

D = Duplicate
TB = Trip blank
EB = Equipment blank

SB=Source blank
OTHER:

	Client ID	Lab ID	Matrix	Date
1	LDW21-SS668	21G0127-01	Sediment	07/12/21
2	LDW21-SS667	21G0127-02	Sediment	07/12/21
3	LDW21-SS651	21G0127-03	Sediment	07/12/21
4	LDW21-SS633	21G0127-05	Sediment	07/12/21
5	LDW21-SS645	21G0127-08	Sediment	07/12/21
6	LDW21-SS656	21G0127-10	Sediment	07/12/21
7	LDW21-SS647	21G0127-11	Sediment	07/12/21
8	LDW21-SS634	21G0127-12	Sediment	07/12/21
9	LDW21-SS668DUP	21G0127-01DUP	Sediment	07/12/21
10	LDW21-SS647DUP	21G0127-11DUP	Sediment	07/12/21
11				

Notes:

1	#110186				
2	#110645				

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes: _____

VALIDATION FINDINGS WORKSHEET

Blanks

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Were all samples associated with a method blank?
- N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- N N/A Was the method blank contaminated?

Blank extraction date: 8/7/21

Blank analysis date: 8/4/21

Associated samples: 1-6

Conc. units: 15/Kg

Compound	Blank ID	Sample Identification							
F	0186-BK1								
F	0.228								
F	1.55								
W	0.0476								
T	0.180								
U	0.103								

Blank extraction date: 8/26/21

Blank analysis date: 9/15/21

Associated Samples: 7-8

Conc. units: 12/Kg

Compound	Blank ID	Sample Identification							
F	BH0645-BK1								
F	3.88								

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
 All contaminants within five times the method blank concentration were qualified as not detected, "U".

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported RLs

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A
 Y N N/A

Were the correct labeled compound, quantitation ions and relative response factors (RRF) used to quantitate the compound?
 Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	All compounds reported as estimated maximum possible concentration (EMPC) > RL		Jdets/A
		All	All compounds reported as estimated maximum possible concentration (EMPC) < RL		U/A

Comments: See sample calculation verification worksheet for recalculations

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Duwamish AOC4

LDC Report Date: November 4, 2021

Parameters: Polychlorinated Dioxins/Dibenzofurans

Validation Level: Stage 4

Laboratory: Analytical Resources, Inc., Tukwila, WA

Sample Delivery Group (SDG): 21G0138

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
LDW21-IT662B	21G0138-02	Sediment	07/13/21
LDW21-IT662BDL	21G0138-02DL	Sediment	07/13/21
LDW21-IT662C	21G0138-03	Sediment	07/13/21
LDW21-IT662E	21G0138-04	Sediment	07/13/21
LDW21-IT659B	21G0138-05	Sediment	07/13/21
LDW21-IT659C	21G0138-06	Sediment	07/13/21
LDW21-IT659E	21G0138-07	Sediment	07/13/21
LDW21-IT658B	21G0138-08	Sediment	07/13/21
LDW21-IT658C	21G0138-09	Sediment	07/13/21
LDW21-IT658E	21G0138-10	Sediment	07/13/21
LDW21-IT657B	21G0138-11	Sediment	07/13/21
LDW21-IT657C	21G0138-12	Sediment	07/13/21
LDW21-IT648B	21G0138-13	Sediment	07/13/21
LDW21-IT648C	21G0138-14	Sediment	07/13/21
LDW21-IT662BDUP	21G0138-02DUP	Sediment	07/13/21
LDW21-IT662BDLDUP	21G0138-02DL DUP	Sediment	07/13/21

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with the Final Lower Duwamish Waterway Quality Assurance Project Plan for Remedial Design of Upper Reach: Pre-Design Investigation (May 2020) and a modified outline of the USEPA National Functional Guidelines (NFG) for Organic Superfund Methods Data Review (January 2017). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Dioxins/Dibenzofurans by Environmental Protection Agency (EPA) Method 1613B

All sample results were subjected to Stage 4 data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The analyte was analyzed for and positively identified by the laboratory; however the analyte should be considered not detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25%.

The static resolving power was at least 10,000 (10% valley definition).

III. Initial Calibration and Initial Calibration Verification

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all analytes and labeled compounds.

The ion abundance ratios for all PCDDs/PCDFs were within method and validation criteria.

The minimum S/N ratio was greater than or equal to 2.5 for each analyte and greater than or equal to 10 for each labeled compound.

The percent differences (%D) of the initial calibration verification (ICV) standard were within the QC limits for all analytes and labeled compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

All of the continuing calibration percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were less than or equal to 20.0% for all analytes and less than or equal to 30.0% for labeled compounds.

The ion abundance ratios for all PCDDs and PCDFs were within method and validation criteria.

The minimum S/N ratio was greater than or equal to 10 for each analyte and labeled compound.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks with the following exceptions:

Blank ID	Extraction Date	Analyte	Concentration	Associated Samples
BJH0150-BLK	08/24/21	1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDD OCDD Total HxCDD	0.0955 ug/Kg 0.180 ug/Kg 2.03 ug/Kg 0.159 ug/Kg	All samples in SDG 21G0138

Sample concentrations were compared to concentrations detected in the laboratory blanks. The sample concentrations were either not detected or were significantly greater than the concentrations found in the associated laboratory blanks with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
LDW21-IT662E	1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDD	0.697 ug/Kg 1.72 ug/Kg	0.697U ug/Kg 1.72U ug/Kg
LDW21-IT659E	1,2,3,4,6,7,8-HpCDD	2.10 ug/Kg	2.10U ug/Kg

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicate Sample Analysis

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Analyte	RPD (Limits)	Flag	A or P
LDW21-IT662BDUP (LDW21-IT662B LDW21-IT662BDUP)	1,2,3,4,6,7,8-HpCDD OCDD	65.2 (≤ 25) 161 (≤ 25)	J (all detects) J (all detects)	A
LDW21-IT662BDLDUP (LDW21-IT662BDL LDW21-IT662BDLDUP)	1,2,3,4,6,7,8-HpCDD OCDF OCDD	70.7 (≤ 25) 28.6 (≤ 25) 170 (≤ 25)	J (all detects) J (all detects) J (all detects)	A

VIII. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) were analyzed as required by the method. Percent recoveries (%R) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Internal Standards

All internal standard areas and retention times were within QC limits.

XI. Target Analyte Quantitation

All target analyte quantitations met validation criteria with the following exceptions:

Sample	Analyte	Flag	A or P
All samples in SDG 21G0138	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	U	A
All samples in SDG 21G0138	All analytes flagged "X" due to chlorinated diphenyl either interference	J (all detects)	A
All samples in SDG 21G0138	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	J (all detects)	A

Sample	Analyte	Finding	Criteria	Flag	A or P
LDW21-IT659B LDW21-IT658B	OCDD	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

XII. Target Analyte Identification

All target analyte identifications met validation criteria.

XIII. System Performance

The system performance was acceptable.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

In the case where more than one result was reported for an individual sample, the least technically acceptable results were deemed not reportable as follows:

Sample	Analyte	Reason	Flag	A or P
LDW21-IT662B	1,2,3,4,6,7,8-HpCDD OCDD	Results exceeded calibration range.	Not reportable	-
LDW21-IT662BDL	All analytes except 1,2,3,4,6,7,8-HpCDD OCDD	Results exceeded calibration range.	Not reportable	-

Due to DUP RPD, results reported by the laboratory as EMPCs, CDPE interference, and results exceeding calibration range, data were qualified as estimated in sixteen samples.

Due to laboratory blank contamination, data were qualified as not detected in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable.

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Data Qualification Summary - SDG 21G0138**

Sample	Analyte	Flag	A or P	Reason
LDW21-IT662BDL LDW21-IT662BDLDUP	1,2,3,4,6,7,8-HpCDD OCDD	J (all detects) J (all detects)	A	Duplicate sample analysis (RPD)
LDW21-IT662B LDW21-IT662BDL LDW21-IT662C LDW21-IT662E LDW21-IT659B LDW21-IT659C LDW21-IT659E LDW21-IT658B LDW21-IT658C LDW21-IT658E LDW21-IT657B LDW21-IT657C LDW21-IT648B LDW21-IT648C LDW21-IT662BDUP LDW21-IT662BDLDUP	All analytes reported as estimated maximum possible concentration (EMPC) and greater than the reporting limit.	U	A	Target analyte quantitation (EMPC)
LDW21-IT662B LDW21-IT662BDL LDW21-IT662C LDW21-IT662E LDW21-IT659B LDW21-IT659C LDW21-IT659E LDW21-IT658B LDW21-IT658C LDW21-IT658E LDW21-IT657B LDW21-IT657C LDW21-IT648B LDW21-IT648C LDW21-IT662BDUP LDW21-IT662BDLDUP	All analytes flagged "X" due to chlorinated diphenyl either interference	J (all detects)	A	Target analyte quantitation (CPDE interference)
LDW21-IT662B LDW21-IT662BDL LDW21-IT662C LDW21-IT662E LDW21-IT659B LDW21-IT659C LDW21-IT659E LDW21-IT658B LDW21-IT658C LDW21-IT658E LDW21-IT657B LDW21-IT657C LDW21-IT648B LDW21-IT648C LDW21-IT662BDUP LDW21-IT662BDLDUP	All analytes reported as estimated maximum possible concentration (EMPC) and less than the reporting limit.	J (all detects)	A	Target analyte quantitation (EMPC)
LDW21-IT659B LDW21-IT658B	OCDD exceeded calibration range	J (all detects)	P	Target analyte quantitation (CPDE interference)

Sample	Analyte	Flag	A or P	Reason
LDW21-IT662B	1,2,3,4,6,7,8-HpCDD OCDD	Not reportable	-	Overall assessment of data
LDW21-IT662BDL	All analytes except 1,2,3,4,6,7,8-HpCDD OCDD	Not reportable	-	Overall assessment of data

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Laboratory Blank Data Qualification
Summary - SDG 21G0138**

Sample	Analyte	Modified Final Concentration	A or P
LDW21-IT662E	1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDD	0.697U ug/Kg 1.72U ug/Kg	A
LDW21-IT659E	1,2,3,4,6,7,8-HpCDD	2.10U ug/Kg	A

**Duwamish AOC4
Polychlorinated Dioxins/Dibenzofurans - Field Blank Data Qualification Summary -
SDG 21G0138**

No Sample Data Qualified in this SDG

LDC #: 52274E21

VALIDATION COMPLETENESS WORKSHEET

SDG #: 21G0138

Stage 4

Laboratory: Analytical Resources, Inc., Tukwila, WA

Date: 10/26/17

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A	
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	RSD < 20/35/70. 1σV ≤ QC limits
IV.	Continuing calibration	A	COV ≤ QC limits
V.	Laboratory Blanks	W	
VI.	Field blanks	N	
VII.	Matrix spike/Matrix spike duplicates / BDP	N/W	
VIII.	Laboratory control samples / SW	A	LCS
IX.	Field duplicates	N	
X.	Internal standards	A	
XI.	Target analyte quantitation	W	
XII.	Target analyte identification	A	
XIII.	System performance	A	
XIV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	LDW21-IT662B	21G0138-02	Sediment	07/13/21
2	LDW21-IT662BDL	21G0138-02DL	Sediment	07/13/21
3	LDW21-IT662C	21G0138-03	Sediment	07/13/21
4	LDW21-IT662E	21G0138-04	Sediment	07/13/21
5	LDW21-IT659B	21G0138-05	Sediment	07/13/21
6	LDW21-IT659C	21G0138-06	Sediment	07/13/21
7	LDW21-IT659E	21G0138-07	Sediment	07/13/21
8	LDW21-IT658B	21G0138-08	Sediment	07/13/21
9	LDW21-IT658C	21G0138-09	Sediment	07/13/21
10	LDW21-IT658E	21G0138-10	Sediment	07/13/21
11	LDW21-IT657B	21G0138-11	Sediment	07/13/21
12	LDW21-IT657C	21G0138-12	Sediment	07/13/21
13	LDW21-IT648B	21G0138-13	Sediment	07/13/21
14	LDW21-IT648C	21G0138-14	Sediment	07/13/21
15	LDW21-IT662BDUP	21G0138-02DUP	Sediment	07/13/21

LDC #: 52274E21

VALIDATION COMPLETENESS WORKSHEET

Date: 07/13/21

SDG #: 21G0138

Stage 4

Page: 5 of 7

Laboratory: Analytical Resources, Inc., Tukwila, WA

Reviewer: [Signature]

2nd Reviewer: _____

METHOD: HRGC/HRMS Polychlorinated Dioxins/Dibenzofurans (EPA Method 1613B)

	Client ID	Lab ID	Matrix	Date
16	LDW21-IT662BDLDUP	21G0138-02DL DUP	Sediment	07/13/21
17				
18				
19				

Notes:

<u>BH0150-2K</u>				

Method: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	√			
Cooler temperature criteria were met.	√			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	√			
Were the retention time windows established for all homologues?	√			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$?	√			
Is the static resolving power at least 10,000 (10% valley definition)?	√			
Was the mass resolution adequately check with PFK?	√			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	√			
III. Initial calibration and Initial calibration verification				
Was the initial calibration performed at 5 concentration levels?	√			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled compounds and $\leq 35\%$ for unlabeled compounds?	√			
Did all calibration standards meet the Ion Abundance Ratio criteria?	√			
Was the signal to noise ratio for each target compound and labeled compound ≥ 10 ?	√			
Was an initial calibration verification (ICV) standard analyzed after each initial calibration for each instrument?	√			
Were all ICV concentrations for the unlabeled and labeled compounds within QC limits?	√			
IV. Continuing calibration				
Was a continuing calibration performed at the beginning of each 12-hour period?	√			
Were all continuing calibration concentrations for the unlabeled and labeled compounds within QC limits?	√			
Did all continuing calibration standards meet the Ion Abundance Ratio criteria?	√			
V. Blanks				
Was a method blank associated with every sample in this SDG?	√			
Was a method blank performed for each matrix and whenever a sample extraction was performed?	√			
Was there contamination in the method blanks?	√	⊕		
VI. Field blanks				
Were field blanks identified in this SDG?		√		
Were target compounds detected in the field blanks?			√	
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?		√		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			√	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Laboratory control samples				
Was an LCS analyzed per extraction batch?	√			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	√			
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?		√		
Were target compounds detected in the field duplicates?			√	
X. Labeled Compounds				
Were labeled compounds within QC limits?	√	0		
Was the minimum S/N ratio of all labeled compound peaks ≥ 10 ?	√			
XI. Compound quantitation				
Did the laboratory LOQs/RLs meet the QAPP LOQs/RLs?	√			
Were the correct labeled compound, quantitation ion and relative response factor (RRF) used to quantitate the compound?	√			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	√			
XII. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	√			
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	√			
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	√			
Did selected ion current profile (SICP) contain all characteristic ions listed in Method 1613B, Table 8?	√			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?		√		
Was the signal to noise ratio for each target compound ≥ 2.5 and ≥ 10 for the labeled compound?	√			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	√			
For PCDF identification, was any signal ($S/N \geq 2.5$, at \pm seconds RT) detected in the corresponding PCDPE channel?			√	
Was an acceptable lock mass recorded and monitored?	√			
XIII. System performance				
System performance was found to be acceptable.	√			
XIV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	√			

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes: _____

VALIDATION FINDINGS WORKSHEET
Blanks

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Were all samples associated with a method blank?
- Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y N N/A Was the method blank contaminated?

Blank extraction date: 8/21/21 Blank analysis date: 9/11/21 Associated samples: All

Conc. units: ng/kg

Compound	Blank ID	Sample Identification							
0	0150-BA	+	-						
0	0.0955	0.69T/U							
F	0.180	1.73/U	2.10/U						
+	203								
T	0.159								

Blank extraction date: _____ Blank analysis date: _____
Conc. units: _____ Associated Samples: _____

Compound	Blank ID	Sample Identification							

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported RLs

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were the correct labeled compound, quantitation ions and relative response factors (RRF) used to quantitate the compound?
 Y N N/A Compound quantitation and RLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	All compounds reported as estimated maximum possible concentration (EMPC) < RL		U/A
		All	All compounds flagged "X" due to chlorinated diphenyl ether interference		Jdets/A
		All	All compounds reported as estimated maximum possible concentration (EMPC) > RL		Jdets/A
		5, 8	G exceeded calibration range		Jdets/P

Comments: See sample calculation verification worksheet for recalculations

VALIDATION FINDINGS WORKSHEET

Initial Calibration Calculation Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

$$RRF = (A_x)(C_{is}) / (A_{is})(C_x)$$

average RRF = sum of the RRFs/number of standards

$$\%RSD = 100 * (S/X)$$

A_x = Area of compound,

C_x = Concentration of compound,

S = Standard deviation of the RRFs,

A_{is} = Area of associated internal standard

C_{is} = Concentration of internal standard

X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
				Average RRF (initial)	Average RRF (initial)	RRF (CS3 std)	RRF (CS3 std)	%RSD	%RSD
1	10A	8/11/21	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.107593	1.107593	1.083206	1.083146	3.6	3.6
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	0.9202875	0.9202874	0.9085786	0.90839	3.1	3.1
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.00898	1.00898	1.005616	1.005605	1.0	1.0
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)	1.068088	1.068088	1.051009	1.051062	6.6	6.6
			OCDF (¹³ C-OCDF)	1.446901	1.446901	1.440564	1.44059	5.7	5.7
2			2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)						
			OCDF (¹³ C-OCDF)						
3			2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)						
			OCDF (¹³ C-OCDF)						

Comments: Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					Conc (CC)	Conc (CC)	%D	%D
1	21091113	9/11/21	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.107593	0.994543	0.99453	10.2	10.2
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	0.9202875	0.9806681	0.98068	6.6	6.6
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.00898	1.002200	1.00204	0.7	0.7
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)	1.068088	1.1526340	1.15277	7.9	7.9
			OCDF (¹³ C-OCDF)	1.446901	1.3084910	1.30845	9.6	9.6
2	21091123	9/12/21	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.107593	1.0171930	1.01696	8.2	8.2
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	0.9202875	0.9490668	0.94909	3.1	3.1
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.00898	1.0008820	1.000877	0.8	0.8
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)	1.068088	1.1490000	1.14885	7.6	7.6
			OCDF (¹³ C-OCDF)	1.446901	1.2340690	1.23404	14.7	14.7
3	21091317	9/14/21	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.107593	1.0306810	1.030764	6.9	6.9
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	0.9202875	0.9754769	0.97539	6.0	6.0
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.00898	0.9795780	0.97939	2.9	2.9
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)	1.068088	1.1611420	1.161191	8.7	8.7
			OCDF (¹³ C-OCDF)	1.446901	1.2305840	1.23037	15.0	15.0

Comments: Refer to Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method 1613B)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate (if applicable) were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * \frac{SSC}{SA}$ Where: SSC = Spiked sample concentration
SA = Spike added

RPD = $\frac{|LCS - LCSD|}{LCS + LCSD} * 2$ LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: BH0150-BS1

Compound	Spike Added (NS/K)		Spiked Sample Concentration (NS/K)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	20.1	NA	101	101				
1,2,3,7,8-PeCDD	100	↓	110	↓	110	110				
1,2,3,4,7,8-HxCDD	↓	↓	94.4	↓	94.4	94.4				
1,2,3,4,7,8,9-HpCDF	↓	↓	106	↓	106	106				
OCDF	200	↓	181	↓	90.7	90.7				

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

