



DATA VALIDATION REPORT

SEATTLE IRON AND METALS - DUST MONITORING PHASE II OCT, NOV, DEC 2020

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Approved for Release:

A handwritten signature in black ink, appearing to read "Christine Ransom", written over a horizontal line.

Christine Ransom
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PROJECT NARRATIVE

Basis for the Data Validation

This report summarizes the results of full validation (EPA Stage 4) performed on PUF cartridge and associated quality control sample data for the Seattle Iron and Metals Dust monitoring project. A complete list of samples is provided in the **Sample Index**.

Analyses were performed by ALS Life Sciences, Burlington, Ontario. The analytical methods and EcoChem project chemists are listed in the following table:

ANALYSIS	METHOD	PRIMARY REVIEW	SECONDARY REVIEW
Dioxins/Furans	EPA TO-09A	E. Clayton	C. Ransom
PCB Congeners	EPA 1668C		

The data were reviewed using guidance and quality control criteria documented in the analytical methods; *Seattle Iron & Metals Dust Monitoring Plan: Phase II* (June 2020); *National Functional Guidelines for Organic Superfund Methods Data Review* (USEPA 2017); and *National Functional Guidelines for High Resolution Superfund Methods Data Review* (USEPA 2016).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. Data that have been rejected are flagged with (R). Rejected data should not be used for any purpose. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Validation criteria are included as **Appendix A**. The qualified data summary table (QDST) is included as **Appendix B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted.

DATA VALIDATION REPORT

Seattle Iron and Metals - Dust Monitoring Phase II Oct, Nov, Dec 2020 Dioxin/Furan Compounds by EPA TO-09A

This report documents the review of analytical data from the analyses of PUF cartridge samples and the associated laboratory quality control (QC) samples. Samples were analyzed by ALS Life Sciences, Burlington, Ontario. Refer to the **Sample Index** for a complete list of samples.

SDG	NUMBER OF SAMPLES AND MATRIX	VALIDATION LEVEL
L2541483	5 PUF Composites	Stage 4

DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

All sample IDs and results reported in the electronic data deliverable (EDD) were verified (100% verification) by comparing the EDD to the hardcopy laboratory data package. Ten percent (10%) of the laboratory QC results were also verified.

The collection dates and times were missing from the EDD for all samples. Dates were added during validation. The values for the final week of collection were used for the composites.

TECHNICAL DATA VALIDATION

The quality control (QC) requirements that were reviewed are listed in the following table.

1	Sample Receipt, Preservation, and Holding Times	1	Laboratory Control Samples
✓	System Performance and Resolution Checks	1	Matrix Spike/Matrix Spike Duplicates (MS/MSD)
✓	Initial Calibration (ICAL)	1	Field Duplicates
1	Calibration Verification (CCAL)	✓	Reporting Limits and Sample Quantitation
1	Laboratory Blanks	✓	Target Analyte List
1	Field Standard Labeled Compounds	2	Compound Identification
1	Extraction Standard Labeled Compounds	1	Calculation Verification
✓	Cleanup Standards		

✓ *Method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.*

1 *Quality control results are discussed below, but no data were qualified.*

2 *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

Sample Receipt, Preservation, and Holding Times

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of $\leq 6^{\circ}\text{C}$.

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of $\leq 6^{\circ}\text{C}$. Weekly samples were stored frozen until samples for all 4 individual sampling events for the month were collected. These were then composited into a single sample. A holding time of one year was used to evaluate the sample extraction and analysis.

SDG L2541483: One sample cooler temperature was greater than the upper control limit at 10.8°C . This outlier did not impact data quality; no data were qualified.

For the samples received 12/16/20, the samples were logged-in with identities supplied by the client, rather than the ones written on the chain-of-custody.

Calibration Verification

All calibration verification (CCAL) criteria were met. The CCAL were analyzed at the beginning of each sequence as required. All compound concentrations fell within the acceptance limits specified in the method. All ion ratios were acceptable. The S/N ratio was greater than 10, as required, for all compounds. All relative retention times for all target compounds met the required criteria.

Laboratory Blanks

To assess the impact of any blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration reported in the blank. If a contaminant is reported in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U-7). No action is taken if the sample result is greater than the action level, or for non-detected results. The laboratory assigned EMPC-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks (media blanks and reagent blanks) were analyzed at the appropriate frequency. For detected results, the highest concentrations from the two blanks were used to evaluate field sample data. Several target analytes were detected in the method blanks; however, no results required qualification.

Field Standard Labeled Compounds

Five labeled compounds were added to the sample cartridges prior to sampling. The percent recovery (%R) values method specified control limits are 70-130%. These surrogates are used to evaluate the sampling system and are not used for sample quantitation. No action was taken unless three or more of the compounds were outside of the control limits.

Extraction Standard Labeled Compounds

Isotope-stable labeled compounds were added to each field and QC sample. All recoveries were within the method criteria of 40-130% (25-130% for hepta and octa-chlorinated compounds).

Laboratory Control Samples

Laboratory control samples were analyzed at the proper frequency. All recovery values were within the control limits.

Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSD) were not analyzed. These are not required by the method. Accuracy was assessed using labeled compound and LCS recoveries. Precision within an analytical batch could not be evaluated.

Field Duplicates

No field duplicates were collected.

Compound Identification

The laboratory reported EMPC or "estimated maximum possible concentrations" values for one or more of the target analytes in all samples. An EMPC value was reported when a peak was detected but did not meet identification criteria as required by the method; therefore, the result cannot be considered as positive identification for the analyte. The lab flagged these results "R". All EMPC results were qualified as not-detected (U-25) at the reported concentrations.

The laboratory uses DB5 MS column, which provides adequate resolution of the TCDF isomers as indicated by the acceptable peak to valley ratios. There were no positive results for 2,3,7,8-TCDF in the field samples. No second column confirmation was necessary.

Calculation Verification

Several results were verified by recalculation from the raw data. No calculation or transcription errors were found.

OVERALL ASSESSMENT

As determined by this evaluation, the laboratory performed the specified analytical method. Accuracy was acceptable as demonstrated by the labeled compound and laboratory control sample recoveries. Precision within an analytical batch could not be evaluated.

Detection limits were elevated based on ion ratio outliers.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT

Seattle Iron and Metals - Dust Monitoring Phase II Oct, Nov, Dec 2020 PCB Congeners by EPA 1668C

This report documents the review of analytical data from the analysis of PUF cartridge samples and the associated laboratory quality control (QC) samples. Samples were analyzed by ALS Life Sciences, Burlington, Ontario. Refer to the Sample Index for a complete list of samples.

SDG	NUMBER OF SAMPLES AND MATRIX	VALIDATION LEVEL
L2541483	5 PUF Composites	Stage 4

DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

The collection dates and times were missing from the EDD for all samples. Dates were added during validation. The values for the final week of collection were used for the composites.

TECHNICAL DATA VALIDATION

This report documents the review of analytical QC requirements as listed in the following table.

1	Sample Receipt, Preservation, and Holding Times	1	Laboratory Control Samples
✓	Instrument Performance Check	1	Matrix Spike/Matrix Spike Duplicates (MS/MSD)
✓	Initial Calibration (ICAL)	1	Field Duplicates
✓	Continuing Calibration Verification (CCV)	✓	Reporting Limits and Sample Quantitation
1	Laboratory Blanks	✓	Target Analyte List
✓	Field Standard Labeled Compounds	2	Compound Identification
✓	Extraction Standard Labeled Compounds	✓	Compound Quantitation
✓	Cleanup Standards	1	Calculation Verification

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted.

Data qualifiers were issued as discussed below.

Sample Receipt, Preservation, and Holding Times

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of $\leq 6^{\circ}\text{C}$. Weekly samples were stored frozen until 4 weeks' worth of samples

were collected. These were then composited into a single sample. A holding time of one year was used to evaluate the sample extraction and analysis.

SDG L2541483: One sample cooler temperature was greater than the upper control limit, at 10.8°C. These outliers did not impact data quality; no data were qualified.

For the samples received 12/16/20, the samples were logged-in with identities supplied by the client, rather than the ones written on the chain-of-custody.

Laboratory Blanks

To assess the impact of any blank contaminant on the reported sample results, an action level is established at five times (5x) the concentration reported in the blank. If a contaminant is reported in an associated field sample and the concentration is less than the action level, the result is qualified as not detected (U-7). No action is taken if the sample result is greater than the action level, or for non-detected results. The laboratory assigned EMPC-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks (media blanks and reagent blanks) were analyzed at the appropriate frequency. For detected results, the highest concentrations from the two blanks were used to evaluate field sample data. Although several target analytes were detected in each blank for every batch, all associated detected results were greater than the 5x action levels. No qualification of data was required.

Laboratory Control Samples

Laboratory control samples (LCS) were analyzed with each batch. All recoveries were within the control limits of 70%-130%.

Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSD) were not analyzed. These are not required by the method. Accuracy was assessed using labeled compound and laboratory control sample recoveries. Precision could not be assessed.

Field Duplicates

No field duplicates were submitted.

Compound Identification

The laboratory reported EMPC or "estimated maximum possible concentrations" values for one or more of the target analytes in all samples. An EMPC value was reported when a peak was detected but did not meet identification criteria as required by the method; therefore, the result cannot be considered as positive identification for the analyte. The lab flagged these results "R". All EMPC results were qualified as not-detected (U-25) at the reported concentrations.

Calculation Verification

Several results were verified by recalculation from the raw data. No transcription or calculation errors were found.

OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory performed the specified analytical method. With the exceptions noted above, accuracy was acceptable as demonstrated by the labeled compound and LCS recoveries. Precision within the batches could not be assessed.

Detection limits were elevated due to ion ratio outliers.

All data, as qualified, are acceptable for use.



APPENDIX A

DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

DATA VALIDATION QUALIFIER CODES **Based on National Functional Guidelines**

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents the approximate concentration.
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.
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.

The following is an EcoChem qualifier that may also be assigned during the data review process:

DNR	Do not report; a more appropriate result is reported from another analysis or dilution.
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DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times
Instrument Performance	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)
	5A	Initial Calibration (RF, %RSD, r^2)
	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) ¹ where appropriate
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) ¹ where appropriate
Blank Contamination	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)
	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) ¹ for negative instrument blanks
Precision and Accuracy	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) ¹ where appropriate
	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)
	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) ¹ where appropriate
	12	Reference Material Use bias flags (H,L) ¹ where appropriate
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) ¹ where appropriate
Interferences	16	ICP/ICP-MS Serial Dilution Percent Difference
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) ¹ where appropriate
	19	Internal Standard Performance (i.e., area, retention time, recovery)
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)
Identification and Quantitation	2	Chromatographic pattern in sample does not match pattern of calibration standard
	3	2 nd column confirmation (RPD or %D)
	4	Tentatively Identified Compound (TIC) (associated with NJ only)
	20	Calibration Range or Linear Range Exceeded
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)
Miscellaneous	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re-extractions, etc. Associated with "R" and "DNR" only)
	14	Other (See DV report for details)
	26	Method QC information not provided

¹H = high bias indicated

L = low bias indicated

Dioxin/Furan Analysis by HRMS
(Based on Dioxin NFG 2011 and Methods EPA 1613B and SW-846 8290)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids ≤ 6°C & in the dark Tissues < -10°C & in the dark Preservation Aqueous: If Cl ₂ is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG ⁽¹⁾ Method ⁽²⁾	J(pos)/R(ND) if thiosulfate not added if Cl ₂ present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C	1	EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG ⁽¹⁾ Method ⁽²⁾	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H ₂ O is 7 days.
Instrument Performance					
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG ⁽¹⁾ Method ⁽²⁾	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG ⁽¹⁾ Method ⁽²⁾	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG ⁽¹⁾ Method ⁽²⁾	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG ⁽¹⁾ Method ⁽²⁾	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration Selectivity	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG ⁽¹⁾ Method ⁽²⁾	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

Dioxin/Furan Analysis by HRMS
(Based on Dioxin NFG 2011 and Methods EPA 1613B and SW-846 8290)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Performance (continued)					
Initial Calibration (Minimum 5 stds.) Stability	%RSD < 20% for native compounds %RSD < 30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG ⁽¹⁾ Method ⁽²⁾	J(pos) natives if %RSD > 20%	5A	EcoChem PJ, see TM-05, Rev. 2
	Absolute RT of ¹³ C ₁₂ -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG ⁽¹⁾ Method ⁽²⁾	Narrate, no action		
Continuing Calibration (Prior to each 12 hr. shift) Sensitivity	S/N ratio for CS3 standard > 10	NFG ⁽¹⁾ Method ⁽²⁾	If <10, elevate Det. Limit or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) Selectivity	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG ⁽¹⁾ Method ⁽²⁾	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05
Continuing Calibration (Prior to each 12 hr. shift) Stability	%D +/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG ⁽¹⁾ Method ⁽²⁾	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND) if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75% J(pos)/R(ND) if %D > 75%	5B (H,L) ³	
	Absolute RT of ¹³ C ₁₂ -1234-TCDD and ¹³ C ₁₂ -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG ⁽¹⁾ Method ⁽²⁾	Narrate, no action	5B	EcoChem PJ, see TM-05
Blank Contamination					
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG ⁽¹⁾ Method ⁽²⁾	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed #2 - Review FB, qualify as needed
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL		U(pos) if result is < 5X action level.	6	

**Dioxin/Furan Analysis by HRMS
(Based on Dioxin NFG 2011 and Methods EPA 1613B and SW-846 8290)**

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Precision and Accuracy					
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias PJ if only one %R outlier	8 (H,L) ³	No action if only one spike %R is outside criteria. No action if parent concentration is > 4x the amount spiked. Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits or Limits from Table 6 of 1613B	NFG ⁽¹⁾ Method ⁽²⁾	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) ³	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method ⁽²⁾ EcoChem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG ⁽¹⁾ Method ⁽²⁾	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) ³	
Field Duplicates	Solids: RPD < 50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD < 35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment

**Dioxin/Furan Analysis by HRMS
(Based on Dioxin NFG 2011 and Methods EPA 1613B and SW-846 8290)**

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Compound ID and Calculation					
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio >2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG ⁽¹⁾ Method ⁽²⁾	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG ⁽¹⁾ Method ⁽²⁾	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG ⁽¹⁾ Method ⁽²⁾	J(pos)/UJ(ND) if present	23	See TM-16
	Lock masses must not deviate $\pm 20\%$ from values in Table 8 of 1613B	Method ⁽²⁾	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG ⁽¹⁾ Method ⁽²⁾	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. EcoChem PJ, see TM-05
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Deliverable (EDD)					
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re-extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

¹ National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

² Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

² EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

³ NFG 2013 suggests using "+" / "-" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.

PCB Congener Analysis by HRMS
(Based on EPA DV Guidance¹ and Method EPA 1668C)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}\text{C}$ & in the dark Tissues $< -10^{\circ}\text{C}$ & in the dark Preservation Aqueous: If Cl_2 is present Thiosulfate must be added and if needed adjust pH to 2 - 3 (drinking water requirement)	EPA ⁽¹⁾ Method ⁽²⁾	J(pos)/R(ND) if thiosulfate not added if Cl_2 present and J(pos)/UJ(ND) if pH not adjusted; J(pos)/UJ(ND) if temp $> 20^{\circ}\text{C}$	1	Note: EPA DV guidance documents use $< 4^{\circ}\text{C}$, method uses $\leq 6^{\circ}\text{C}$. Info in EcoChem TM-05 also generally applies.
Holding Time	If properly stored, 1 year prior to extraction. If extracts properly stored ($< -10^{\circ}\text{C}$ & in dark), 1 year from extraction to analysis.	EPA ⁽¹⁾ Method ⁽²⁾	If not properly stored or HT exceeded: J(pos)/UJ(ND)	1	May be dictated by QAPP Info in EcoChem TM-05 also generally applies
Instrument Performance					
Mass Resolution (Tuning)	$\geq 10,000$ resolving power at m/z 330.9792 < 5 ppm deviation from each m/z listed in Table 7 of method. Analyzed prior to ICAL and at the beginning and end of each 12 hr. shift	EPA ⁽¹⁾ Method ⁽²⁾	R all analytes in all samples associated with a failed tune	24	PFK (Perfluorokerosene) tuning compound
Column Resolution	Mix of all 209 PCBs run prior to each ICAL/12 hours RT of PCB209 must be > 55 min PCB156 & 157 must coelute w/in 2 sec PCB34 & 23 and PCB187 & 182 must be resolved where $(x/y)*100\% < 40\%$ $x = \text{ht of valley}$ and $y = \text{ht of shortest peak}$ RRT of all congeners must fall within the range in Table 2 of the method	EPA ⁽¹⁾ Method ⁽²⁾	If criteria are not met, review sample chromatograms to determine if sample results are negatively impacted. If so, discuss with client for possible reanalyses, or J(pos) all data.	24	Criteria are for SPB-octyl column. If different column used, see Section 6.9.1.2 of method. Appendix A provides info for DB-1 column
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled congeners in CS1 std.	EPA ⁽¹⁾ Method ⁽²⁾	If < 10 , elevate Det. Limit or R(ND)	5A	
Initial Calibration Selectivity	Ion Abundance ratios within QC limits (Table 8 of Method 1668C)	EPA ⁽¹⁾ Method ⁽²⁾	If ion ratios are out for a given congener in 2 or more standards in ICAL, J(pos) results for that congener in all samples	5A	Professional judgement. The info in EcoChem TM-05 also generally applies
Initial Calibration (Minimum 5 stds.) Stability	%RSD $< 20\%$ for congeners listed in Table 3 of method RRT of all congeners must meet Table 2 of method	EPA ⁽¹⁾ Method ⁽²⁾	J(pos) natives if %RSD $> 20\%$ RRT outliers: narrate, no action	5A	RRT outliers: professional judgement. The info in EcoChem TM-05 also generally applies
Continuing Calibration (Prior to each 12 hr. shift) Sensitivity	S/N ratio for CS3 standard > 10	EPA ⁽¹⁾ Method ⁽²⁾	If < 10 , elevate Det. Limit to lowest calibration or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) Selectivity	Ion Abundance ratios within QC limits (Table 8 of Method 1668C)	EPA ⁽¹⁾ Method ⁽²⁾	No action if %D acceptable, review sample ion ratios, U(pos) if ion ratio outside limits	5B	Professional judgement. The info in EcoChem TM-05 also generally applies.

PCB Congener Analysis by HRMS
(Based on EPA DV Guidance¹ and Method EPA 1668C)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Continuing Calibration (Prior to each 12 hr. shift) Stability	Recoveries must meet VER% limits in Table 6 , Method 1668C	EPA ⁽¹⁾ Method ⁽²⁾	Labeled congeners: Narrate, no action. Native congeners: J(pos)/UJ(ND) for low bias J(pos) for high bias	5B (H,L) ³	
	Absolute RT of all Labeled congeners and Window Defining Congeners must be +/- 15 sec of RT in ICAL RRT of all congeners must be within range in Table 2 of method	EPA ⁽¹⁾ Method ⁽²⁾	Narrate, no action	5B	Professional judgement. The info in EcoChem TM-05 also generally applies
Blank Contamination					
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected congeners	EPA ⁽¹⁾ Method ⁽²⁾	U(pos) if sample result is < 5X blank concentration	7	Heirarchy of blank review: #1 - Review MB, qualify as needed #2 - Review FB , qualify as needed EMPC values in blanks as considered to be non-detects
Field Blank (FB)	FB: frequency as per QAPP No detected congeners		U(pos) if sample result is < 5X blank concentration	6	
Precision and Accuracy					
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias PJ if only one %R outlier	8 (H,L) ³	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked. Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) %R must meet limits in Table 6 Method 1668C	EPA ⁽¹⁾ Method ⁽²⁾	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) ³	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCS/LCSD not typically required for HRMS analyses. If lab analyzes LCS/LCSD then one set per matrix and batch of 20 samples RPD < 35%	EcoChem standard policy	J(pos) assoc. congener in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD) (if required)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	Optional element. Qualify parent sample only.

PCB Congener Analysis by HRMS
 (Based on EPA DV Guidance¹ and Method EPA 1668C)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Labeled congeners (Internal Standards)	Added to all samples %R must meet limits in Table 6 Method 1668C	EPA ⁽¹⁾ Method ⁽²⁾	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 5% - very low bias J(pos)/UJ(ND) if %R between 5-10% for two or more labeled compounds in a substitution group (ie, mono-, di-, trichlorinated) - very low bias	13 (H,L) ³	See next tab for labeled congener associations as per Table 2 Method 1668
Field Duplicates	Solids: RPD < 50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD < 35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project (EcoChem PJ)	9	RPD values may be dictated by QAPP 35% and 50% are EcoChem defaults
Compound ID and Calculation					
Quantitation/ Identification	All ions for each isomer must maximize within +/- 2 seconds. S/N ratio > 2.5 Ion ratios must meet criteria listed in Table 8 of 1668C; RRTs w/in limits in Table 2 of 1668C	EPA ⁽¹⁾ Method ⁽²⁾	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	The info in EcoChem TM-05 also generally applies
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	EPA ⁽¹⁾ Method ⁽²⁾	If laboratory correctly reported an EMPC value, qualify the native congener U to indicate that the value is an elevated detection limit and qualify total homolog groups J(+)	25	Use professional judgment. See TM-18
Interferences	Lock masses must not deviate +/- 20% from values in Table 7 of 1668C	Method ⁽²⁾	J(pos)/UJ(ND) if present	24	Use professional judgment. See TM-17
Calibration Range	Results greater than highest calibration standard	EcoChem standard policy	Qualify J (pos)	20	If result from dilution analysis is not reported.
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Deliverable (EDD)					
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re-extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

¹ USEPA Region 2 Data Validation, Standard Operating Procedure for EPA Method 1668A, Revision 1, September 2008
 USEPA Region 3 Interim Guidelines for the Validation of Data Generated Using Method 1668 PCB Congener Data, Revision 0, April 2004
 USEPA Region 10 SOP For the Validation of Method 1668 Toxic, Dioxin-like, PCB Data, Revision 1, December 1995
² EPA Method 1668, Rev.C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, April 2010
³ "H" = high bias indicated; "L" = low bias indicated

(pos): Positive Result(s)
 (ND): Non-detects



ECO-CHEM
Data Quality

APPENDIX B

QUALIFIED DATA SUMMARY TABLE

Qualified Data Summary Table
Seattle Iron and Metals - Dust Monitoring Phase II Oct, Nov, Dec 2020

Sample ID	Lab ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	Reason Code
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-121	5.7	pg	J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-111	4	pg	J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-127	5.6	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-169	4.8	pg	J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-182	5.5	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA1668C	PCB-181	3	pg	J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA1668C	PCB-112	360	pg	M,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA1668C	PCB-182	8.9	pg	M,J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA1668C	PCB-181	17	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA1668C	PCB-155	4.8	pg	J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA1668C	PCB-152	20	pg	M,J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA1668C	PCB-155	5.6	pg	M,J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA1668C	PCB-188	6.3	pg	J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA1668C	PCB-184	4.3	pg	J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA1668C	PCB-182	10	pg	M,J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA1668C	PCB-155	8.6	pg	J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA1668C	PCB-186	1.5	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA-TO-09A	1,2,3,4,7,8-HxCDD	6	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA-TO-09A	1,2,3,6,7,8-HxCDD	9	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA-TO-09A	1,2,3,7,8,9-HxCDD	11	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA-TO-09A	1,2,3,4,7,8-HxCDF	3.1	pg	M,J,R	U	25
SITE 1 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-1	EPA-TO-09A	OCDF	24	pg	J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA-TO-09A	1,2,3,7,8-PeCDD	2.2	pg	M,J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA-TO-09A	1,2,3,6,7,8-HxCDD	5.5	pg	J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA-TO-09A	1,2,3,4,7,8-HxCDF	4.5	pg	J,R	U	25
SITE 2 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-2	EPA-TO-09A	1,2,3,4,7,8,9-HpCDF	1.5	pg	M,J,R	U	25

Qualified Data Summary Table
Seattle Iron and Metals - Dust Monitoring Phase II Oct, Nov, Dec 2020

Sample ID	Lab ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	Reason Code
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,6,7,8-HxCDD	6.5	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,7,8,9-HxCDD	6.7	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,4,7,8-HxCDF	3.4	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,6,7,8-HxCDF	2.1	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,4,6,7,8-HpCDF	18	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	OCDF	21	pg	M,J,R	U	25
SITE 3 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-3	EPA-TO-09A	1,2,3,6,7,8-HxCDD-13C	100	%	R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA-TO-09A	1,2,3,7,8-PeCDD	6.2	pg	M,J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA-TO-09A	1,2,3,7,8,9-HxCDD	13	pg	M,J,R	U	25
SITE 4 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-4	EPA-TO-09A	1,2,3,4,6,7,8-HpCDF	28	pg	J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	1,2,3,4,7,8-HxCDD	9.4	pg	M,J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	1,2,3,7,8-PeCDF	3.1	pg	M,J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	1,2,3,4,7,8-HxCDF	5	pg	M,J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	1,2,3,6,7,8-HxCDF	3.7	pg	J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	2,3,4,6,7,8-HxCDF	2.7	pg	J,R	U	25
SITE 5 - COMPOSITE 4 (WET SEASON - OCT, NOV, DEC)	L2541483-5	EPA-TO-09A	1,2,3,4,7,8,9-HpCDF	3.3	pg	M,J,R	U	25

Sample Index
Seattle Iron and Metals - Dust Monitoring Phase II Oct, Nov, Dec 2020

Sample ID	Laboratory ID	Dioxins	PCB Congeners
Site 1 - Composite 4 (Wet Season - Oct, Nov, Dec)	L2541483-1	✓	✓
Site 2 - Composite 4 (Wet Season - Oct, Nov, Dec)	L2541483-2	✓	✓
Site 3 - Composite 4 (Wet Season - Oct, Nov, Dec)	L2541483-3	✓	✓
Site 4 - Composite 4 (Wet Season - Oct, Nov, Dec)	L2541483-4	✓	✓
Site 5 - Composite 4 (Wet Season - Oct, Nov, Dec)	L2541483-5	✓	✓