



LABORATORY DATA CONSULTANTS, INC.

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Northgate Environmental Management, Inc.
1100 Quail Street Ste. 102
Newport Beach, CA 92660
ATTN: Ms. Cindy Arnold

November 5, 2010

SUBJECT: Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada,
Data Validation

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on October 12, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 24163:

<u>SDG #</u>	<u>Fraction</u>
G0H250527, G0H280489 G0I010540	Dioxins/Dibenzofurans

The data validation was performed under Stage 2B/4 guidelines. The analyses were validated using the following documents, as applicable to each method:

- Standard Operating Procedures (SOP) 40, Data Review/Validation, BRC 2009
- Quality Assurance Project Plan Tronox LLC Facility, Henderson Nevada, June 2009
- NDEP Guidance, May 2006
- USEPA, Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Diobenzofurans Data Review, September 2005

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

Sincerely,

Erlinda T. Rauto
Operations Manager/Senior Chemist

EDD CHECKLIST

LDC #: 24163
 SDG #: G0H250527, G0H280489, G0I010540

Page: 1 of 1
 Reviewer: JE
 2nd Reviewer: BC

Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	X			
III. EDD Lab Anomalies				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			X	See EDD_discrepancy_ form_LDC24163_110410.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

**Laboratory Data Consultants, Inc.
Data Validation Report**

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,
Henderson, Nevada

Collection Date: August 23 through August 24, 2010

LDC Report Date: October 28, 2010

Matrix: Soil

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0H250527

Sample Identification

BDT-3-S-20-10BPC	BDT-3-S-15-8BPCMS
BDT-3-S-20-12BPC	BDT-3-S-15-8BPCMSD
BDT-3-S-20-14BPC	
BDT-3-S-20-16BPC	
BDT-3-S-20-18BPC**	
BDT-3-S-20-2BPC	
BDT-3-S-20-4BPC	
BDT-3-S-20-6BPC	
BDT-3-S-20-6BPC_FD	
BDT-3-S-20-8BPC	
BDT-3-S-15-10BPC	
BDT-3-S-15-12BPC	
BDT-3-S-15-14BPC	
BDT-3-S-15-14BPC_FD	
BDT-3-S-15-16BPC	
BDT-3-S-15-18BPC**	
BDT-3-S-15-2BPC	
BDT-3-S-15-4BPC	
BDT-3-S-15-6BPC	
BDT-3-S-15-8BPC	

**Indicates sample underwent Stage 4 review

Introduction

This data review covers 22 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily 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 exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

III. Initial Calibration

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

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds.

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated dioxin/dibenzofuran contaminants were found in the method blanks with the following exceptions:

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0259189-MB	9/16/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.34 pg/g 0.54 pg/g 0.16 pg/g 0.11 pg/g 0.11 pg/g	All samples in SDG G0H250527

Sample concentrations were compared to concentrations detected in the method blanks as required by the QAPP. No sample data was qualified with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
BDT-3-S-20-6BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	1.6 pg/L 1.6 pg/L	1.6U pg/L 1.6U pg/L

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-3-S-20-10BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 13 (40-135) 19 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-3-S-20-12BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	18 (40-135) 11 (40-135) 16 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-14BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	14 (40-135) 8.9 (40-135) 14 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-16BPC	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,6,7,8-HxCDF ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 11 (40-135) 5.8 (40-135) 29 (40-135) 9.3 (40-135)	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-18BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 20 (40-135) 29 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-2BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 20 (40-135) 27 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-4BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 16 (40-135) 27 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-6BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	19 (40-135) 11 (40-135) 17 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-20-6BPC_FD	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	20 (40-135) 11 (40-135) 19 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-3-S-20-8BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 14 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-10BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 17 (40-135) 26 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-12BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 14 (40-135) 19 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-14BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	22 (40-135) 13 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-14BPC_FD	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 14 (40-135) 22 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-16BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	18 (40-135) 11 (40-135) 17 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-18BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	18 (40-135) 10 (40-135) 17 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-2BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 19 (40-135) 33 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-4BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 12 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-3-S-15-6BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 16 (40-135) 25 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-3-S-15-8BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 22 (40-135) 32 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XI. Project Quantitation Limit

All compound quantitation and PQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-3-S-20-10BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
BDT-3-S-15-10BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
BDT-3-S-15-2BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H250527	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H250527	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XII. System Performance

The system performance was acceptable for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples BDT-3-S-20-6BPC and BDT-3-S-20-6BPC_FD and samples BDT-3-S-15-14BPC and BDT-3-S-15-14BPC_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-S-20-6BPC	BDT-3-S-20-6BPC_FD				
1,2,3,7,8-PeCDD	0.25	0.21	-	0.04 (≤ 2.8)	-	-
1,2,3,4,7,8-HxCDD	0.18	2.6U	-	2.42 (≤ 2.6)	-	-
1,2,3,6,7,8-HxCDD	0.36	0.35	-	0.01 (≤ 2.8)	-	-
1,2,3,7,8,9-HxCDD	0.28	0.36	-	0.08 (≤ 2.8)	-	-
1,2,3,4,6,7,8-HpCDD	2.0	1.6	-	0.4 (≤ 2.8)	-	-
OCDD	3.2	1.6	-	1.6 (≤ 5.5)	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-S-20-6BPC	BDT-3-S-20-6BPC_FD				
2,3,7,8-TCDF	1.5	1.4	-	0.1 (≤ 0.55)	-	-
1,2,3,7,8-PeCDF	2.9	2.6	-	0.3 (≤ 2.8)	-	-
2,3,4,7,8-PeCDF	1.5	1.4	-	0.1 (≤ 2.8)	-	-
1,2,3,4,7,8-HxCDF	6.0	5.4	-	0.6 (≤ 2.8)	-	-
1,2,3,6,7,8-HxCDF	3.8	3.1	-	0.7 (≤ 2.8)	-	-
2,3,4,6,7,8-HxCDF	0.88	0.67	-	0.21 (≤ 2.8)	-	-
1,2,3,7,8,9-HxCDF	0.70	0.70	-	0 (≤ 2.8)	-	-
1,2,3,4,6,7,8-HpCDF	17	14	19 (≤ 50)	-	-	-
1,2,3,4,7,8,9-HpCDF	8.7	6.9	-	1.8 (≤ 2.8)	-	-
OCDF	34	31	9 (≤ 50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-S-15-14BPC	BDT-3-S-15-14BPC_FD				
2,3,7,8-TCDD	2.2	1.9	-	0.3 (≤ 0.54)	-	-
1,2,3,7,8-PeCDD	7.9	6.8	-	1.1 (≤ 2.7)	-	-
1,2,3,4,7,8-HxCDD	5.4	4.1	-	1.3 (≤ 2.7)	-	-
1,2,3,6,7,8-HxCDD	12	8.7	-	3.3 (≤ 2.7)	J (all detects)	A
1,2,3,7,8,9-HxCDD	9.1	6.5	-	2.6 (≤ 2.7)	-	-
1,2,3,4,6,7,8-HpCDD	38	28	30 (≤ 50)	-	-	-
OCDD	44	26	51 (≤ 50)	-	J (all detects)	A
2,3,7,8-TCDF	56	54	4 (≤ 50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-S-15-14BPC	BDT-3-S-15-14BPC_FD				
1,2,3,7,8-PeCDF	130	110	17 (≤ 50)	-	-	-
2,3,4,7,8-PeCDF	65	59	10 (≤ 50)	-	-	-
1,2,3,4,7,8-HxCDF	240	200	18 (≤ 50)	-	-	-
1,2,3,6,7,8-HxCDF	140	110	24 (≤ 50)	-	-	-
2,3,4,6,7,8-HxCDF	28	22	24 (≤ 50)	-	-	-
1,2,3,7,8,9-HxCDF	26	23	12 (≤ 50)	-	-	-
1,2,3,4,6,7,8-HpCDF	550	410	29 (≤ 50)	-	-	-
1,2,3,4,7,8,9-HpCDF	290	210	32 (≤ 50)	-	-	-
OCDF	1200	870	32 (≤ 50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H250527**

SDG	Sample	Compound	Flag	A or P	Reason
G0H250527	BDT-3-S-20-10BPC BDT-3-S-20-12BPC BDT-3-S-20-14BPC BDT-3-S-20-18BPC** BDT-3-S-20-2BPC BDT-3-S-20-4BPC BDT-3-S-20-6BPC BDT-3-S-20-6BPC_FD BDT-3-S-20-8BPC BDT-3-S-15-10BPC BDT-3-S-15-12BPC BDT-3-S-15-14BPC BDT-3-S-15-14BPC_FD BDT-3-S-15-16BPC BDT-3-S-15-18BPC** BDT-3-S-15-2BPC BDT-3-S-15-4BPC BDT-3-S-15-6BPC BDT-3-S-15-8BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H250527	BDT-3-S-20-16BPC	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H250527	BDT-3-S-20-10BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H250527	BDT-3-S-15-10BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H250527	BDT-3-S-15-2BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)

SDG	Sample	Compound	Flag	A or P	Reason
G0H250527	BDT-3-S-20-10BPC BDT-3-S-20-12BPC BDT-3-S-20-14BPC BDT-3-S-20-16BPC BDT-3-S-20-18BPC** BDT-3-S-20-2BPC BDT-3-S-20-4BPC BDT-3-S-20-6BPC BDT-3-S-20-6BPC_FD BDT-3-S-20-8BPC BDT-3-S-15-10BPC BDT-3-S-15-12BPC BDT-3-S-15-14BPC BDT-3-S-15-14BPC_FD BDT-3-S-15-16BPC BDT-3-S-15-18BPC** BDT-3-S-15-2BPC BDT-3-S-15-4BPC BDT-3-S-15-6BPC BDT-3-S-15-8BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H250527	BDT-3-S-20-10BPC BDT-3-S-20-12BPC BDT-3-S-20-14BPC BDT-3-S-20-16BPC BDT-3-S-20-18BPC** BDT-3-S-20-2BPC BDT-3-S-20-4BPC BDT-3-S-20-6BPC BDT-3-S-20-6BPC_FD BDT-3-S-20-8BPC BDT-3-S-15-10BPC BDT-3-S-15-12BPC BDT-3-S-15-14BPC BDT-3-S-15-14BPC_FD BDT-3-S-15-16BPC BDT-3-S-15-18BPC** BDT-3-S-15-2BPC BDT-3-S-15-4BPC BDT-3-S-15-6BPC BDT-3-S-15-8BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H250527	BDT-3-S-15-14BPC BDT-3-S-15-14BPC_FD	1,2,3,6,7,8-HxCDD	J (all detects)	A	Field duplicates (Difference) (fd)
G0H250527	BDT-3-S-15-14BPC BDT-3-S-15-14BPC_FD	OCDD	J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
 Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG
 G0H250527**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H250527	BDT-3-S-20-6BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	1.6U pg/L 1.6U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
 Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H250527.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 24163A21

SDG #: G0H250527

Laboratory: Test America

Date: 10/27/10

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

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

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.	Technical holding times	A	Sampling dates: 8/23 - 8/24/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	ICV
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D: 8, 9 13, 14
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

1	BDT-3-S-20-10BPC	11	BDT-3-S-15-10BPC	21	BDT-3-S-15-8BPCMS	31	0259189
2	BDT-3-S-20-12BPC	12	BDT-3-S-15-12BPC	22	BDT-3-S-15-8BPCMSD	32	
3	BDT-3-S-20-14BPC	13	BDT-3-S-15-14BPC	23		33	
4	BDT-3-S-20-16BPC	14	BDT-3-S-15-14BPC_FD	24		34	
5	BDT-3-S-20-18BPC**	15	BDT-3-S-15-16BPC	25		35	
6	BDT-3-S-20-2BPC	16	BDT-3-S-15-18BPC**	26		36	
7	BDT-3-S-20-4BPC	17	BDT-3-S-15-2BPC	27		37	
8	BDT-3-S-20-6BPC	18	BDT-3-S-15-4BPC	28		38	
9	BDT-3-S-20-6BPC_FD	19	BDT-3-S-15-6BPC	29		39	
10	BDT-3-S-20-8BPC	20	BDT-3-S-15-8BPC	30		40	

Notes: _____

LDC #: 24163A21
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

Method: Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
V. Blanks				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 24163A2
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

VIII: Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?			/
Were the performance evaluation (PE) samples within the acceptance limits?			/
IX: Internal standards			
Were internal standard recoveries within the 40-135% criteria?		/	
Was the minimum S/N ratio of all internal standard peaks ≥ 10 ?	/		
X: 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 compound spectra contain all characteristic ions listed in the table attached?	/		
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	/		
Was the signal to noise ratio for each target compound and labeled standard > 2.5 ?	/		
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?	/		
XI: Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/		
XII: System performance			
System performance was found to be acceptable.	/		
XIII: Overall assessment of data			
Overall assessment of data was found to be acceptable.	/		
XIV: Field duplicates			
Field duplicate pairs were identified in this SDG.	/		
Target compounds were detected in the field duplicates.	/		
XV: Field blanks			
Field blanks were identified in this SDG.		/	
Target compounds were detected in the field blanks.			/

VALIDATION FINDINGS WORKSHEET

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

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
Matrix Spike/Matrix Spike Duplicates

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 Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>21422</u>	F	()	75 (80-143)	()	20	NO QUAL RESIN
			f	()	85 (86-134)	()		
			H	()	0 (79-137)	41 (30)		
			I	()	27 (81-134)	()		
			J	()	70 (76-132)	()		
			K	51 (72-140)	0 (72-140)	()		
			L	55 (63-152)	0 (63-152)	()		
			P	3.0 (81-137)	0 (81-137)	()		
			P	()	6.1 (79-139)	()		
			Q	14 (75-141)	0 (75-141)	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		

VALIDATION FINDINGS WORKSHEET
Internal Standards

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".

X N/A Are all internal standard recoveries within the 40-135% criteria?

X N/A Was the S/N ratio all internal standard peaks > 10?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	H	21 (40-135)	√/M, P (1) qual F
			I	13	G, Q
			G	19	e, p
		2	H	18	
			I	11	
			G	16	
		3	H	14	
			I	8.9	
			G	14	▽
		4	F	33	G, P, E
			H	11	F
			I	5.8	G, Q
			E	29	K, L, M, N
			G	9.3	√/e, p
Internal Standards					
A	¹³ C-2,3,7,8-TCDF			Recovery Standards	
B	¹³ C-2,3,7,8-TCDD			K. ¹³ C-1,2,3,4-TCDD	
C	¹³ C-1,2,3,7,8-PeCDF			L. ¹³ C-1,2,3,7,8,9-HxCDD	
D	¹³ C-1,2,3,7,8-PeCDD			M.	
E	¹³ C-1,2,3,6,7,8-HxCDF			N.	
F	¹³ C-1,2,3,6,7,8-HxCDD			O.	
G	¹³ C-1,2,3,4,6,7,8-HpCDF			P.	
H	¹³ C-1,2,3,4,6,7,8-HpCDD			Q.	
I	¹³ C-OCDD			R.	
	Check Standard Used			Check Standard Used	

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".

Y N N/A Are all internal standard recoveries within the 40-135% criteria?

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

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		5	H	32 (40-135)	J/UJ/P OUAL F
			I	20	G, Q
			G	29	O, P
		6	H	29	
			I	20	
			G	27	
		7	H	29	
			I	16	
			G	27	
		8	H	19	
			I	11	
			G	17	

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
A. ¹³ C-2,3,7,8-TCDF		K. ¹³ C-1,2,3,4-TCDD	
B. ¹³ C-2,3,7,8-TCDD		L. ¹³ C-1,2,3,7,8,9-HxCDD	
C. ¹³ C-1,2,3,7,8-PeCDF		M.	
D. ¹³ C-1,2,3,7,8-PeCDD		N.	
E. ¹³ C-1,2,3,6,7,8-HxCDF		O.	
F. ¹³ C-1,2,3,6,7,8-HxCDD		P.	
G. ¹³ C-1,2,3,4,6,7,8-HpCDF		Q.	
H. ¹³ C-1,2,3,4,6,7,8-HpCDD		R.	
I. ¹³ C-OCDD		T.	

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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".

Y/N N/A
Y/N N/A

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

#	Date	Sample-ID	Finding	Associated Samples	Qualifications
		<i>compd</i>			
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		<i>H, K, O, P, Q</i>	<i>x'd cal range</i>	<i>1, 17</i>	<i>J/Pdet (c)</i>
		<i>H, O, P, Q</i>	<i>↓</i>	<i>11</i>	<i>J/Pdet (c)</i>
		<i>Q</i>	<i>↓</i>	<i>17</i>	<i>J/Pdet (c)</i>

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24163A21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 1
Reviewer: [Signature]
2nd Reviewer: [Signature]

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

Y N NA Were field duplicate pairs identified in this SDG?
Y N NA Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	8	9				
B	0.25	0.21		0.04	≤2.8	
C	0.18	2.6U		2.42	≤2.6	
D	0.36	0.35		0.01	≤2.8	
E	0.28	0.36		0.08	≤2.8	
F	2.0	1.6		0.4	≤2.8	
G	3.2	1.6		1.6	≤5.5	
H	1.5	1.4		0.1	≤2.8 0.55	
I	2.9	2.6		0.3	≤2.8	
J	1.5	1.4		0.1	≤2.8	
K	6.0	5.4		0.6	≤2.8	
L	3.8	3.1		0.7	≤2.8	
M	0.88	0.67		0.21	≤2.8	
N	0.70	0.70		0	≤2.8	
O	17	14	19			
P	8.7	6.9	28	1.8	≤ 2.8	
Q	34	31	9			

V:\FIELD DUPLICATES\24163A21.wpd

LDC#:24163A21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 2
Reviewer: [Signature]
2nd Reviewer: [Signature]

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

Y N NA
Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

(fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	13	14				
A	2.2	1.9		0.3	≤0.54	
B	7.9	6.8		1.1	≤2.7	
C	5.4	4.1		1.3	≤2.7	
D	12	8.7		3.3	≤2.7	J/A det
E	9.1	6.5		2.6	≤2.7	
F	38	28	30			
G	44	26	51			J/A det
H	56	54	4			
I	130	110	17			
J	65	59	10			
K	240	200	18			
L	140	110	24			
M	28	22	24			
N	26	23	12			
O	550	410	29			
P	290	210	32			
Q	1200	870	32			

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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	
				Average RRF (Initial)	Average RRF (Initial)	RRF (RRF std)	RRF (RRF std)	%RSD	%RSD		
1	KAL DB 5	9/14/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.984	0.984	1.05	1.0547	11.8	11.8	11.8	11.8
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06300	10.8	10.8	10.8	10.8
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.2454	12.7	12.7	12.7	12.7
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.134	1.134	1.26	1.2560	12.3	12.3	12.3	12.3
			OCDF (¹³ C-OCDD)	2.110	2.110	2.36	2.3599	15.3	15.3	15.3	15.3
2	KAL DB 25	7/26/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	1.056	1.02	1.0237	3.32	3.32	3.32	3.32
			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-OCDD)								
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-OCDD)								

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
Routine Calibration Results Verification

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

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 = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = $(A_x)(C_b) / (A_b)(C_x)$ RRF = continuing calibration RRF
 A_x = Area of compound, A_b = Area of associated internal standard
 C_x = Concentration of compound, C_b = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	cen 7:35	9/27/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	1.02	3.3	1.02	3.3
	DB-225		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-OCDD)					
2	cen 20:08	9/26/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	1.07	1.6	1.07	1.6
	DB-225		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-OCDD)					
3	cen 04:22	9/24/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.984	1.08	9.7	1.0793	9.7
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.10	6.7	1.1008	6.7
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.141	1.24	9.0	1.2444	9.0
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.134	1.24	9.4	1.2408	9.4
			OCDF (¹³ C-OCDD)	2.118	2.13	0.5	2.1286	0.5

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
Routine Calibration Results Verification

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

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 = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = $(A_x)(C_{is}) / (A_{is})(C_x)$ 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	
					RRF (CC)	%D	RRF (CC)	%D
1	cel 7:28	9/25/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.984	1.12	14.3	1.1243	14.3
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.09	5.4	1.0869	5.4
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.14	1.27	11.1	1.2679	11.1
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.134	1.26	10.9	1.2585	10.9
			OCDF (¹³ C-OCDD)	2.118	2.07	2.4	2.0659	2.4
2	cel 20:24	9/23/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.984	1.11	13.0	1.1118	13.0
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.07	3.8	1.0704	3.8
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.14	1.29	13.3	1.2924	13.3
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.134	1.29	13.5	1.2879	13.5
			OCDF (¹³ C-OCDD)	2.134	2.06	2.9	2.0551	2.9
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-OCDD)					

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
Matrix Spike/Matrix Spike Duplicates Results Verification

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * (SSR - SR) / SA$ Where: SSR = Spiked sample result, SR = Sample result
 SA = Spike added

RPD = $|MSR - MSDR| * 2 / (MSR + MSDR)$ MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 2 / + 2 2

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	21.9	21.8	2.8	27.4	26.0	112	112	106	106	5.3	5.3
1,2,3,7,8-PeCDD	109	109	14	122	117	99	99	95	95	4.0	4.0
1,2,3,4,7,8-HxCDD	109	109	7.0	120	116	102	102	98	98	3.6	3.6
1,2,3,4,7,8,9-HpCDF	410	410	410	514	419	93	93	6.1	6.1	2.0	2.0
OCDF	219	218	1800	1880	1490	14	36	0	0	0	23.0

Comments: Refer to Matrix Spike/Matrix Spike Duplicate 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: GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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 = $100 \cdot \text{SSC}/\text{SA}$ Where: SSC = Spiked sample concentration
 SA = Spike added

RPD = $100 \cdot \text{LCS} - \text{LCSD} / 2(\text{LCS} + \text{LCSD})$ LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0259189-105

Compound	Spike Added (123/9)		Spiked Sample Concentration (123/9)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	NA	21.2	NA	106	106	106	106						
1,2,3,7,8-PeCDD	100		102		102	102	102	102						
1,2,3,4,7,8-HxCDD	100		108		108	108	108	108						
1,2,3,4,7,8,9-HpCDF	100		101		101	101	101	101						
OCDF	200		185		92	92	92	92	NA					

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.

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass ^(a)	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C ₁₂ H ₄ ³⁵ Cl ₂ O	TCDF	4	407.7818	M+2	C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO	HpCDF		
	305.8987	M+2	C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO	TCDF		409.7788	M+4	C ₁₂ H ₃₅ Cl ₅ ³⁷ Cl ₂ O	HpCDF		
	315.9419	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₂ O	TCDF (S)		417.8250	M	¹³ C ₁₂ H ₃₅ Cl ₃ O	HpCDF (S)		
	317.9389	M+2	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO	TCDF (S)		419.8220	M+2	¹³ C ₁₂ H ₃₅ Cl ₅ ³⁷ ClO	HpCDF		
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD		423.7767	M+2	¹³ C ₁₂ H ₃₅ Cl ₆ ³⁷ ClO ₂	HpCDD		
	321.8936	M+2	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO ₂	TCDD		425.7737	M+2	C ₁₂ H ₃₅ Cl ₆ ³⁷ ClO ₂	HpCDD		
	331.9368	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD (S)		435.8169	M+4	¹³ C ₁₂ H ₃₅ Cl ₅ ³⁷ Cl ₂ O ₂	HpCDD (S)		
	333.9338	M+2	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO ₂	TCDD (S)		437.8140	M+2	¹³ C ₁₂ H ₃₅ Cl ₆ ³⁷ ClO ₂	HpCDD (S)		
	375.8364	M+2	C ₁₂ H ₄ ³⁵ Cl ₅ ³⁷ ClO	HxCDFPE		479.7165	M+4	¹³ C ₁₂ H ₃₅ Cl ₆ ³⁷ Cl ₂ O ₂	HpCDD (S)		
	[354.9792]	LOCK	C ₉ F ₁₃	PFK		[430.9728]	LOCK	C ₁₂ H ₃₅ Cl ₇ ³⁷ Cl ₂ O	NCDPE		
								C ₉ F ₁₇	PFK		
	2	339.8597	M+2	C ₁₂ H ₃₅ Cl ₄ ³⁷ ClO		PeCDF	5	441.7428	M+2	C ₁₂ ³⁵ Cl ₃ ³⁷ ClO	OCDF
		341.8567	M+4	C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O		PeCDF		443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	OCDF
351.9000		M+2	¹³ C ₁₂ H ₃₅ Cl ₄ ³⁷ ClO	PeCDF (S)	457.7377	M+2		C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD		
353.8970		M+4	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O	PeCDF (S)	459.7348	M+4		C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD		
355.8546		M+2	C ₁₂ H ₃₅ Cl ₄ ³⁷ ClO ₂	PeCDD	469.7780	M+2		¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD (S)		
357.8516		M+4	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD	471.7750	M+4		¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD (S)		
367.8949		M+2	¹³ C ₁₂ H ₃₅ Cl ₄ ³⁷ ClO ₂	PeCDD (S)	513.6775	M+4		C ₁₂ ³⁵ Cl ₃ ³⁷ Cl ₂ O	DCDPE		
369.8919		M+4	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD (S)	[422.9278]	LOCK		C ₁₀ F ₁₇	PFK		
409.7974		M+2	C ₁₂ H ₃₅ Cl ₆ ³⁷ ClO	HxCDFPE							
[354.9792]		LOCK	C ₉ F ₁₃	PFK							
3		373.8208	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF						
		375.8178	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O	HxCDF						
	383.8639	M	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ O	HxCDF (S)							
	385.8610	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ ClO	HxCDF (S)							
	389.8156	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD							
	391.8127	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ ClO ₂	HxCDD							
	401.8559	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ ClO ₂	HxCDD (S)							
	403.8529	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD (S)							
	445.7555	M+4	C ₁₂ H ₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	HxCDD (S)							
	[430.9728]	LOCK	C ₉ F ₁₇	OCDFPE							
				PFK							

(a) The following nuclidic masses were used:

H = 1.007825
 C = 12.000000
¹³C = 13.003355
 F = 18.9984
 O = 15.994915
³⁵Cl = 34.968853
³⁷Cl = 36.965903

S = internal/recovery standard

LDC #: 24163A21
 SDG #: pe cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd reviewer: [Signature]

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

Y / N / N/A
Y / N / N/A

Were all reported results recalculated and verified for all level IV samples?
 Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(DF)}{(A_s)(RRF)(V_o)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_s = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- RRF = Relative Response Factor (average) from the initial calibration
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. #5, 1, 2, 3, 7, 8 - TCDF

$$\text{Conc.} = \frac{(469174000)(2000)}{(469583660)(1.09)(9.67)(0.943)}$$

= 201 pg/g

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
	#5	2,3,7,8-TCDF (DBXX)			
				= $\frac{62416600(2000)}{147625800(1.06)(9.67)(0.943)}$	= 87.483 pg/g

**Laboratory Data Consultants, Inc.
Data Validation Report**

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,
Henderson, Nevada

Collection Date: August 26, 2010

LDC Report Date: October 28, 2010

Matrix: Soil/Water

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0H280489

Sample Identification

BDT-2-S-5-10BPC
BDT-2-S-5-12BPC
BDT-2-S-5-14BPC**
BDT-2-S-5-12BPC_FD
BDT-2-S-5-8BPC
BDT-2-S-5-2BPC
BDT-2-S-5-4BPC
BDT-2-S-5-6BPC
BDT-2-S-10-10BPC
BDT-2-S-10-12BPC
BDT-2-S-10-14BPC**
BDT-2-S-10-2BPC
BDT-2-S-10-4BPC
BDT-2-S-10-6BPC
BDT-2-S-10-8BPC
EB-08262010
BDT-2-S-5-10BPCMS
BDT-2-S-5-10BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

This data review covers 17 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily 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 exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

III. Initial Calibration

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

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds.

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated dioxin/dibenzofuran contaminants were found in the method blanks with the following exceptions:

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0246227-MB	9/3/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	1.7 pg/L 0.88 pg/L 1.4 pg/L 2.3 pg/L 9.0 pg/L 1.7 pg/L 0.68 pg/L 1.0 pg/L 1.8 pg/L 2.8 pg/L 1.5 pg/L	All water samples in SDG G0H280489
0257153-MB	9/14/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.11 pg/g 0.57 pg/g 0.089 pg/g 0.059 pg/g 0.16 pg/g 0.18 pg/g	All soil samples in SDG G0H280489

Sample concentrations were compared to concentrations detected in the method blanks as required by the QAPP. No sample data was qualified with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-08262010	OCDD 1,2,3,4,6,7,8-HpCDF	18 pg/L 4.4 pg/L	18U pg/L 4.4U pg/L

Sample EB-08262010 was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-08262010	8/26/10	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	18 pg/L 4.4 pg/L 4.7 pg/L	All soil samples in SDG G0H280489

Sample concentrations were compared to concentrations detected in the field blanks as required by the QAPP. No sample data was qualified.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-2-S-5-10BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD	38 (40-135) 26 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-5-12BPC	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 27 (40-135) 17 (40-135) 26 (40-135)	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-5-14BPC**	¹³ C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-2-S-5-12BPC_FD	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 23 (40-135) 15 (40-135) 22 (40-135)	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-5-8BPC	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 27 (40-135) 16 (40-135) 25 (40-135)	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-2-S-5-4BPC	¹³ C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-2-S-5-6BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 17 (40-135) 28 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-10-10BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 22 (40-135) 33 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-10-12BPC	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,6,7,8-HxCDF ¹³ C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 10 (40-135) 5.3 (40-135) 27 (40-135) 12 (40-135)	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-10-14BPC**	¹³ C-OCDD	34 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-2-S-10-2BPC	¹³ C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-2-S-10-4BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD	39 (40-135) 29 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-10-6BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 20 (40-135) 35 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-2-S-10-8BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 26 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XI. Project Quantitation Limit

All compound quantitation and PQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-2-S-5-10BPC	2,3,7,8-TCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
BDT-2-S-5-12BPC_FD	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
BDT-2-S-5-2BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects)	P
BDT-2-S-10-14BPC**	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
BDT-2-S-10-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-2-S-10-4BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
BDT-2-S-10-6BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
BDT-2-S-10-8BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H280489	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H280489	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XII. System Performance

The system performance was acceptable for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples BDT-2-S-5-12BPC and BDT-2-S-5-12BPC_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-2-S-5-12BPC	BDT-2-S-5-12BPC_FD				
2,3,7,8-TCDD	8.2	24	98 (≤ 50)		J (all detects)	A
1,2,3,7,8-PeCDD	27	81	100 (≤ 50)		J (all detects)	A
1,2,3,4,7,8-HxCDD	19	62	106 (≤ 50)		J (all detects)	A
1,2,3,6,7,8-HxCDD	33	110	108 (≤ 50)		J (all detects)	A
1,2,3,7,8,9-HxCDD	19	69	114 (≤ 50)		J (all detects)	A
1,2,3,4,6,7,8-HpCDD	110	400	114 (≤ 50)	-	J (all detects)	A
OCDD	440	1200	93 (≤ 50)	-	J (all detects)	A
2,3,7,8-TCDF	150	410	93 (≤ 50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	290	870	100 (≤ 50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	130	400	102 (≤ 50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	410	1100	91 (≤ 50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	320	1000	103 (≤ 50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	66	210	104 (≤ 50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	82	220	91 (≤ 50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	1300	4500	110 (≤ 50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	620	2000	105 (≤ 50)	-	J (all detects)	A
OCDF	3900	14000	113 (≤ 50)	-	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H280489**

SDG	Sample	Compound	Flag	A or P	Reason
G0H280489	BDT-2-S-5-10BPC BDT-2-S-10-4BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280489	BDT-2-S-5-12BPC BDT-2-S-5-12BPC_FD BDT-2-S-5-8BPC	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280489	BDT-2-S-5-14BPC** BDT-2-S-5-4BPC BDT-2-S-10-14BPC** BDT-2-S-10-2BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects)	P	Internal standards (%R) (i)
G0H280489	BDT-2-S-5-6BPC BDT-2-S-10-10BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280489	BDT-2-S-10-12BPC	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280489	BDT-2-S-5-10BPC	2,3,7,8-TCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280489	BDT-2-S-5-12BPC_FD	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)

SDG	Sample	Compound	Flag	A or P	Reason
G0H280489	BDT-2-S-5-2BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280489	BDT-2-S-10-14BPC**	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280489	BDT-2-S-10-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280489	BDT-2-S-10-4BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280489	BDT-2-S-5-10BPC BDT-2-S-5-12BPC BDT-2-S-5-14BPC** BDT-2-S-5-12BPC_FD BDT-2-S-5-8BPC BDT-2-S-5-2BPC BDT-2-S-5-4BPC BDT-2-S-5-6BPC BDT-2-S-10-10BPC BDT-2-S-10-12BPC BDT-2-S-10-14BPC** BDT-2-S-10-2BPC BDT-2-S-10-4BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC EB-08262010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H280489	BDT-2-S-5-10BPC BDT-2-S-5-12BPC BDT-2-S-5-14BPC** BDT-2-S-5-12BPC_FD BDT-2-S-5-8BPC BDT-2-S-5-2BPC BDT-2-S-5-4BPC BDT-2-S-5-6BPC BDT-2-S-10-10BPC BDT-2-S-10-12BPC BDT-2-S-10-14BPC** BDT-2-S-10-2BPC BDT-2-S-10-4BPC BDT-2-S-10-6BPC BDT-2-S-10-8BPC EB-08262010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

SDG	Sample	Compound	Flag	A or P	Reason
G0H280489	BDT-2-S-5-12BPC BDT-2-S-5-12BPC_FD	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-HxCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG
G0H280489**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H280489	EB-08262010	OCDD 1,2,3,4,6,7,8-HpCDF	18U pg/L 4.4U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H280489.**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 24163B21
SDG #: G0H280489
Laboratory: Test America

Stage 2B/4

Date: 10/27/10
Page: 1 of 1
Reviewer: FZ
2nd Reviewer: [Signature]

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

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.	Technical holding times	Δ	Sampling dates: <u>8/26/10</u>
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration/ ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	<u>LCs</u>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	<u>D = 2, 4</u>
XV.	Field blanks	SW	<u>EB = 16</u>

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

soil + water

1	BDT-2-S-5-10BPC	11	BDT-2-S-10-14BPC**	21	<u>0257153</u>	31	
2	BDT-2-S-5-12BPC <u>D</u>	12	BDT-2-S-10-2BPC	22	<u>0246227</u>	32	
3	BDT-2-S-5-14BPC**	13	BDT-2-S-10-4BPC	23		33	
4	BDT-2-S-5-12BPC <u>FD D</u>	14	BDT-2-S-10-6BPC	24		34	
5	BDT-2-S-5-8BPC	15	BDT-2-S-10-8BPC	25		35	
6	BDT-2-S-5-2BPC	16	<u>EB-08262010</u> <u>W</u>	26		36	
7	BDT-2-S-5-4BPC	17	BDT-2-S-5-10BPCMS	27		37	
8	BDT-2-S-5-6BPC	18	BDT-2-S-5-10BPCMSD	28		38	
9	BDT-2-S-10-10BPC	19		29		39	
10	BDT-2-S-10-12BPC	20		30		40	

Notes: _____

LDC #: 2416382/
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
 Reviewer: _____
 2nd Reviewer: _____

Method: Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	<input checked="" type="checkbox"/>			
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>			
II. GC/MS instrument performance check				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>			
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$?	<input checked="" type="checkbox"/>			
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>			
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>			
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10 ?	<input checked="" type="checkbox"/>			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>			
V. Blanks				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>			
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		<input checked="" type="checkbox"/>		
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>			
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>			

LDC #: 24163B21
 SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

VIII. Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
IX. Internal standards			
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the minimum S/N ratio of all internal standard peaks ≥ 10 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
X. 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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the signal to noise ratio for each target compound and labeled standard > 2.5 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
For PCDF identification, was any signal ($S/N \geq 2.5$, at \pm seconds RT) detected in the corresponding PCDF channel?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XII. System performance			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XV. Field blanks			
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

VALIDATION FINDINGS WORKSHEET

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

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

Internal Standards

Reviewer: FT

2nd Reviewer: [Signature]

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".

X N / N/A Are all internal standard recoveries were within the 40-135% criteria?

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

(L)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications																																								
		1	H	38 (40-135)	J/M/P Q,M,N F																																								
			I	26 ()	G,B																																								
2			F	39 ()	C,D,E K,L,M,N																																								
			H	27 ()	F																																								
			I	17 ()	G,Q																																								
			G	26 ()	O,P																																								
3			I	33 ()	G,Q																																								
4			F	33 ()	C,D,E K,L,M,N																																								
			H	23 ()	F																																								
			I	15 ()	G,Q																																								
			G	22 ()	O,P																																								
5			F	37 ()	C,D,E K,L,M,N																																								
			H	27 ()	F																																								
			I	16 ()	G,Q																																								
			G	25 ()	O,P																																								
<table border="1"> <thead> <tr> <th>Internal Standards</th> <th>Check Standard Used</th> <th>Recovery Standards</th> <th>Check Standard Used</th> </tr> </thead> <tbody> <tr> <td>¹³C-2,3,7,8-TCDF</td> <td></td> <td>¹³C-1,2,3,4-TCDD</td> <td></td> </tr> <tr> <td>¹³C-2,3,7,8-TCDD</td> <td></td> <td>¹³C-1,2,3,7,8,9-HxCDD</td> <td></td> </tr> <tr> <td>¹³C-1,2,3,7,8-PeCDF</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-1,2,3,7,8-PeCDD</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-1,2,3,6,7,8-HxCDF</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-1,2,3,6,7,8-HxCDD</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-1,2,3,4,6,7,8-HpCDF</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-1,2,3,4,6,7,8-HpCDD</td> <td></td> <td></td> <td></td> </tr> <tr> <td>¹³C-OCDF</td> <td></td> <td></td> <td></td> </tr> </tbody> </table>						Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used	¹³ C-2,3,7,8-TCDF		¹³ C-1,2,3,4-TCDD		¹³ C-2,3,7,8-TCDD		¹³ C-1,2,3,7,8,9-HxCDD		¹³ C-1,2,3,7,8-PeCDF				¹³ C-1,2,3,7,8-PeCDD				¹³ C-1,2,3,6,7,8-HxCDF				¹³ C-1,2,3,6,7,8-HxCDD				¹³ C-1,2,3,4,6,7,8-HpCDF				¹³ C-1,2,3,4,6,7,8-HpCDD				¹³ C-OCDF			
Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used																																										
¹³ C-2,3,7,8-TCDF		¹³ C-1,2,3,4-TCDD																																											
¹³ C-2,3,7,8-TCDD		¹³ C-1,2,3,7,8,9-HxCDD																																											
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¹³ C-1,2,3,4,6,7,8-HpCDD																																													
¹³ C-OCDF																																													

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

#	Date	comps Sample-ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
		H, L, K, O, P, R	x'd cal Range	1	J/P det (e)
		H, O, R	↓	4	
		H, O, P, R	↓	6	
		O	↓	11	
		H, I, K, L, O, P, R	↓	12	

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24163B21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 1
Reviewer: [Signature]
2nd Reviewer: [Signature]

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

Y N NA
Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

* EMPC
(fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	4				
A	8.2	24	98			J/A dt
B	27	81	100			
C	19 *	62	106			
D	33	110	108			
E	19	69	114			
F	110	400	114			
G	440	1200	93			
H	150	410	93			
I	290	870	100			
J	130	400	102			
K	410	1100	91			
L	320	1000	103			
M	66	210	104			
N	82	220	91			
O	1300	4500	110			
P	620	2000	105			
Q	3900	14000	113			✓

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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

$RRF = (A_s)(C_{is}) / (A_{is})(C_s)$
 average RRF = sum of the RRFs / number of standards
 $\%RSD = 100 * (S / X)$
 A_s = Area of compound,
 A_{is} = Area of associated internal standard
 C_s = Concentration of compound,
 C_{is} = Concentration of internal standard
 S = Standard deviation of the RRFs,
 X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (initial)	%RSD	Average RRF (initial)	%RSD	RRF (RRF ² std)	%RSD	RRF (RRF ² std)	%RSD
1	ICAL	9/18/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.02408	9.91493	1.02408	9.91493	1.14942	9.91493	1.14942	9.91493
	DB5		2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.18998	4.16239	1.18998	4.16239	1.18451	4.16239	1.18451	4.16239
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.16375	7.14786	1.16375	7.14786	1.23468	7.14786	1.23468	7.14786
2	DB225	7/26/10	1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.08281	6.58155	1.08281	6.58155	1.16859	6.58155	1.16859	6.58155
	ICAL		OCDF (¹³ C-OCDF)	1.54183	8.11137	1.54183	8.11137	1.64541	8.11137	1.64541	8.11137
			2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	3.32	1.056	3.32	1.0237	3.32	1.0237	3.32
3			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)								

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
Routine Calibration Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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 = $100 \times (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 $\text{RRF} = (A_s)(C_{is}) / (A_{is})(C_s)$ A_s = Area of compound, A_{is} = Area of associated internal standard
 C_s = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	00V 1-23	9/25/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.02408	1.05452	1.05452	3.0	3.0
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.18998	1.20384	1.20384	1.2	1.2
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.16375	1.22133	1.22133	4.9	4.9
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.08281	1.6627	1.6627	7.7	7.7
			OCDF (¹³ C-OCDF)	1.54183	1.73465	1.73465	12.5	12.5
2	00V 9-16	9/25/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)		1.07334	1.07334	4.8	4.8
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)		1.19780	1.19780	0.7	0.7
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)		1.20852	1.20852	3.8	3.8
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)		1.14611	1.14611	5.8	5.8
			OCDF (¹³ C-OCDF)		1.73761	1.73761	12.7	12.7
3	00V DB25		2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.050	0.93	0.9345	11.5	11.5
			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 Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 241631321

SDG #: per cons

VALIDATION FINDINGS WORKSHEET

Matrix Spike/Matrix Spike Duplicates Results Verification

Page: 1 of 1

Reviewer: F7

2nd Reviewer: [Signature]

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * (SSR - SR) / SA$ Where: SSR = Spiked sample result, SR = Sample result
SA = Spike added

RPD = $|MSR - MSDR| * 2 / (MSR + MSDR)$ MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 17 + 18

Compound	Spike Added ()		Sample Concentration ()		Spiked Sample Concentration ()		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported	Recalculated
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc.	Reported	Recalc.	RPD	RPD
2,3,7,8-TCDD	22.7	22.4	49	55.7	75.1	55.7	117	117	32	32	30	30
1,2,3,7,8-PeCDD	114	112	170	231	287	231	107	107	59	59	22	22
1,2,3,4,7,8-HxCDD	114	112	130	211	266	211	118	118	70	70	23	23
1,2,3,4,7,8,9-HpCDF	114	112	360	330	5170	330	1350	1350	0	0	0	43
OCDF	227	224	28000	22200	28600	22200	477	477	0	0	0	25

Comments: Refer to Matrix Spike/Matrix Spike Duplicate 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: GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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 = $100 \cdot \text{SSC}/\text{SA}$ Where: SSC = Spiked sample concentration
 SA = Spike added

RPD = $100 \cdot \frac{\text{LCS} - \text{LCSD}}{\text{LCS} + \text{LCSD}}$

LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0251153

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
2,3,7,8-TCDD	20.0	NA	18.4	NA	92	92	92	92						
1,2,3,7,8-PeCDD	100		95.8		96	96	96	96						
1,2,3,4,7,8-HxCDD	100		93.4		93	93	93	93						
1,2,3,4,7,8,9-HpCDF	100		98.2		98	98	98	98						
OCDF	200	↓	187	↓	94	94	94	94	NA					

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.

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass ^(a)	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C ₁₂ H ₄ ³⁵ Cl ₂ O	TCDF	4	407.7818	M+2	C ₁₂ H ₃₅ ³⁷ Cl ₂ O	HpCDF		
	305.8987	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10	TCDF		409.7788	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10	HpCDF		
	315.9419	M	C ₁₂ H ₄ ³⁵ Cl ₄ O	TCDF (S)		417.8250	M	C ₁₂ H ³⁵ Cl ₅ O	HpCDF (S)		
	317.9389	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10	TCDF (S)		419.8220	M+2	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10	HpCDF		
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₂ O ₂	TCDD		423.7767	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ C10	HpCDD		
	321.8936	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	TCDD		425.7737	M+2	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10 ₂	HpCDD		
	331.9368	M	C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD (S)		435.8169	M+4	C ₁₂ H ³⁵ Cl ₆ ³⁷ C10 ₂	HpCDD (S)		
	333.9338	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	TCDD (S)		437.8140	M+2	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10 ₂	HpCDD (S)		
	375.8364	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	HxCDFE		479.7165	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ C10 ₂	HpCDD (S)		
	[354.9792]	LOCK	C ₉ F ₁₃	PFK		[430.9728]	LOCK	C ₁₂ H ³⁵ Cl ₇ ³⁷ C10 ₂	NCDFE		
								C ₉ F ₁₇	PFK		
	2	339.8597	M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10		PeCDF	5	441.7428	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ C10	OCDF
		341.8567	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10		PeCDF		443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ C10	OCDF
351.9000		M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10	PeCDF (S)	457.7377	M+2		C ₁₂ ³⁵ Cl ₅ ³⁷ C10 ₂	OCDD		
353.8970		M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10	PeCDF (S)	459.7348	M+4		C ₁₂ ³⁵ Cl ₆ ³⁷ C10 ₂	OCDD		
355.8546		M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	PeCDD	469.7780	M+2		C ₁₂ ³⁵ Cl ₅ ³⁷ C10 ₂	OCDD (S)		
357.8516		M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10 ₂	PeCDD	471.7750	M+4		C ₁₂ ³⁵ Cl ₆ ³⁷ C10 ₂	OCDD (S)		
367.8949		M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	PeCDD (S)	513.6775	M+2		C ₁₂ ³⁵ Cl ₅ ³⁷ C10 ₂	OCDD (S)		
369.8919		M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10 ₂	PeCDD (S)	[422.9278]	M+4		C ₁₂ ³⁵ Cl ₆ ³⁷ C10 ₂	DCDFE		
409.7974		M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ C10 ₂	HpCDFE		LOCK		C ₁₀ F ₁₇	PFK		
[354.9792]		LOCK	C ₉ F ₁₃	PFK							
3		373.8208	M+2	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10	HxCDF						
		375.8178	M+4	C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ C10 ₂	HxCDF						
	383.8639	M	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10	HxCDF (S)							
	385.8610	M+2	C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ C10	HxCDF (S)							
	389.8156	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ C10 ₂	HxCDD							
	391.8127	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ C10 ₂	HxCDD							
	401.8559	M+2	C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ C10 ₂	HxCDD (S)							
	403.8529	M+4	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ C10 ₂	HxCDD (S)							
	445.7555	M+4	C ₁₂ H ₂ ³⁵ Cl ₃ ³⁷ C10 ₂	HxCDD (S)							
	[430.9728]	LOCK	C ₉ F ₁₇	OCDFE							
				PFK							

(a) The following nuclidic masses were used:

- H = 1.007825
- C = 12.000000
- ¹³C = 13.003355
- F = 18.9984
- O = 15.994915
- ³⁵Cl = 34.968853
- ³⁷Cl = 36.965903

S = internal/recovery standard

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: Tronox LLC Facility, PCS Additional Sampling,
Henderson, Nevada

Collection Date: August 30, 2010

LDC Report Date: November 3, 2010

Matrix: Soil

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0I010540

Sample Identification

BDT-1-S-15-10BPC
BDT-1-S-15-12BPC
BDT-1-S-15-14BPC
BDT-1-S-15-2BPC
BDT-1-S-15-4BPC
BDT-1-S-15-6BPC
BDT-1-S-15-8BPC
BDT-1-S-15-2BPC_FD
BDT-1-S-10-10BPC
BDT-1-S-10-12BPC
BDT-1-S-10-14BPC**
BDT-1-S-10-2BPC
BDT-1-S-10-4BPC
BDT-1-S-10-6BPC
BDT-1-S-10-8BPC
BDT-1-S-10-8BPCMS
BDT-1-S-10-8BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

This data review covers 17 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

This review follows the Standard Operating Procedures (SOP) 40, Data Review/Validation (BRC 2009), the Quality Assurance Project Plan Tronox LLC Facility, Henderson, Nevada (June 2009), NDEP guidance (May 2006), and USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005).

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.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.
- B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.
- JK The analytical result is an estimated maximum possible concentration (EMPC).
- X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.
- J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.
- J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.
- J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.
- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.
- None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily 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 exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

III. Initial Calibration

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

Percent relative standard deviations (%RSD) were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds.

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

All of the routine calibration percent differences (%D) between the initial calibration RRF and the routine calibration RRF were less than or equal to 20.0% for unlabeled compounds and less than or equal to 30.0% for labeled compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
9/30/10	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD	27.3 (≤ 25) 26.2 (≤ 25)	BDT-1-S-15-10BPC BDT-1-S-15-12BPC BDT-1-S-15-14BPC BDT-1-S-15-2BPC BDT-1-S-15-4BPC BDT-1-S-15-6BPC BDT-1-S-15-8BPC BDT-1-S-15-2BPC_FD 0260236-MB	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

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

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated dioxin/dibenzofuran contaminants were found in the method blanks with the following exceptions:

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0260236-MB	9/17/10	OCDD OCDF	0.91 pg/g 0.13 pg/g	All samples in SDG G01010540

Sample concentrations were compared to concentrations detected in the method blanks as required by the QAPP. No sample data was qualified with the following exceptions:

Sample	Compound	Reported Concentration	Modified Final Concentration
BDT-1-S-15-10BPC	OCDD	1.8 pg/g	1.8U pg/g
BDT-1-S-15-14BPC	OCDD	1.5 pg/g	1.5U pg/g
BDT-1-S-15-2BPC	OCDD	0.95 pg/g	0.95U pg/g
BDT-1-S-15-4BPC	OCDD	1.8 pg/g	1.8U pg/g
BDT-1-S-15-6BPC	OCDD	3.8 pg/g	3.8U pg/g
BDT-1-S-15-8BPC	OCDD	4.0 pg/g	4.0U pg/g
BDT-1-S-15-2BPC_FD	OCDD	2.7 pg/g	2.7U pg/g
BDT-1-S-10-10BPC	OCDD	1.7 pg/g	1.7U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
BDT-1-S-10-12BPC	OCDD	0.83 pg/g	0.83U pg/g
BDT-1-S-10-14BPC**	OCDD	1.3 pg/g	1.3U pg/g
BDT-1-S-10-2BPC	OCDD	0.86 pg/g	0.86U pg/g
BDT-1-S-10-4BPC	OCDD	0.87 pg/g	0.87U pg/g
BDT-1-S-10-6BPC	OCDD	1.5 pg/g	1.5U pg/g
BDT-1-S-10-8BPC	OCDD	1.9 pg/g	1.9U pg/g

Sample EB-08302010 (from SDG G0I010538) was identified as an equipment rinsate. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-08302010	8/30/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	2.3 pg/L 2.7 pg/L 2.1 pg/L 1.7 pg/L 1.2 pg/L 5.2 pg/L	All samples in SDG G0I010540

Sample concentrations were compared to concentrations detected in the field blanks as required by the QAPP. No sample data was qualified.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-S-15-12BPC	¹³ C-OCDD	29 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-S-15-14BPC	¹³ C-OCDD	34 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-S-15-4BPC	¹³ C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-S-15-6BPC	¹³ C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-S-10-10BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 29 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-1-S-10-12BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 30 (40-135) 36 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 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
BDT-1-S-10-14BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	31 (40-135) 26 (40-135) 30 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-1-S-10-4BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 30 (40-135) 35 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-1-S-10-6BPC	¹³ C-OCDD	29 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-S-10-8BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 25 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XI. Project Quantitation Limit

All compound quantitation and PQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-1-S-15-2BPC BDT-1-S-15-6BPC BDT-1-S-15-2BPC_FD BDT-1-S-10-10BPC BDT-1-S-10-12BPC BDT-1-S-10-2BPC BDT-1-S-10-6BPC BDT-1-S-10-8BPC	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0I010540	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0I010540	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XII. System Performance

The system performance was acceptable for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Duplicates

Samples BDT-1-S-15-2BPC and BDT-1-S-15-2BPC_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-1-S-15-2BPC	BDT-1-S-15-2BPC_FD				
1,2,3,4,6,7,8-HpCDD	0.16	0.28	-	0.12 (≤ 2.6)	-	-
OCDD	0.95	2.7	-	1.75 (≤ 5.3)	-	-
2,3,7,8-TCDF	0.21	0.14	-	0.07 (≤ 0.53)	-	-
1,2,3,4,7,8-HxCDF	0.23	0.21	-	0.02 (≤ 2.6)	-	-
1,2,3,6,7,8-HxCDF	0.24	0.16	-	0.08 (≤ 2.6)	-	-
2,3,4,6,7,8-HxCDF	0.082	2.6U	-	2.518 (≤ 2.6)	-	-
1,2,3,4,6,7,8-HpCDF	0.53	0.59	-	0.06 (≤ 2.6)	-	-
1,2,3,4,7,8,9-HpCDF	0.24	0.16	-	0.08 (≤ 2.6)	-	-
OCDF	0.97	1.1	-	0.13 (≤ 5.3)	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I010540**

SDG	Sample	Compound	Flag	A or P	Reason
G0I010540	BDT-1-S-15-10BPC BDT-1-S-15-12BPC BDT-1-S-15-14BPC BDT-1-S-15-2BPC BDT-1-S-15-4BPC BDT-1-S-15-6BPC BDT-1-S-15-8BPC BDT-1-S-15-2BPC_FD	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Continuing calibration (%D) (c)
G0I010540	BDT-1-S-15-12BPC BDT-1-S-15-14BPC BDT-1-S-15-4BPC BDT-1-S-15-6BPC BDT-1-S-10-6BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I010540	BDT-1-S-10-10BPC BDT-1-S-10-12BPC BDT-1-S-10-14BPC** BDT-1-S-10-4BPC BDT-1-S-10-8BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I010540	BDT-1-S-15-2BPC BDT-1-S-15-6BPC BDT-1-S-15-2BPC_FD BDT-1-S-10-10BPC BDT-1-S-10-12BPC BDT-1-S-10-2BPC BDT-1-S-10-6BPC BDT-1-S-10-8BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (e)
G0I010540	BDT-1-S-15-10BPC BDT-1-S-15-12BPC BDT-1-S-15-14BPC BDT-1-S-15-2BPC BDT-1-S-15-4BPC BDT-1-S-15-6BPC BDT-1-S-15-8BPC BDT-1-S-15-2BPC_FD BDT-1-S-10-10BPC BDT-1-S-10-12BPC BDT-1-S-10-14BPC** BDT-1-S-10-2BPC BDT-1-S-10-4BPC BDT-1-S-10-6BPC BDT-1-S-10-8BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason
G0I010540	BDT-1-S-15-10BPC BDT-1-S-15-12BPC BDT-1-S-15-14BPC BDT-1-S-15-2BPC BDT-1-S-15-4BPC BDT-1-S-15-6BPC BDT-1-S-15-8BPC BDT-1-S-15-2BPC_FD BDT-1-S-10-10BPC BDT-1-S-10-12BPC BDT-1-S-10-14BPC** BDT-1-S-10-2BPC BDT-1-S-10-4BPC BDT-1-S-10-6BPC BDT-1-S-10-8BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG
G0I010540**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I010540	BDT-1-S-15-10BPC	OCDD	1.8U pg/g	A	bl
G0I010540	BDT-1-S-15-14BPC	OCDD	1.5U pg/g	A	bl
G0I010540	BDT-1-S-15-2BPC	OCDD	0.95U pg/g	A	bl
G0I010540	BDT-1-S-15-4BPC	OCDD	1.8U pg/g	A	bl
G0I010540	BDT-1-S-15-6BPC	OCDD	3.8U pg/g	A	bl
G0I010540	BDT-1-S-15-8BPC	OCDD	4.0U pg/g	A	bl
G0I010540	BDT-1-S-15-2BPC_FD	OCDD	2.7U pg/g	A	bl
G0I010540	BDT-1-S-10-10BPC	OCDD	1.7U pg/g	A	bl
G0I010540	BDT-1-S-10-12BPC	OCDD	0.83U pg/g	A	bl
G0I010540	BDT-1-S-10-14BPC**	OCDD	1.3U pg/g	A	bl
G0I010540	BDT-1-S-10-2BPC	OCDD	0.86U pg/g	A	bl
G0I010540	BDT-1-S-10-4BPC	OCDD	0.87U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I010540	BDT-1-S-10-6BPC	OCDD	1.5U pg/g	A	bl
G0I010540	BDT-1-S-10-8BPC	OCDD	1.9U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I010540**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 24163C21
 SDG #: G0I010540
 Laboratory: Test America

Date: 10/27/10
 Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

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

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.	Technical holding times	A	Sampling dates: 8/30/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 4 & 8
XV.	Field blanks	SW	EB = EB-08302010 SDG #

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

G0I010538

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

1	BDT-1-S-15-10BPC	11	BDT-1-S-10-14BPC**	21	0260236-NR	31
2	BDT-1-S-15-12BPC	12	BDT-1-S-10-2BPC	22		32
3	BDT-1-S-15-14BPC	13	BDT-1-S-10-4BPC	23		33
4	BDT-1-S-15-2BPC D	14	BDT-1-S-10-6BPC	24		34
5	BDT-1-S-15-4BPC	15	BDT-1-S-10-8BPC	25		35
6	BDT-1-S-15-6BPC	16	BDT-1-S-10-8BPCMS	26		36
7	BDT-1-S-15-8BPC	17	BDT-1-S-10-8BPCMSD	27		37
8	BDT-1-S-15-2BPC_FD D	18		28		38
9	BDT-1-S-10-10BPC	19		29		39
10	BDT-1-S-10-12BPC	20		30		40

Notes: _____

LDC #: 24163021
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
 Reviewer: _____
 2nd Reviewer: _____

Method: Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was 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				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $< 30\%$ for labeled standards?	/	/		(ending CCV)
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10 ?	/			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?		/		(ending CCV)
Did all routine 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 concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	/			
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			

LDC #: 24163021
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

VIII. Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>
IX. Internal standards			
Were internal standard recoveries within the 40-135% criteria?		<input checked="" type="checkbox"/>	
Was the minimum S/N ratio of all internal standard peaks ≥ 10 ?	<input checked="" type="checkbox"/>		
X. 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?	<input checked="" type="checkbox"/>		
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?	<input checked="" type="checkbox"/>		
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?	<input checked="" type="checkbox"/>		
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>		
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>		
Was the signal to noise ratio for each target compound and labeled standard > 2.5 ?	<input checked="" type="checkbox"/>		
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	<input checked="" type="checkbox"/>		
For PCDF identification, was any signal ($S/N \geq 2.5$, at \pm seconds RT) detected in the corresponding PCDPE channel?	<input checked="" type="checkbox"/>		
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>		
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>		
XII. System performance			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>		
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>		
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>		
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>		
XV. Field blanks			
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>		
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>		

VALIDATION FINDINGS WORKSHEET

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

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
Internal Standards

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".
 X N / N/A Are all internal standard recoveries were within the 40-135% criteria?
 Y N / N/A Was the S/N ratio all internal standard peaks > 10?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		16	I	35 (40-135)	no qual MS
		17	I	34	no qual MS
			G	37	
		2	I	29	MS/P equal G,Q
		3	I	34	G,Q
		5	I	33	G,Q
		6	I	27	G,Q
		9	H	35	F
			I	29	G,Q
			G	34	O,P
		10	H	38	F
			I	30	G,Q
			G	36	O,P
Internal Standards			Check Standard Used	Recovery Standards	Check Standard Used
A	¹³ C-2,3,7,8-TCDF				¹³ C-1,2,3,4-TCDD
B	¹³ C-2,3,7,8-TCDD				
C	¹³ C-1,2,3,7,8-PeCDF				¹³ C-1,2,3,7,8,9-HxCDD
D	¹³ C-1,2,3,7,8-PeCDD				
E	¹³ C-1,2,3,6,7,8-HxCDF				
F	¹³ C-1,2,3,6,7,8-HxCDD				
G	¹³ C-1,2,3,4,6,7,8-HpCDF				
H	¹³ C-1,2,3,4,6,7,8-HpCDD				
I	¹³ C-OCDD				

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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".

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

#	Date	Compound -Sample ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		H	no 2nd column confirmation was performed	4, 6, 8, 9 10, 12, 14, 15	none / p (e)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24163C21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 1
Reviewer: [Signature]
2nd Reviewer: [Signature]

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

Y N NA Were field duplicate pairs identified in this SDG?
Y N NA Were target analytes detected in the field duplicate pairs?

* E M P C

Compound	Concentration (pg/g)		(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	4	8			
F	0.16 *	0.28	0.12	≤2.6	
G	0.95 *	2.7	1.75	≤5.3	
H	0.21	0.14 *	0.07	≤0.53	
K	0.23 *	0.21	0.02	≤2.6	
L	0.24 *	0.16	0.08	≤2.6	
M	0.082	2.6U	2.518	≤2.6	
O	0.53	0.59	0.06	≤2.6	
P	0.24 *	0.16	0.08	≤2.6	
Q	0.97 *	1.1	0.13	≤5.3	

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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	
				Average RRF (initial)	%RSD	Average RRF (initial)	%RSD	RRF (RRF ³ std)	%RSD	RRF (RRF ³ std)	%RSD
1	ICAL	8/30/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.169	5.52	1.169	5.52	1.2609	5.52	1.2609	5.52
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.252	4.0	1.252	4.0	1.2887	4.0	1.2887	4.0
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.165	6.20	1.165	6.20	1.2152	6.20	1.2152	6.20
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.180	5.62	1.180	5.62	1.2654	5.62	1.2654	5.62
			OCDF (¹³ C-OCDF)	1.892	6.95	1.892	6.95	1.9979	6.95	1.9979	6.95
2	ICAL	7/26/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	3.32	1.056	3.32	1.020	3.32	1.020	3.32
	PB225		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.

LDC #: 24163C21
 SDG #: see coms

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: _____

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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 = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = $(A_x)(C_{is}) / (A_{is})(C_x)$ 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	
					RRF (CC)	%D	RRF (CC)	%D
1	cen 22:55	9/30/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.169	1.14	2.4	1.14099	2.4
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.091252	1.09	12.7	1.0936	12.7
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.165	1.19	1.8	1.1861	1.8
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.180	1.20	1.5	1.1974	1.5
			OCDF (¹³ C-OCDF)	1.892	1.60	15.6	1.5974	15.6
2	cen 20:20 PB225	10/4/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	1.15	8.5	1.15	8.5
			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 Routine Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 24163 C2/

SDG #: per coner

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates Results Verification

Page: 1 of 1
Reviewer: F7
2nd Reviewer: [Signature]

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * (SSR - SR) / SA$ Where: SSR = Spiked sample result, SR = Sample result
SA = Spike added

RPD = $|MSR - MSDR| * 2 / (MSR + MSDR)$ MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 16 + 17

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported	Recalculated
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc	RPD	RPD
2,3,7,8-TCDD	211	211	ND	18.3	17.7	87	87	84	84	3.7	3.7
1,2,3,7,8-PeCDD	106	106	ND	93.4	95.6	88	88	91	91	2.4	2.4
1,2,3,4,7,8-HxCDD	106	106	ND	96.6	95.6	91	91	91	91	0.99	0.99
1,2,3,4,7,8,9-HpCDF	106	106	0.34	114	119	108	108	112	112	4.5	4.5
OCDF	211	211	2.3	203	214	95	95	100	100	5.3	5.3

Comments: Refer to Matrix Spike/Matrix Spike Duplicate 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: GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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 = $100 \cdot \text{SSC}/\text{SA}$ Where: SSC = Spiked sample concentration
 SA = Spike added

RPD = $100 \cdot \frac{\text{LCS} - \text{LCS}_D}{\text{LCS} + \text{LCS}_D}$ LCS = Laboratory control sample percent recovery LCS_D = Laboratory control sample duplicate percent recovery

LCS ID: 0260236 LCS

Compound	Spike Added (<u>ppb/g</u>)		Spiked Sample Concentration (<u>ppb/g</u>)		LCS		LCS _D		Percent Recovery		Percent Recovery		RPD	
	LCS	LCS _D	LCS	LCS _D	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	na	17.5	na	88	88	91	91						
1,2,3,7,8-PeCDD	100		90.9		82	82	99	99						
1,2,3,4,7,8-HxCDD	100		81.5		95	95	NA	NA						
1,2,3,4,7,8,9-HpCDF	200		190											
OCDF														

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.

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass ^(a)	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C ₁₂ H ₄ ³⁵ Cl ₄ O	TCDF	4	407.7818	M+2	C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO	HpCDF		
	305.8987	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl ₁₀	TCDF		409.7788	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O	HpCDF		
	315.9419	M	¹⁹ C ₁₂ H ₄ ³⁵ Cl ₄ O	TCDF (S)		417.8250	M	¹⁹ C ₁₂ H ³⁵ Cl ₄ O	HpCDF (S)		
	317.9389	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO	TCDF (S)		419.8220	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO	HpCDF		
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD		423.7767	M+2	¹³ C ₁₂ H ³⁵ Cl ₆ ³⁷ ClO ₂	HpCDD		
	321.8936	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl ₁₀ ₂	TCDD		425.7737	M+2	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂	HpCDD		
	331.9368	M	¹⁹ C ₁₂ H ₄ ³⁵ Cl ₄ O ₂	TCDD (S)		435.8169	M+4	¹⁹ C ₁₂ H ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	HpCDD (S)		
	333.9338	M+2	¹³ C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ Cl ₁₀ ₂	TCDD (S)		437.8140	M+2	¹³ C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂	HpCDD (S)		
	375.8984	M+2	C ₁₂ H ₄ ³⁵ Cl ₃ ³⁷ ClO	HxCDFE		479.7165	M+4	¹³ C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₂ O	HxCDFE		
	[354.9792]	LOCK	C ₉ F ₁₃	PFK		[430.9728]	LOCK	C ₉ F ₁₇	PFK		
	2	339.8597	M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO		PeCDF	5	441.7428	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ ClO	OCDF
		341.8567	M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O		PeCDF		443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	OCDF
		351.9000	M+2	¹³ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO		PeCDF (S)		457.7377	M+2	¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD
353.8970		M+4	¹⁹ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O	PeCDF (S)	459.7348	M+4		¹⁹ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD		
355.8546		M+2	C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂	PeCDD	469.7780	M+2		¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD (S)		
357.8516		M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD	471.7750	M+4		¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD (S)		
367.8949		M+2	¹⁹ C ₁₂ H ₃ ³⁵ Cl ₄ ³⁷ ClO ₂	PeCDD (S)	513.6775	M+4		¹⁹ C ₁₂ ³⁵ Cl ₇ ³⁷ Cl ₂ O	OCDD (S)		
369.8919		M+4	¹³ C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD (S)	[422.9278]	LOCK		C ₁₂ ³⁵ Cl ₈ ³⁷ Cl ₂ O	DCDPE		
409.7974		M+2	C ₁₂ H ₃ ³⁵ Cl ₃ ³⁷ ClO	HpCDPE				C ₁₀ F ₁₇	PFK		
[354.9792]		LOCK	C ₉ F ₁₃	PFK							
3	373.8208	M+2	C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF							
	375.8178	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O	HxCDF							
	383.8639	M	¹⁹ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF (S)							
	385.8610	M+2	C ₁₂ H ₂ ³⁵ Cl ₆ O	HxCDF (S)							
	389.8156	M+2	¹³ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO	HxCDF (S)							
	391.8127	M+4	C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD							
	401.8559	M+2	¹⁹ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD							
	403.8529	M+4	¹³ C ₁₂ H ₂ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD (S)							
	445.7555	M+4	¹⁹ C ₁₂ H ₂ ³⁵ Cl ₅ ³⁷ Cl ₂ O ₂	HxCDD (S)							
	[430.9728]	LOCK	C ₉ F ₁₇	OCDFE							
				PFK							

(a) The following nucleic masses were used:

H = 1.007825
 C = 12.000000
¹³C = 13.003355
 F = 18.9984
 O = 15.994915
³⁵Cl = 34.968853
³⁷Cl = 36.965903

S = internal/recovery standard

