



**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

December 3, 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 25, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 24340:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
G0H210435, G0H250513, G0H270547, G0H310579 G0H310592, G0I010541, G0I020531, G0I030578 G0I040477, G0I070430, G0I100609, G0I100610 G0I130476, G0I180492, G0I230576, G0I250521	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 #: 24340  
 SDG #: G0H210435, G0H250513, G0H270547, G0H310579, G0H310592  
 G0I010541, G0I020531, G0I030578, G0I040477, G0I070430  
 G0I100610, G0I130476, G0I180492, G0I230576, G0I250521

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

Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
<b>I. Completeness</b>				
Is there an EDD for the associated Tronox validation report?	X			
<b>II. EDD Qualifier Population</b>				
Were all qualifiers from the validation report populated into the EDD?	X			
<b>III. EDD Lab Anomalies</b>				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			X	See EDD_discrepancy_form LDC24340_120210.doc
<b>IV. EDD Delivery</b>				
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 16 through August 17, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H210435

### Sample Identification

RSAJ3-0BPC  
SSAI3-02-SW-E-1BPC  
SSAI3-02-SW-E-1BPC\_FD  
SSAI3-03-SW-E-1BPC  
SSAI3-04-SW-E-1BPC  
SSAJ3-02-SW-E-1BPC\*\*  
SSAJ3-05-SW-E-1BPC  
SSAJ3-07-SW-E-1BPC  
SSAJ3-07-SW-E-1BPC\_FD  
SSAI3-02-SW-W-1BPC  
SSAI3-03-SW-W-1BPC  
SSAI3-04-SW-W-1BPC  
SSAJ3-02-SW-W-1BPC  
SSAJ3-05-SW-W-1BPC  
SSAJ3-07-SW-W-1BPC  
SA172-0BPC\*\*  
SA172-0BPC\_FD  
SSAJ3-02-SW-W-1BPCMS  
SSAJ3-02-SW-W-1BPCMSD

\*\*Indicates sample underwent Stage 4 review



## Introduction

This data review covers 19 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.
- UU 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 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
0250250-MB	9/7/10	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.21 pg/g 0.10 pg/g 0.067 pg/g 0.047 pg/g 0.071 pg/g 0.083 pg/g	All samples in SDG G0H210435

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

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
SSAJ3-02-SW-E-1BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 28 (40-135) 34 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAJ3-05-SW-E-1BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 37 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAI3-02-SW-W-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	37 (40-135) 28 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAI3-03-SW-W-1BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 39 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAJ3-02-SW-W-1BPC	<sup>113</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135)	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
SSAJ3-05-SW-W-1BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA172-0BPC**	<sup>13</sup> C-OCDD	32 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA172-0BPC_FD	<sup>13</sup> C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) 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
RSAJ3-0BPC SSAI3-04-SW-W-1BPC	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SSAI3-02-SW-E-1BPC SSAI3-03-SW-E-1BPC	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)	P
SSAI3-02-SW-E-1BPC_FD SSAI3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC	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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAI3-04-SW-E-1BPC	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
SSAJ3-05-SW-E-1BPC SSAJ3-07-SW-W-1BPC	2,3,7,8-TCDF 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)	P
SSAI3-03-SW-W-1BPC	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H210435	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 G0H210435	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 SSAI3-02-SW-E-1BPC and SSAI3-02-SW-E-1BPC\_FD, samples SSAJ3-07-SW-E-1BPC and SSAJ3-07-SW-E-1BPC\_FD, and samples SA172-0BPC\*\* and SA172-0BPC\_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
	SSAI3-02-SW-E-1BPC	SSAI3-02-SW-E-1BPC_FD				
2,3,7,8-TCDD	570	300	62 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDD	2200	1000	75 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDD	1300	780	50 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	2900	1500	64 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	2500	1400	56 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	8800	4900	57 (≤50)	-	J (all detects)	A
OCDD	11000	7500	38 (≤50)	-	-	-
2,3,7,8-TCDF	12000	7500	46 (≤50)	-	-	-
1,2,3,7,8-PeCDF	34000	17000	67 (≤50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	17000	8900	63 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	66000	34000	64 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	46000	22000	71 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	11000	5000	75 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	8300	4100	68 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	150000	69000	74 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	66000	31000	72 (≤50)	-	J (all detects)	A
OCDF	450000	300000	40 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAJ3-07-SW-E-1BPC	SSAJ3-07-SW-E-1BPC_FD				
2,3,7,8-TCDD	0.13	0.14	-	0.01 ( $\leq 0.50$ )	-	-
1,2,3,7,8-PeCDD	0.39	0.41	-	0.02 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDD	0.38	0.38	-	0 ( $\leq 2.5$ )	-	-
1,2,3,6,7,8-HxCDD	1.0	0.97	-	0.03 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDD	0.81	0.66	-	0.15 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDD	3.0	3.0	-	0 ( $\leq 2.5$ )	-	-
OCDD	4.8	8.3	-	3.5 ( $\leq 5.0$ )	-	-
2,3,7,8-TCDF	3.2	3.1	3 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDF	6.4	6.3	-	0.1 ( $\leq 2.5$ )	-	-
2,3,4,7,8-PeCDF	3.1	2.9	-	0.2 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDF	15	9.3	-	5.7 ( $\leq 2.5$ )	J (all detects)	A
1,2,3,6,7,8-HxCDF	9.5	9.9	-	0.4 ( $\leq 2.5$ )	-	-
2,3,4,6,7,8-HxCDF	2.8	2.5	-	0.3 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDF	1.1	1.5	-	0.4 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDF	30	32	6 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	11	12	-	1 ( $\leq 2.5$ )	-	-
OCDF	71	74	4 ( $\leq 50$ )	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SA172-0BPC**	SA172-0BPC_FD				
1,2,3,7,8-PeCDD	2.5U	0.57	-	1.93 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDD	0.33	0.42	-	0.09 ( $\leq 2.5$ )	-	-
1,2,3,6,7,8-HxCDD	0.78	1.1	-	0.32 ( $\leq 2.5$ )	-	-



Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SA172-0BPC**	SA172-0BPC_FD				
1,2,3,7,8,9-HxCDD	0.46	0.67	-	0.21 (≤2.5)	-	-
1,2,3,4,6,7,8-HpCDD	2.8	3.7	-	0.9 (≤2.5)	-	-
OCDD	25	9.5	-	15.5 (≤5.0)	J (all detects)	A
2,3,7,8-TCDF	2.8	4.3	42 (≤50)	-	-	-
1,2,3,7,8-PeCDF	5.2	8.4	-	3.2 (≤2.5)	J (all detects)	A
2,3,4,7,8-PeCDF	2.8	4.3	-	1.5 (≤2.5)	-	-
1,2,3,4,7,8-HxCDF	8.8	11	-	2.2 (≤2.5)	-	-
1,2,3,6,7,8-HxCDF	6.9	11	-	4.1 (≤2.5)	J (all detects)	A
2,3,4,6,7,8-HxCDF	1.7	2.9	-	1.2 (≤2.5)	-	-
1,2,3,7,8,9-HxCDF	1.4	1.9	-	0.5 (≤2.5)	-	-
1,2,3,4,6,7,8-HpCDF	26	42	47 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	11	19	-	8 (≤2.5)	-	-
OCDF	73	120	49 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H210435**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H210435	SSAJ3-02-SW-E-1BPC**	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H210435	SSAJ3-05-SW-E-1BPC SSAI3-03-SW-W-1BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H210435	SSAI3-02-SW-W-1BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H210435	SSAJ3-02-SW-W-1BPC	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	Internal standards (%R) (i)
G0H210435	SSAJ3-05-SW-W-1BPC SA172-0BPC** SA172-0BPC_FD	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H210435	RSAJ3-0BPC SSAI3-04-SW-W-1BPC	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H210435	SSAI3-02-SW-E-1BPC SSAI3-03-SW-E-1BPC	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)	P	Project Quantitation Limit (exceeded range) (e)
G0H210435	SSAI3-02-SW-E-1BPC_FD SSAI3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H210435	SSAI3-04-SW-E-1BPC	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)
G0H210435	SSAJ3-05-SW-E-1BPC SSAJ3-07-SW-W-1BPC	2,3,7,8-TCDF 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)	P	Project Quantitation Limit (exceeded range) (e)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H210435	SSAI3-03-SW-W-1BPC	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)
G0H210435	RSAJ3-0BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC_FD SSAI3-03-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAJ3-02-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAJ3-07-SW-E-1BPC SSAJ3-07-SW-E-1BPC_FD SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAJ3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC SA172-0BPC** SA172-0BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H210435	RSAJ3-0BPC SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC_FD SSAI3-03-SW-E-1BPC SSAI3-04-SW-E-1BPC SSAJ3-02-SW-E-1BPC** SSAJ3-05-SW-E-1BPC SSAJ3-07-SW-E-1BPC SSAJ3-07-SW-E-1BPC_FD SSAI3-02-SW-W-1BPC SSAI3-03-SW-W-1BPC SSAI3-04-SW-W-1BPC SSAJ3-02-SW-W-1BPC SSAJ3-05-SW-W-1BPC SSAJ3-07-SW-W-1BPC SA172-0BPC** SA172-0BPC_FD	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H210435	SSAI3-02-SW-E-1BPC SSAI3-02-SW-E-1BPC_FD	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 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) 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)
G0H210435	SSAJ3-07-SW-E-1BPC SSAJ3-07-SW-E-1BPC_FD	1,2,3,4,7,8-HxCDF	J (all detects)	A	Field duplicates (Difference) (fd)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H210435	SA172-0BPC** SA172-0BPC_FD	OCDD 1,2,3,7,8-PeCDF 1,2,3,6,7,8-HxCDF	J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0H210435**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H210435.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340A21  
 SDG #: G0H210435  
 Laboratory: Test America

Stage 2B/4

Date: 11/19/10  
 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	△	Sampling dates: 8/16 - 8/17/10
II.	HRGC/HRMS Instrument performance check	△	
III.	Initial calibration	△	
IV.	Routine calibration/ECV	△	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	△	LC5
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	△	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	△	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 2, 3 8, 9 14, 17
XV.	Field blanks	N	

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

Validated Samples: \*\* Indicates sample underwent Stage 4 validation

soil

1	RSAJ3-0BPC	11	SSAI3-03-SW-W-1BPC	21	025 0250	31	
2	SSAI3-02-SW-E-1BPC	12	SSAI3-04-SW-W-1BPC	22		32	
3	SSAI3-02-SW-E-1BPC_FD	13	SSAJ3-02-SW-W-1BPC	23		33	
4	SSAI3-03-SW-E-1BPC	14	SSAJ3-05-SW-W-1BPC	24		34	
5	SSAI3-04-SW-E-1BPC	15	SSAJ3-07-SW-W-1BPC	25		35	
6	SSAJ3-02-SW-E-1BPC**	16	SA172-0BPC**	26		36	
7	SSAJ3-05-SW-E-1BPC	17	SA172-0BPC_FD	27		37	
8	SSAJ3-07-SW-E-1BPC	18	SSAJ3-02-SW-W-1BPCMS	28		38	
9	SSAJ3-07-SW-E-1BPC_FD	19	SSAJ3-02-SW-W-1BPCMSD	29		39	
10	SSAI3-02-SW-W-1BPC	20		30		40	

Notes: 16

LDC #: 24340A21  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: FJ  
 2nd Reviewer: N

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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"/>	
<b>III. Initial calibration</b>				
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 $< 30\%$ 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 $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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"/>	
<b>VII. Laboratory control samples</b>				
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 #: 24340A21  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: JA

<b>VIII: Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>
<b>IX: Internal standards</b>			
Were internal standard recoveries within the 40-135% criteria?			<input checked="" type="checkbox"/>
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?			<input checked="" type="checkbox"/>
<b>X: Target compound identification</b>			
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 $\pm 2$ 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 PCDF channel?			<input checked="" type="checkbox"/>
Was an acceptable lock mass recorded and monitored?			<input checked="" type="checkbox"/>
<b>XI: Compound quantitation/CRQLs</b>			
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"/>
<b>XII: System performance</b>			
System performance was found to be acceptable.			<input checked="" type="checkbox"/>
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.			<input checked="" type="checkbox"/>
<b>XIV: Field duplicates</b>			
Field duplicate pairs were identified in this SDG.			<input checked="" type="checkbox"/>
Target compounds were detected in the field duplicates.			<input checked="" type="checkbox"/>
<b>XV: Field blanks</b>			
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**  
**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).

(e)

#	Date	Compound Sample ID	Finding	Associated Samples	Qualifications
		H, Q	recal Range	1, 12	J/P det
		H, K, e, P, Q		2, 4	
		e, Q		3, 10, 14	
		H, e, P, Q		4, 5	
		H, K, L, e, P, Q		7, 15	
		H, I, K, L, e, P, Q		11	

Comments: See sample calculation verification worksheet for recalculations



**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

**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?

(fd)

Compound	Concentration (pg/g)		%RSD ≤50	Qualifications (Parent Only)
	2	3		
A	570	300	62	J/A DET
B	2200	1000	75	J/A DET
C	1300	780	50	
D	2900	1500	64	J/A DET
E	2500	1400	56	J/A DET
F	8800	4900	57	J/A DET
G	11000	7500	38	
H	12000	7500	46	
I	34000	17000	67	J/A DET
J	17000	8900	63	J/A DET
K	66000	34000	64	J/A DET
L	46000	22000	71	J/A DET
M	11000	5000	75	J/A DET
N	8300	4100	68	J/A DET
O	150000	69000	74	J/A DET
P	66000	31000	72	J/A DET
Q	450000	300000	40	

Field Duplicates

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)
	8	9				
A	0.13	0.14		0.01	≤0.50	
B	0.39	0.41		0.02	≤2.5	
C	0.38	0.38		0	≤2.5	
D	1.0	0.97		0.03	≤2.5	
E	0.81	0.66		0.15	≤2.5	
F	3.0	3.0		0	≤2.5	
G	4.8	8.3		3.5	≤5.0	
H	3.2	3.1	3			
I	6.4	6.3		0.1	≤2.5	
J	3.1	2.9		0.2	≤2.5	
K	15	9.3		5.7	≤2.5	J/A DET
L	9.5	9.9		0.4	≤2.5	
M	2.8	2.5		0.3	≤2.5	
N	1.1	1.5		0.4	≤2.5	
O	30	32	6			
P	11	12		1	≤2.5	
Q	71	74	4			



**VALIDATION FINDINGS WORKSHEET**  
Field Duplicates

**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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	16	17				
B	2.5U	0.57		1.93	≤2.5	
C	0.33	0.42		0.09	≤2.5	
D	0.78	1.1		0.32	≤2.5	
E	0.46	0.67		0.21	≤2.5	
F	2.8	3.7		0.9	≤2.5	
G	25	9.5		15.5	≤5.0	J/A DET
H	2.8	4.3	42			
I	5.2	8.4		3.2	≤2.5	J/A DET
J	2.8	4.3		1.5	≤2.5	
K	8.8	11		2.2	≤2.5	
L	6.9	11		4.1	≤2.5	J/A DET
M	1.7	2.9		1.2	≤2.5	
N	1.4	1.9		0.5	≤2.5	
O	26	42	47			
P	11	19		8	≤2.5	
Q	73	120	49			

V:\FIELD DUPLICATES\24340A21.wpd

**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:

$RRF = (A_s)(C_s)/(A_x)(C_x)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$

$A_s$  = Area of compound,  
 $C_s$  = Concentration of compound,  
 $S$  = Standard deviation of the RRFs,  
 $A_x$  = Area of associated internal standard  
 $C_x$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (initial)	(LS) 3 std	Average RRF (initial)	(LS) 3 std	%RSD	%RSD	RRF	(LS) 3 std
1	ICAL	8/30/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.2609	1.169	1.2609	5.52	5.52	1.2609	5.52
	PBS		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.2587	1.252	1.2587	4.0	4.0	1.2587	4.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.2152	1.165	1.2152	6.20	6.20	1.2152	6.20
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.2654	1.180	1.2654	5.62	5.62	1.2654	5.62
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.9979	1.892	1.9979	6.95	6.95	1.9979	6.95
2	ICAL	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.052	1.020	1.052	1.020	3.32	3.32	1.020	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

**VALIDATION FINDINGS WORKSHEET**  
**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 =  $(A_s)(C_b) / (A_b)(C_s)$  RRF = continuing calibration RRF  
 $A_s$  = Area of compound,  $A_b$  = Area of associated internal standard  
 $C_s$  = 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	15SE1011P5	9/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.09	7.1	1.09	7.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.15	8.1	1.15	8.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.22	4.9	1.22	4.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.17	0.5	1.17	0.5
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.80	4.8	1.80	4.8
2	09SE10A11 511	9/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.12	1.12	4.5	1.12	4.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.11	1.11	11.6	1.11	11.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.16	1.16	0.4	1.16	0.4
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.15	1.15	2.4	1.15	2.4
			OCDF ( <sup>13</sup> C-OCDF)	1.76	1.76	6.8	1.76	6.8
3	12SE105D2 DB 225	9/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.052	0.97	7.9	0.97	7.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
4	13SE105D2	9/13/10	OCDF ( <sup>13</sup> C-OCDF) 2, 3, 7, 8-TCDF	1.052	0.93	11.9	0.93	11.9

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.

**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 =  $100 * |MSR - MSDR| * 2 / (MSR + MSDR)$

MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 18 + 19

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported	Recalculated
	MS	MSD		MS	MSD	Percent Recovery	Recalc	Percent Recovery	Recalc		
2,3,7,8-TCDD	20.2	19.3	3.7	23.0	19.0	95	95	79	79	19	19
1,2,3,7,8-PeCDD	19.1	96.5	14	116	100	101	101	89	89	15	15
1,2,3,4,7,8-HxCDD	↓	↓	12	136	116	122	122	108	108	15	15
1,2,3,4,7,8,9-HpCDF	↓	↓	42.0	785	352	365	365	0	0	76	76
OCDF											

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.



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	NCDPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	OCDF
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF		
355.8546		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD		
357.8516		M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO	HpCDFE				C <sub>9</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> O	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDD (S)							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	OCDFE							
				PFK							

(a) The following nuclidic masses were used:

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

S = Internal/recovery standard

LDC #: 2434042  
 SDG #: pe cover

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification

Page: 1 of 1  
 Reviewer: FJ  
 2nd reviewer: J

**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<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = 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. #6, 2, 3, 7, 8-TCDF

$$\text{Conc.} = \frac{(3640060)(2000)}{(151238200)(1.25)(9.99)(0.9943)}$$

$$= 3.85 \text{ pg/g}$$

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#6 2, 3, 7, 8 TCDF			
		= 24493528 (2000)			
		452521184 (1.06)(9.99)(0.9943)			
		= 103 pg/g			

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

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

**Collection Date:** August 23, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H250513

**Sample Identification**

BDT-3-N-15-2BPC	BDT-3-N-15-2BPCMS
BDT-3-N-15-4BPC	BDT-3-N-15-2BPCMSD
BDT-3-N-15-6BPC	
BDT-3-N-15-8BPC	
BDT-3-N-15-10BPC	
BDT-3-N-15-12BPC	
BDT-3-N-15-14BPC	
BDT-3-N-15-16BPC	
BDT-3-N-15-18BPC**	
BDT-3-N-15-6BPC_FD	
BDT-3-N-5-10BPC	
BDT-3-N-5-12BPC	
BDT-3-N-5-14BPC	
BDT-3-N-5-16BPC	
BDT-3-N-5-18BPC**	
BDT-3-N-5-2BPC	
BDT-3-N-5-4BPC	
BDT-3-N-5-6BPC	
BDT-3-N-5-8BPC_FD	
BDT-3-N-5-8BPC	

\*\*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 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
0250288-MB	9/7/10	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 OCDF	0.12 pg/g 0.086 pg/g 0.13 pg/g 0.40 pg/g 1.8 pg/g 0.14 pg/g 0.16 pg/g 0.11 pg/g 0.10 pg/g 0.28 pg/g 0.49 pg/g	All samples in SDG G0H250513

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-N-15-4BPC	OCDD	5.1 pg/g	5.1U pg/g
BDT-3-N-15-8BPC	1,2,3,4,7,8-HxCDD	0.46 pg/g	0.46U pg/g
BDT-3-N-15-10BPC	1,2,3,4,7,8-HxCDD	0.47 pg/g	0.47U pg/g
BDT-3-N-15-12BPC	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.081 pg/g 0.24 pg/g 0.21 pg/g 0.92 pg/g 3.5 pg/g 0.35 pg/g 0.26 pg/g	0.081U pg/g 0.24U pg/g 0.21U pg/g 0.92U pg/g 3.5U pg/g 0.35U pg/g 0.26U pg/g
BDT-3-N-15-16BPC	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 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.072 pg/g 0.34 pg/g 0.20 pg/g 1.9 pg/g 0.49 pg/g 0.14 pg/g 0.11 pg/g	0.072U pg/g 0.34U pg/g 0.20U pg/g 1.9U pg/g 0.49U pg/g 0.14U pg/g 0.11U pg/g
BDT-3-N-15-18BPC**	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 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	0.12 pg/g 0.43 pg/g 0.56 pg/g 0.38 pg/g 0.090 pg/g 0.11 pg/g 1.2 pg/g	0.12U pg/g 0.43U pg/g 0.56U pg/g 0.38U pg/g 0.090U pg/g 0.11U pg/g 1.2U pg/g

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-N-15-4BPC	<sup>13</sup> C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-15-8BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 20 (40-135) 31 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-3-N-15-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 22 (40-135) 33 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-3-N-15-12BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	24 (40-135) 38 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
BDT-3-N-15-14BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-15-16BPC	<sup>13</sup> C-OCDD	36 (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-3-N-15-6BPC_FD	<sup>13</sup> C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-12BPC	<sup>13</sup> C-OCDD	36 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-14BPC	<sup>13</sup> C-OCDD	37 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-16BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-18BPC**	<sup>13</sup> C-OCDD	34 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-2BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-3-N-5-8BPC_FD	<sup>13</sup> C-OCDD	36 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) 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-N-15-2BPC	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

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-3-N-5-4BPC	2,3,7,8-TCDF 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)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H250513	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 G0H250513	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-N-15-6BPC and BDT-3-N-15-6BPC\_FD and samples BDT-3-N-5-8BPC and BDT-3-N-5-8BPC\_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-N-15-6BPC	BDT-3-N-15-6BPC_FD				
2,3,7,8-TCDD	1.5	0.82	-	0.68 (≤0.55)	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-N-15-6BPC	BDT-3-N-15-6BPC_FD				
1,2,3,7,8-PeCDD	4.2	2.3	-	1.9 (≤2.7)	-	-
1,2,3,4,7,8-HxCDD	1.7	1.6	-	0.1 (≤2.7)	-	-
1,2,3,6,7,8-HxCDD	3.4	3.0	-	0.4 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	2.8	2.6	-	0.2 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	9.0	9.4	-	0.4 (≤2.7)	-	-
OCDD	9.9	14	-	4.1 (≤5.5)	-	-
2,3,7,8-TCDF	41	16	88 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	56	28	67 (≤50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	30	15	67 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	69	59	16 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	49	36	31 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	10	6.9	-	3.1 (≤2.7)	J (all detects)	A
1,2,3,7,8,9-HxCDF	11	6.6	-	4.4 (≤2.7)	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	130	120	8 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	68	60	13 (≤50)	-	-	-
OCDF	320	350	9 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-N-5-8BPC_FD	BDT-3-N-5-8BPC				
2,3,7,8-TCDD	1.9	1.1	-	0.8 (≤0.52)	J (all detects)	A
1,2,3,7,8-PeCDD	4.8	3.9	-	0.9 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	2.9	2.6	-	0.3 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	5.8	5.3	-	0.5 (≤2.6)	-	-



Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-3-N-5-8BPC_FD	BDT-3-N-5-8BPC				
1,2,3,7,8,9-HxCDD	5.7	4.9	-	0.8 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	18	19	3 (≤50)	-	-	-
OCDD	21	22	-	1 (≤5.2)	-	-
2,3,7,8-TCDF	67	24	95 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	67	49	31 (≤50)	-	-	-
2,3,4,7,8-PeCDF	34	26	27 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	110	110	0 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	72	71	1 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	16	14	13 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	13	13	0 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	240	250	4 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	120	110	9 (≤50)	-	-	-
OCDF	650	640	2 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H250513**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H250513	BDT-3-N-15-4BPC BDT-3-N-15-14BPC BDT-3-N-15-16BPC BDT-3-N-15-6BPC_FD BDT-3-N-5-12BPC BDT-3-N-5-14BPC BDT-3-N-5-16BPC BDT-3-N-5-18BPC** BDT-3-N-5-2BPC BDT-3-N-5-8BPC_FD	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H250513	BDT-3-N-15-8BPC BDT-3-N-15-10BPC	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H250513	BDT-3-N-15-12BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H250513	BDT-3-N-15-2BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H250513	BDT-3-N-5-4BPC	2,3,7,8-TCDF 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)	P	Project Quantitation Limit (exceeded range) (e)
G0H250513	BDT-3-N-15-2BPC BDT-3-N-15-4BPC BDT-3-N-15-6BPC BDT-3-N-15-8BPC BDT-3-N-15-10BPC BDT-3-N-15-12BPC BDT-3-N-15-14BPC BDT-3-N-15-16BPC BDT-3-N-15-18BPC** BDT-3-N-15-6BPC_FD BDT-3-N-5-10BPC BDT-3-N-5-12BPC BDT-3-N-5-14BPC BDT-3-N-5-16BPC BDT-3-N-5-18BPC** BDT-3-N-5-2BPC BDT-3-N-5-4BPC BDT-3-N-5-6BPC BDT-3-N-5-8BPC_FD BDT-3-N-5-8BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H250513	BDT-3-N-15-2BPC BDT-3-N-15-4BPC BDT-3-N-15-6BPC BDT-3-N-15-8BPC BDT-3-N-15-10BPC BDT-3-N-15-12BPC BDT-3-N-15-14BPC BDT-3-N-15-16BPC BDT-3-N-15-18BPC** BDT-3-N-15-6BPC_FD BDT-3-N-5-10BPC BDT-3-N-5-12BPC BDT-3-N-5-14BPC BDT-3-N-5-16BPC BDT-3-N-5-18BPC** BDT-3-N-5-2BPC BDT-3-N-5-4BPC BDT-3-N-5-6BPC BDT-3-N-5-8BPC_FD BDT-3-N-5-8BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H250513	BDT-3-N-15-6BPC BDT-3-N-15-6BPC_FD	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF	J (all detects) J (all detects) J (all detects)	A	Field duplicates (RPD) (fd)
G0H250513	BDT-3-N-15-6BPC BDT-3-N-15-6BPC_FD	2,3,7,8-TCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)
G0H250513	BDT-3-N-5-8BPC_FD BDT-3-N-5-8BPC	2,3,7,8-TCDD	J (all detects)	A	Field duplicates (Difference) (fd)
G0H250513	BDT-3-N-5-8BPC_FD BDT-3-N-5-8BPC	2,3,7,8-TCDF	J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0H250513**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H250513	BDT-3-N-15-4BPC	OCDD	5.1U pg/g	A	bl
G0H250513	BDT-3-N-15-8BPC	1,2,3,4,7,8-HxCDD	0.46U pg/g	A	bl
G0H250513	BDT-3-N-15-10BPC	1,2,3,4,7,8-HxCDD	0.47U pg/g	A	bl
G0H250513	BDT-3-N-15-12BPC	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.081U pg/g 0.24U pg/g 0.21U pg/g 0.92U pg/g 3.5U pg/g 0.35U pg/g 0.26U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H250513	BDT-3-N-15-16BPC	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 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.072U pg/g 0.34U pg/g 0.20U pg/g 1.9U pg/g 0.49U pg/g 0.14U pg/g 0.11U pg/g	A	bl
G0H250513	BDT-3-N-15-18BPC**	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 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	0.12U pg/g 0.43U pg/g 0.56U pg/g 0.38U pg/g 0.090U pg/g 0.11U pg/g 1.2U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H250513.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24340B21

VALIDATION COMPLETENESS WORKSHEET

SDG #: G0H250513

Stage 2B/4

Laboratory: Test America

Date: 11/19/10

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: 8/23/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration#CV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	105
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 = 3, 10 P = 19, 20
XV.	Field blanks	N	

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

1	BDT-3-N-15-2BPC	11	BDT-3-N-5-10BPC	21	BDT-3-N-15-2BPCMS	31	0250288
2	BDT-3-N-15-4BPC	12	BDT-3-N-5-12BPC	22	BDT-3-N-15-2BPCMSD	32	
3	BDT-3-N-15-6BPC D	13	BDT-3-N-5-14BPC	23		33	
4	BDT-3-N-15-8BPC	14	BDT-3-N-5-16BPC	24		34	
5	BDT-3-N-15-10BPC	15	BDT-3-N-5-18BPC**	25		35	
6	BDT-3-N-15-12BPC	16	BDT-3-N-5-2BPC	26		36	
7	BDT-3-N-15-14BPC	17	BDT-3-N-5-4BPC	27		37	
8	BDT-3-N-15-16BPC	18	BDT-3-N-5-6BPC	28		38	
9	BDT-3-N-15-18BPC**	19	BDT-3-N-5-8BPC FD D	29		39	
10	BDT-3-N-15-6BPC FD D	20	BDT-3-N-5-8BPC D	30		40	

Notes: 16

LDC #: 2434082  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: FJ  
 2nd Reviewer: J

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
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?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	/			
<b>IV. Continuing calibration</b>				
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?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
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?	/			
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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?	/			
<b>VII. Laboratory control samples</b>				
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 #: 24340B21  
 SDG #: mu con

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: EA  
 2nd Reviewer: J

<b>VIII: Regional Quality Assurance and Quality Control</b>			
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"/>
<b>IX: Internal standards</b>			
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"/>
<b>X: Target compound identification</b>			
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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE 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"/>
<b>XI: Compound quantitation/CRQLs</b>			
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"/>
<b>XII: System performance</b>			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIV: Field duplicates</b>			
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"/>
<b>XV: Field blanks</b>			
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:



(bl)

\* EMRC

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 all samples associated with a method blank?

Y N N/A  
Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A  
Was the method blank contaminated?

Blank extraction date: 9/7/10 Blank analysis date: 9/18/10 Associated samples: A 11

Conc. units: pg/g

Compound	Blank ID	Sample Identification													
		5X	Z	4	5	6	8	9							
C	0.12 *	0.4		0.46/u	0.47/u	0.08*/u	0.072*/u	0.12*/u							
P	0.086 *	0.43				0.24*/u	0.34/u								
E	0.13	0.65				0.21/u	0.20/u	0.43/u							
F	0.40	2.0				0.92/u	1.9/u								
G	1.8	9.0	5.1/u			3.5/u									
K	0.14	0.70													
L	0.16 *	0.8						0.56/u							
M	0.11 *	0.55						0.38/u	0.49/u						
N	0.10	0.5				0.35/u	0.14/u	0.090*/u	0.11*/u						
O	0.28	1.4				0.26*/u	0.11*/u	0.11*/u							
R	0.49	2.45						1.2/u							

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



**VALIDATION FINDINGS WORKSHEET**  
**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?

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

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications																																								
		2	I	35 ( 40-135 )	2/11/11 P G, R																																								
		4	H	33	F																																								
			I	20	G, R																																								
			G	3	O, P																																								
		5	H	35	F																																								
			I	22	G, R																																								
			G	23	O, P																																								
		6	I	24	G, R																																								
			G	38	O, P																																								
		7	I	37	G, R																																								
		8	I	36	G, R																																								
		10	I	35	G, R																																								
		12	I	36	G, R																																								
<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>A. <sup>13</sup>C-2,3,7,8-TCDF</td> <td></td> <td>K. <sup>13</sup>C-1,2,3,4-TCDD</td> <td></td> </tr> <tr> <td>B. <sup>13</sup>C-2,3,7,8-TCDD</td> <td></td> <td>L. <sup>13</sup>C-1,2,3,7,8,9-HxCDD</td> <td></td> </tr> <tr> <td>C. <sup>13</sup>C-1,2,3,7,8-PeCDF</td> <td></td> <td>M.</td> <td></td> </tr> <tr> <td>D. <sup>13</sup>C-1,2,3,7,8-PeCDD</td> <td></td> <td>N.</td> <td></td> </tr> <tr> <td>E. <sup>13</sup>C-1,2,3,6,7,8-HxCDF</td> <td></td> <td>O.</td> <td></td> </tr> <tr> <td>F. <sup>13</sup>C-1,2,3,6,7,8-HxCDD</td> <td></td> <td>P.</td> <td></td> </tr> <tr> <td>G. <sup>13</sup>C-1,2,3,4,6,7,8-HpCDF</td> <td></td> <td>Q.</td> <td></td> </tr> <tr> <td>H. <sup>13</sup>C-1,2,3,4,6,7,8-HpCDD</td> <td></td> <td>R.</td> <td></td> </tr> <tr> <td>I. <sup>13</sup>C-OCDD</td> <td></td> <td>T.</td> <td></td> </tr> </tbody> </table>						Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used	A. <sup>13</sup> C-2,3,7,8-TCDF		K. <sup>13</sup> C-1,2,3,4-TCDD		B. <sup>13</sup> C-2,3,7,8-TCDD		L. <sup>13</sup> C-1,2,3,7,8,9-HxCDD		C. <sup>13</sup> C-1,2,3,7,8-PeCDF		M.		D. <sup>13</sup> C-1,2,3,7,8-PeCDD		N.		E. <sup>13</sup> C-1,2,3,6,7,8-HxCDF		O.		F. <sup>13</sup> C-1,2,3,6,7,8-HxCDD		P.		G. <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		Q.		H. <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		R.		I. <sup>13</sup> C-OCDD		T.	
Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used																																										
A. <sup>13</sup> C-2,3,7,8-TCDF		K. <sup>13</sup> C-1,2,3,4-TCDD																																											
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I. <sup>13</sup> 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".

(e)

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	compd Sample ID	Finding	Associated Samples	Qualifications
		0, R	x'd cal Range	1	1/P det
		H, K, L, O, P, Q	↓	17	↓

Comments: See sample calculation verification worksheet for recalculations

**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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	10				
A	1.5	0.82		0.68	≤0.55	J/A det
B	4.2	2.3		1.9	≤2.7	
C	1.7	1.6		0.1	≤2.7	
D	3.4	3.0		0.4	≤2.7	
E	2.8	2.6		0.2	≤2.7	
F	9.0	9.4		0.4	≤2.7	
G	9.9	14		4.1	≤5.5	
H	41	16	88			J/A det
I	56	28	67			J/A det
J	30	15	67			J/A det
K	69	59	16			
L	49	36	31			
M	10	6.9		3.1	≤2.7	J/A det
N	11	6.6		4.4	≤2.7	J/A det
O	130	120	8			
P	68	60	13			
Q	320	350	9			

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LDC#: 24340B21

**VALIDATION FINDINGS WORKSHEET**  
Field Duplicates

Page: 2 of 2  
 Reviewer: FT  
 2nd Reviewer: ✓

**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)
	19	20				
A	1.9	1.1		0.8	≤0.52	J/A det
B	4.8	3.9		0.9	≤2.6	
C	2.9	2.6		0.3	≤2.6	
D	5.8	5.3		0.5	≤2.6	
E	5.7	4.9		0.8	≤2.6	
F	18	19	3			
G	21	22		1	≤5.2	
H	67	24	95			J/A det
I	67	49	31			
J	34	26	27			
K	110	110	0			
L	72	71	1			
M	16	14	13			
N	13	13	0			
O	240	250	4			
P	120	110	9			
Q	650	640	2			

V:\FIELD DUPLICATES\24340B21.wpd



**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,  
 $X$  = Mean 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)	Average RRF (initial)	RRF (CS std)	RRF (CS std)	%RSD	%RSD	
1	1 CAL	9/18/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.02402	1.02402	1.14942	1.14942	9.91493	9.91493	9.91493	9.91493
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.8998	1.8998	1.18451	1.18451	4.16239	4.16239	4.16239	4.16239
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.16375	1.16375	1.23468	1.23468	7.14786	7.14786	7.14786	7.14786
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.08281	1.08281	1.16859	1.16859	6.58153	6.58153	6.58153	6.58153
			OCDF ( <sup>13</sup> C-OCDF)	1.54183	1.54183	1.64541	1.64541	8.11137	8.11137	8.11137	8.11137
2	1 CAL PBNS	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.020	1.020	3.32	3.32	3.32	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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 #: 24340B21  
 SDG #: see com

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration Results Verification**

Page: 1 of 1  
 Reviewer: F7  
 2nd Reviewer: R

**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	205E103D5	9/20/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.022408	0.96616	5.7	0.96616	5.7
	52		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.8998	0.98152	17.5	0.98152	17.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.16375	1.06478	8.5	1.06478	8.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.08281	1.00218	7.4	1.00218	7.4
			OCDF ( <sup>13</sup> C-OCDF)	1.54183	1.44342	6.4	1.44342	6.4
2	205E103D5	9/20/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	/	0.96261	6.0	0.96261	6.0
	516		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	/	1.05940	11.0	1.05940	11.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	/	1.08399	6.9	1.08399	6.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	/	0.99281	8.3	0.99281	8.3
			OCDF ( <sup>13</sup> C-OCDF)	/	1.44137	6.5	1.44137	6.5
3	215E105D2	9/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.052	1.06	0.1	1.06	0.1
	DB225		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

**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 =  $100 * (MSR - MSDR) / (MSR + MSDR)$       MSR = Matrix spike percent recovery, MSDR = Matrix spike duplicate percent recovery  
 MS/MSD samples: 21 + 22

Compound	Spike Added ( <u>107g</u> )		Sample Concentration ( <u>101g</u> )	Spiked Sample Concentration ( <u>101g</u> )		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	195	215	7.4	27.7	29.3	107	107	102	102	0.37	0.37
1,2,3,7,8-PeCDD	97.3	107	2.6	13.8	14.4	116	116	110	110	1.5	1.5
1,2,3,4,7,8-HxCDD	↓	↓	15	131	12.8	120	120	105	105	8.0	8.0
1,2,3,4,7,8,9-HpCDF	↓	↓	68.0	767	58.2	131	131	0	0	0	27
OCDF	195	215	380.0	356.0	301.0	0	0	0	0	0	17

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 I**  
**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: 0250288-1C2

Compound	Spike Added (pg)		Spiked Sample Concentration (pg/g)		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	21.5	NA	107	107	107	107					
1,2,3,7,8-PeCDD	100.0		115		115	115	115	115						
1,2,3,4,7,8-HxCDD	100.0		105		105	105	105	105						
1,2,3,4,7,8,9-HpCDF	100.0		131		131	131	131	131						
OCDF	200	↓	227	↓	113	113	113	113	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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte			
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF			
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		M+4	409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		M	417.8250	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		M+2	419.8220	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		M+2	423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		M+4	425.7737	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		M+2	435.8169	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		M+4	437.8140	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE		M+4	479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	NCDPE		
	[354.9792]	LOCK	C <sub>8</sub> F <sub>13</sub>	PFK		LOCK	[430.9728]	LOCK	C <sub>8</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	OCDF	
		341.8567	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		M+4	443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		M+2	457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	M+4	459.7348		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	M+2	469.7780		M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	M+4	471.7750		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDD (S)	M+4	513.6775		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
369.8919		M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	M+4	[422.9278]		LOCK	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDFE	LOCK				C <sub>10</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>8</sub> F <sub>13</sub>	PFK								
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF							
		383.8639	M	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> O	HxCDF (S)							
	385.8610	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)								
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	401.8559	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDD								
	403.8529	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)								
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDD (S)								
	[430.9728]	LOCK	C <sub>8</sub> F <sub>17</sub>	OCDFE								
				PFK								

(e) The following nuclidic masses were used:

- H = 1.007825
- C = 12.000000
- <sup>13</sup>C = 13.003355
- F = 18.9984
- O = 15.994915
- <sup>35</sup>Cl = 34.968853
- <sup>37</sup>Cl = 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 25, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H270547

**Sample Identification**

EB-08252010

## Introduction

This data review covers 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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.
- UU 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% .

## **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.

## **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 samples in SDG G0H270547

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-08252010	1,2,3,4,6,7,8-HpCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	4.7 pg/L 1.7 pg/L 1.2 pg/L 7.2 pg/L	4.7U pg/L 1.7U pg/L 1.2U pg/L 7.2U pg/L

Sample EB-08252010 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-08252010	8/25/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	4.7 pg/L 110 pg/L 1.7 pg/L 1.2 pg/L 7.2 pg/L 7.7 pg/L	No associated samples in this SDG

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

All internal standard recoveries were within QC limits.

### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H270547	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H270547	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

### XII. System Performance

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H270547**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H270547	EB-08252010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H270547	EB-08252010	All compounds reported as 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 G0H270547**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H270547	EB-08252010	1,2,3,4,6,7,8-HpCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	4.7U pg/L 1.7U pg/L 1.2U pg/L 7.2U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0H270547**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340C21

SDG #: G0H270547

Laboratory: Test America

Stage 2B

Date: 11/19/10

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: 8/25/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/CEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	QL Sample
VII.	Laboratory control samples	A	LCs
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	EB = 1

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

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

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples:

water

1	EB-08252010	11	02-46227-MB	21		31	
2		12		22		32	
3		13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: 16

# 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:

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## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** August 27, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H310579

### Sample Identification

BDT-1-N-20-10BPC	BDT-1-N-10-8BPC
BDT-1-N-20-12BPC	BDT-1-N-10-2BPC
BDT-1-N-20-14BPC	BDT-1-N-10-8BPC_FD
BDT-1-N-20-2BPC	BDT-1-N-15-4BPCMS
BDT-1-N-20-4BPC	BDT-1-N-15-4BPCMSD
BDT-1-N-20-6BPC**	BDT-1-N-10-8BPCMS
BDT-1-N-20-8BPC	BDT-1-N-10-8BPCMSD
BDT-1-N-20-10BPC_FD	
BDT-1-N-15-10BPC	
BDT-1-N-15-12BPC	
BDT-1-N-15-14BPC	
BDT-1-N-15-8BPC	
BDT-1-N-15-2BPC	
BDT-1-N-15-4BPC	
BDT-1-N-15-6BPC	
BDT-1-N-10-10BPC	
BDT-1-N-10-12BPC	
BDT-1-N-10-14BPC**	
BDT-1-N-10-4BPC	
BDT-1-N-10-6BPC	

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 27 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.
- UU 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 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
0258355-MB	9/15/10	1,2,3,4,6,7,8-HpCDD OCDD 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	0.25 pg/g 1.2 pg/g 0.17 pg/g 0.15 pg/g 0.38 pg/g 0.20 pg/g 0.70 pg/g 0.26 pg/g 1.2 pg/g	BDT-1-N-10-8BPC BDT-1-N-10-2BPC BDT-1-N-10-8BPC_FD
0258127-MB	9/15/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.19 pg/g 0.93 pg/g 0.15 pg/g 0.078 pg/g 0.27 pg/g 0.32 pg/g	BDT-1-N-20-10BPC BDT-1-N-20-12BPC BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC** BDT-1-N-20-8BPC BDT-1-N-20-10BPC_FD BDT-1-N-15-10BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC BDT-1-N-15-8BPC BDT-1-N-15-2BPC BDT-1-N-15-4BPC BDT-1-N-15-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC** BDT-1-N-10-4BPC BDT-1-N-10-6BPC

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-N-20-10BPC_FD	OCDD	2.4 pg/g	2.4U pg/g

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-1-N-20-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	31 (40-135) 19 (40-135) 34 (40-135) 26 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-20-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 14 (40-135) 34 (40-135) 21 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-20-14BPC	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 35 (40-135) 28 (40-135) 17 (40-135) 11 (40-135) 31 (40-135) 23 (40-135) 14 (40-135)	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 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 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) UJ (all non-detects)	P
BDT-1-N-20-2BPC	<sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 31 (40-135) 17 (40-135) 32 (40-135) 25 (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 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) UJ (all non-detects)	P



Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-N-20-4BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 20 (40-135) 35 (40-135) 29 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-20-6BPC**	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	17 (40-135) 36 (40-135) 32 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-20-8BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 19 (40-135) 37 (40-135) 31 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-20-10BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 15 (40-135) 32 (40-135) 21 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-15-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 21 (40-135) 36 (40-135) 29 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-15-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 22 (40-135) 39 (40-135) 27 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-N-15-14BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 14 (40-135) 35 (40-135) 18 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-15-8BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-N-15-2BPC	<sup>13</sup> C-OCDD	29 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-N-15-4BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 20 (40-135) 35 (40-135) 25 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-15-6BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 20 (40-135) 32 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-10BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 39 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 28 (40-135) 36 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-14BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 18 (40-135) 27 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-4BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 29 (40-135) 38 (40-135)	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	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-N-10-6BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 17 (40-135) 19 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-8BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 39 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-2BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 17 (40-135) 26 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-10-8BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 18 (40-135) 31 (40-135)	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	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-N-20-14BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-6BPC	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-1-N-20-2BPC BDT-1-N-15-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC_FD	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
BDT-1-N-15-10BPC	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

Sample	Compound	Finding	Criteria	Flag	A or P
BDT-1-N-15-12BPC BDT-1-N-15-14BPC	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-1-N-15-2BPC BDT-1-N-10-4BPC BDT-1-N-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
BDT-1-N-15-4BPC BDT-1-N-10-14BPC**	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)	P
BDT-1-N-15-6BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-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) 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 G0H310579	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 G0H310579	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-N-20-10BPC and BDT-1-N-20-10BPC\_FD and samples BDT-1-N-10-8BPC and BDT-1-N-10-8BPC\_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-N-20-10BPC	BDT-1-N-20-10BPC_FD				
1,2,3,7,8-PeCDD	0.35	0.41	-	0.06 (≤0.55)	-	-
1,2,3,4,7,8-HxCDD	0.35	0.19	-	0.16 (≤2.8)	-	-
1,2,3,6,7,8-HxCDD	0.40	0.44	-	0.04 (≤2.8)	-	-
1,2,3,7,8,9-HxCDD	0.40	0.41	-	0.01 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDD	1.9	1.4	-	0.5 (≤2.8)	-	-
OCDD	8.8	2.4	-	6.4 (≤5.5)	J (all detects)	A
2,3,7,8-TCDF	2.5	2.4	-	0.1 (≤0.55)	-	-
1,2,3,7,8-PeCDF	3.9	3.8	-	0.1 (≤2.8)	-	-
2,3,4,7,8-PeCDF	2.0	2.0	-	0 (≤2.8)	-	-
1,2,3,4,7,8-HxCDF	7.8	7.7	-	0.1 (≤2.8)	-	-
1,2,3,6,7,8-HxCDF	5.3	4.8	-	0.5 (≤2.8)	-	-
2,3,4,6,7,8-HxCDF	0.94	1.1	-	0.16 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	1.1	1.0	-	0.1 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	20	20	0 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	12	12	-	0 (≤2.8)	-	-
OCDF	50	45	11 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-1-N-10-8BPC	BDT-1-N-10-8BPC_FD				
2,3,7,8-TCDD	3.1	2.6		0.5 (≤0.52)	-	-
1,2,3,7,8-PeCDD	10	9.3	-	0.7 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	6.0	7.1	-	1.1 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	13	14	7 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	8.6	11	-	2.4 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	49	58	17 (≤50)	-	-	-
OCDD	53	59	11 (≤50)	-	-	-
2,3,7,8-TCDF	60	62	3 (≤50)	-	-	-
1,2,3,7,8-PeCDF	130	160	21 (≤50)	-	-	-
2,3,4,7,8-PeCDF	68	84	21 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	300	310	3 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	210	210	0 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	35	41	16 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	36	39	8 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	780	960	21 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	350	400	13 (≤50)	-	-	-
OCDF	2100	2600	21 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H310579**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H310579	BDT-1-N-20-10BPC BDT-1-N-20-12BPC BDT-1-N-20-4BPC BDT-1-N-20-8BPC BDT-1-N-20-10BPC_FD BDT-1-N-15-10BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC BDT-1-N-15-4BPC	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 OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (I)
G0H310579	BDT-1-N-20-14BPC	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 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 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) UJ (all non-detects)	P	Internal standards (%R) (I)
G0H310579	BDT-1-N-20-2BPC	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 OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (I)
G0H310579	BDT-1-N-20-6BPC**	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 OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (I)
G0H310579	BDT-1-N-15-8BPC BDT-1-N-15-2BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (I)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H310579	BDT-1-N-15-6BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC** BDT-1-N-10-4BPC BDT-1-N-10-6BPC BDT-1-N-10-2BPC BDT-1-N-10-8BPC_FD	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310579	BDT-1-N-10-10BPC BDT-1-N-10-8BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310579	BDT-1-N-20-14BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-6BPC	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)
G0H310579	BDT-1-N-20-2BPC BDT-1-N-15-8BPC BDT-1-N-10-8BPC BDT-1-N-10-8BPC_FD	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H310579	BDT-1-N-15-10BPC	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)	P	Project Quantitation Limit (exceeded range) (e)
G0H310579	BDT-1-N-15-12BPC BDT-1-N-15-14BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H310579	BDT-1-N-15-2BPC BDT-1-N-10-4BPC BDT-1-N-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)
G0H310579	BDT-1-N-15-4BPC BDT-1-N-10-14BPC**	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)	P	Project Quantitation Limit (exceeded range) (e)
G0H310579	BDT-1-N-15-6BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-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) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)



SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H310579	BDT-1-N-20-10BPC BDT-1-N-20-12BPC BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC** BDT-1-N-20-8BPC BDT-1-N-20-10BPC_FD BDT-1-N-15-10BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC BDT-1-N-15-8BPC BDT-1-N-15-2BPC BDT-1-N-15-4BPC BDT-1-N-15-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC** BDT-1-N-10-4BPC BDT-1-N-10-6BPC BDT-1-N-10-8BPC BDT-1-N-10-2BPC BDT-1-N-10-8BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H310579	BDT-1-N-20-10BPC BDT-1-N-20-12BPC BDT-1-N-20-14BPC BDT-1-N-20-2BPC BDT-1-N-20-4BPC BDT-1-N-20-6BPC** BDT-1-N-20-8BPC BDT-1-N-20-10BPC_FD BDT-1-N-15-10BPC BDT-1-N-15-12BPC BDT-1-N-15-14BPC BDT-1-N-15-8BPC BDT-1-N-15-2BPC BDT-1-N-15-4BPC BDT-1-N-15-6BPC BDT-1-N-10-10BPC BDT-1-N-10-12BPC BDT-1-N-10-14BPC** BDT-1-N-10-4BPC BDT-1-N-10-6BPC BDT-1-N-10-8BPC BDT-1-N-10-2BPC BDT-1-N-10-8BPC_FD	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H310579	BDT-1-N-20-10BPC BDT-1-N-20-10BPC_FD	OCDD	J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0H310579**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H310579	BDT-1-N-20-10BPC_FD	OCDD	2.4U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H310579.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340D21  
 SDG #: G0H310579  
 Laboratory: Test America

Stage 2B/4

Date: 11/20/10  
 Page: 1 of 1  
 Reviewer: P7  
 2nd Reviewer: g

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: 8/27/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/TCV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LC5
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 = 1, 8      21, 23
XV.	Field blanks	N	

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

1	BDT-1-N-20-10BPC <i>p</i>	11	BDT-1-N-15-14BPC	21	BDT-1-N-10-8BPC <i>p</i>	31	0258127-MB
2	BDT-1-N-20-12BPC	12	BDT-1-N-15-8BPC	22	BDT-1-N-10-2BPC	32	0258355-MB
3	BDT-1-N-20-14BPC	13	BDT-1-N-15-2BPC	23	BDT-1-N-10-8BPC_FD <i>p</i>	33	
4	BDT-1-N-20-2BPC	14	BDT-1-N-15-4BPC	24	BDT-1-N-15-4BPCMS	34	
5	BDT-1-N-20-4BPC	15	BDT-1-N-15-6BPC	25	BDT-1-N-15-4BPCMSD	35	
6	BDT-1-N-20-6BPC**	16	BDT-1-N-10-10BPC	26	BDT-1-N-10-8BPCMS	36	
7	BDT-1-N-20-8BPC	17	BDT-1-N-10-12BPC	27	BDT-1-N-10-8BPCMSD	37	
8	BDT-1-N-20-10BPC_FD <i>p</i>	18	BDT-1-N-10-14BPC**	28		38	
9	BDT-1-N-15-10BPC	19	BDT-1-N-10-4BPC	29		39	
10	BDT-1-N-15-12BPC	20	BDT-1-N-10-6BPC	30		40	

Notes: *g*

LDC #: 24340D21  
 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
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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"/>	
<b>III. Initial calibration</b>				
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 $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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"/>	
<b>VII. Laboratory control samples</b>				
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 #: 24340D21  
 SDG #: pu cones

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: J

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 ≥ 2.5, at ± 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 PeCDD
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:



**Blanks**

**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 all samples associated with a method blank?

Y  N  N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y  N  N/A Was the method blank contaminated?

Blank extraction date: 9/15/10 Blank analysis date: 9/27/10

Conc. units: 10<sup>-9</sup>

Associated samples: 1-920

EMPZ (bl)

Compound	Blank ID	Sample Identification																		
	0258127-MB	5X	8																	
F	0.19*	0.95																		
G	0.93	4.65	2.4/4																	
K	0.15	0.75																		
L	0.070*	0.39																		
Ø	0.27	1.35																		
Q	0.32	1.6																		

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







Internal Standards

Reviewer: FT

2nd Reviewer: R

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 within the 40-135% criteria?

Y N N/A Was the S/N ratio all internal standard peaks  $\geq 10$ ?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		4	F	36	( 40-135 ) J/L/P G, A, I, F, E
			H	31	( ) F
			I	17	( ) G, A
			E	32	( ) K, L, M, N
			G	25	( ) O, P
		5	H	39	( ) F
			I	20	( ) G, A
			E	35	( ) K, L, M, N
			G	29	( ) O, P
		6	I	17	( ) G, A
			E	36	( ) K, L, M, N
			G	32	( ) O, P
		<del>7</del>	H	39	( ) F
			I	19	( ) G, A
			E	37	( ) K, L, M, N
			G	31	( <input checked="" type="checkbox"/> ) O, P
		Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
A	<sup>13</sup> C-2,3,7,8-TCDF			K	<sup>13</sup> C-1,2,3,4-TCDD
B	<sup>13</sup> C-2,3,7,8-TCDD			L	
C	<sup>13</sup> C-1,2,3,7,8-PeCDF			M	<sup>13</sup> C-1,2,3,7,8,9-HxCDD
D	<sup>13</sup> C-1,2,3,7,8-PeCDD			N	
E	<sup>13</sup> C-1,2,3,6,7,8-HxCDF			O	
F	<sup>13</sup> C-1,2,3,6,7,8-HxCDD			P	
G	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			Q	
H	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			R	
I	<sup>13</sup> C-OCDD			I	

**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".  
 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?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		8	H	28 (40-135)	J/M/P qual F
			I	15	G, Q
			E	32	K, L, M, N
			G	21	O, P
		9	H	34	
			I	21	
			E	36	
			G	29	
		10	H	30	
			I	22	
			E	39	
			G	27	
		11	H	21	
			I	14	
			E	35	
			G	18	
<b>Internal Standards</b>					
A.		<sup>13</sup> C-2,3,7,8-TCDF	Check Standard Used	Recovery Standards	Check Standard Used
B.		<sup>13</sup> C-2,3,7,8-TCDD		<sup>13</sup> C-1,2,3,4-TCDD	
C.		<sup>13</sup> C-1,2,3,7,8-PeCDF		<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
D.		<sup>13</sup> C-1,2,3,7,8-PeCDD			
F.		<sup>13</sup> C-1,2,3,6,7,8-HxCDF			
E.		<sup>13</sup> C-1,2,3,6,7,8-HxCDD			
G.		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			
H.		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			
I.		<sup>13</sup> C-OCDF			

**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".

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

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

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		12	I	37 ( 40-135 )	JWS/P OVAL G, Q
		13	I	29 ( )	↓
		14	H	30 ( )	JWS/P OVAL F
		15	I	20 ( )	G, Q
			E	35 ( )	K, L, M, N
			G	25 ( )	O, P
			H	34 ( )	F
		16	I	20 ( )	G, Q
			<del>G</del>	32 ( )	O, P
		17	I	30 ( )	G, Q
			G	39 ( )	O, P
			H	36 ( )	F
		18	I	28 ( )	G, Q
			G	36 ( )	O, P
			H	36 ( )	F

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

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?

Y (N/A) Was the S/N ratio all internal standard peaks  $\geq 10$ ?

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		18	H	( 40-135 )	JWS P EARL F
			I	( )	
			G	( )	G, Q S, P
		19	H	( )	
			I	( )	
			G	( )	
		20	H	( )	
			I	( )	
			G	( )	
		21	H	( )	
			I	( )	JWS P G, Q S, P
			G	( )	
		22	H	( )	
			I	( )	
			G	( )	G, Q S, P

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
A. <sup>13</sup> C-2,3,7,8-TCDF			
B. <sup>13</sup> C-2,3,7,8-TCDD		<sup>13</sup> C-1,2,3,4-TCDD	
C. <sup>13</sup> C-1,2,3,7,8-PeCDF		<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
D. <sup>13</sup> C-1,2,3,7,8-PeCDD			
E. <sup>13</sup> C-1,2,3,6,7,8-HxCDF			
F. <sup>13</sup> C-1,2,3,6,7,8-HxCDD			
G. <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			
H. <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			
I. <sup>13</sup> C-OCDD			

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".

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

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

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		23	H	32 (40-135)	JMS RP QUAL F
			I	18	G, Q
			G	31	Q, P
		24	F	36	no good MS
			WA H	23	
			T	15	
			E	32	
			G	21	
		25	F	35	no good MS
			H	23	
			I	12	
			E	29	
			G	20	

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

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

#	Date	compd - Sample ID	Finding	Associated Samples	Qualifications
		H, e, f, g	old and Rangy	3, 16, 17, 20	J/P det
		e		4, 12, 21, 23	
		e, f, g		9	
		<del>h</del> e, g		10, 11	
		H, I, K, L, e, f, g		13, 19, 22	
		H, K, e, f, g		14, 18	
		F, H, I, J, K, L, e, f, g	↓	15	↓

Comments: See sample calculation verification worksheet for recalculations



LDC#:24340D21

VALIDATION FINDINGS WORKSHEET

Field Duplicates

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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?

(fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	8				
B	0.35	0.41		0.06	≤0.55	
C	0.35	0.19		0.16	≤2.8	
D	0.40	0.44		0.04	≤2.8	
E	0.40	0.41		0.01	≤2.8	
F	1.9	1.4		0.5	≤2.8	
G	8.8	2.4		6.4	≤5.5	J/A det
H	2.5	2.4		0.1	≤0.55	
I	3.9	3.8		0.1	≤2.8	
J	2.0	2.0		0	≤2.8	
K	7.8	7.7		0.1	≤2.8	
L	5.3	4.8		0.5	≤2.8	
M	0.94	1.1		0.16	≤2.8	
N	1.1	1.0		0.1	≤2.8	
O	20	20	0			
P	12	12		0	≤2.8	
Q	50	45	11			

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LDC#: 24340D21

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

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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)
	21	23				
A	3.1	2.6		0.5	≤0.52	
B	10	9.3		0.7	≤2.6	
C	6.0	7.1		1.1	≤2.6	
D	13	14	7			
E	8.6	11		2.4	≤2.6	
F	49	58	17			
G	53	59	11			
H	60	62	3			
I	130	160	21			
J	68	84	21			
K	300	310	3			
L	210	210	0			
M	35	41	16			
N	36	39	8			
O	780	960	21			
P	350	400	13			
Q	2100	2600	21			

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**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 RRF's,  
 $A_{is}$  = Area of associated internal standard  
 $C_{is}$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)		Recalculated Average RRF (initial)		RRF (CS 3 std)		Recalculated RRF (CS 3 std)		%RSD		
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated	Reported	Recalculated			
1	1 CAL PBS	8/30/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.169	1.2609	1.2609	5.52	5.52	5.52	5.52	5.52	5.52	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.252	1.2887	1.2887	4.0	4.0	4.0	4.0	4.0	4.0	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.165	1.2152	1.2152	6.20	6.20	6.20	6.20	6.20	6.20	6.20
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.180	1.2654	1.2654	5.62	5.62	5.62	5.62	5.62	5.62	5.62
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.892	1.9979	1.9979	6.95	6.95	6.95	6.95	6.95	6.95	
2	1 CAL DB225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.020	1.020	3.32	3.32	3.32	3.32	3.32	3.32	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)											
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)											
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)											
			OCDF ( <sup>13</sup> C-OCDF)											
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)											
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)											
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)											
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)											
			OCDF ( <sup>13</sup> C-OCDF)											

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

**VALIDATION FINDINGS WORKSHEET**  
**Initial Calibration Calculation Verification**

LDC #: 24340D21  
 SDG #: see copy

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 Reviewer: FZ  
 2nd Reviewer: R

**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,  
 $C_s$  = 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 (C.S. 3-std)	RRF (C.S. 3-std)	%RSD	%RSD		
1	1 CAL	7/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	0.985	0.9847	0.9847	3.68	3.68	3.68	3.68
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	0.983	0.9687	0.9687	3.24	3.24	3.24	3.24
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.163	1.1014	1.1014	5.17	5.17	5.17	5.17
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.072	1.1128	1.1128	2.61	2.61	2.61	2.61
			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.370	1.3500	1.3500	1.98	1.98	1.98	1.98
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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 #: 24340021

SDG #: see conts

# VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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2nd Reviewer: [Signature]

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 =  $(A_s)(C_{is}) / (A_{is})(C_s)$       RRF = continuing calibration RRF  
 $A_s$  = 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	020e/05 p2	10/2/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.12	5.7	1.12	5.7
	518							
2	305E/01/PS	10/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.05	10.4	1.05	10.4
	531							
3	275E/02/PS	9/28/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	0.92	7.9	0.92	7.9
	516							

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 =  $100 * MSR - MSDR | * 2 / (MSR + MSDR)$  MSR = Matrix spike percent recovery MS DR = Matrix spike duplicate percent recovery

MS/MSD samples: 24 + 25

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/g)		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	20.2	19.3	3.1	24.9	24.9	109	109	114	114	0.020	0.020
1,2,3,7,8-PeCDD	101	96.3	10	124	119	113	113	114	114	3.8	3.8
1,2,3,4,7,8-HxCDD	101	96.3	6.0	111	110	105	105	109	109	1.1	1.1
1,2,3,4,7,8,9-HpCDF	101	96.3	350	136	370	136	136	370	370	3.8	3.8
OCDF	202	193	2100	2520	3500	261	261	780	780	3.2	3.2

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.





Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(e)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(h)</sup>	Ion ID	Elemental Composition	Analyte			
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>6</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF			
	305.8987	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF			
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)			
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF			
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDD			
	321.8936	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDD			
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDD (S)			
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)			
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDDPE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	DCDPE			
	[354.9792]	LOCK	C <sub>8</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	PFK			
	2	339.8597	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF	
		341.8567	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF	
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDF	
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	<sup>13</sup> C <sub>12</sub> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDF	
355.8546		M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD			
357.8516		M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)			
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)			
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE			
409.7974		M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>2</sub> <sup>37</sup> ClO	HxCDDPE				C <sub>10</sub> F <sub>17</sub>	PFK			
[354.9792]		LOCK	C <sub>8</sub> F <sub>13</sub>	PFK								
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF							
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)							
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD								
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)								
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDD (S)								
	[430.9728]	LOCK	C <sub>8</sub> F <sub>17</sub>	OCDDPE								
				PFK								

(e) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24340D21  
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 NA  
Y N NA

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. #6, OCDF:

$$\text{Conc.} = \frac{(14940820)(4000)}{(24367000)(1.37)(10.55)(0.949)}$$

~~180~~  
= 180 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#6 2,3,7,8 TCDF (DB775)			
		= 20789736 (2000)			
		536377216 (1.06)(10.55)(0.949)			
		= 7.3 pg/g			

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** August 27, 2010

**LDC Report Date:** November 23, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H310592

### Sample Identification

BDT-1-N-5-10BPC  
BDT-1-N-5-12BPC  
BDT-1-N-5-14BPC\*\*  
BDT-1-N-5-8BPC\_FD  
BDT-1-N-5-8BPC  
BDT-1-N-5-2BPC  
BDT-1-N-5-4BPC  
BDT-1-N-5-6BPC  
BDT-1-S-20-10BPC  
BDT-1-S-20-12BPC  
BDT-1-S-20-14BPC\*\*  
BDT-1-S-20-2BPC  
BDT-1-S-20-4BPC  
BDT-1-S-20-6BPC  
BDT-1-S-20-8BPC  
BDT-1-S-20-14BPC\_FD  
BDT-1-S-20-4BPCMS  
BDT-1-S-20-4BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 18 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.
- UU 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 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
0259192-MB	9/16/10	1,2,3,4,6,7,8-HpCDD OCDD 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	0.28 pg/g 0.53 pg/g 0.063 pg/g 0.071 pg/g 0.093 pg/g 0.15 pg/g 0.11 pg/g 0.21 pg/g	All samples in SDG G0H310592

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-20-10BPC	1,2,3,4,6,7,8-HpCDD	1.2 pg/g	1.2U pg/g
BDT-1-S-20-2BPC	1,2,3,4,6,7,8-HpCDD 1,2,3,7,8,9-HxCDF	0.96 pg/g 0.39 pg/g	0.96U pg/g 0.39U pg/g
BDT-1-S-20-4BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.59 pg/g 2.6 pg/g	0.59U pg/g 2.6U pg/g

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. 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-N-5-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	31 (40-135) 23 (40-135) 29 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	31 (40-135) 19 (40-135) 28 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-14BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	19 (40-135) 9.2 (40-135) 16 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-8BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 20 (40-135) 27 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-8BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	22 (40-135) 11 (40-135) 19 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-2BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-N-5-4BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 13 (40-135) 27 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-N-5-6BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 19 (40-135) 35 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 19 (40-135) 29 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	19 (40-135) 11 (40-135) 18 (40-135)	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	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-1-S-20-14BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	21 (40-135) 12 (40-135) 17 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-4BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 15 (40-135) 23 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-2BPC	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 29 (40-135) 27 (40-135) 16 (40-135) 9.6 (40-135) 26 (40-135) 29 (40-135) 28 (40-135) 15 (40-135)	All TCL compounds	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-6BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 18 (40-135) 30 (40-135)	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	J (all detects) UJ (all non-detects)	P
BDT-1-S-20-8BPC	<sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 23 (40-135) 13 (40-135) 39 (40-135) 21 (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 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) UJ (all non-detects)	P
BDT-1-S-20-14BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	23 (40-135) 14 (40-135) 21 (40-135)	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	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 for samples on which Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H310592	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 G0H310592	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-N-5-8BPC and BDT-1-N-5-8BPC\_FD and samples BDT-1-S-20-14BPC\*\* and BDT-1-S-20-14BPC\_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-1-N-5-8BPC_FD	BDT-1-N-5-8BPC				
1,2,3,7,8-PeCDD	0.53	0.55	-	0.02 ( $\leq 2.8$ )	-	-
1,2,3,4,7,8-HxCDD	0.38	0.33	-	0.05 ( $\leq 2.8$ )	-	-
1,2,3,6,7,8-HxCDD	0.74	0.85	-	0.11 ( $\leq 2.8$ )	-	-
1,2,3,7,8,9-HxCDD	0.57	0.43	-	0.14 ( $\leq 2.8$ )	-	-
1,2,3,4,6,7,8-HpCDD	2.5	2.9	-	0.4 ( $\leq 2.8$ )	-	-
OCDD	3.0	5.7	-	2.7 ( $\leq 5.6$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-1-N-5-8BPC_FD	BDT-1-N-5-8BPC				
2,3,7,8-TCDF	4.2	4.9	-	0.7 (≤2.8)	-	-
1,2,3,7,8-PeCDF	7.2	8.4	-	1.2 (≤2.8)	-	-
2,3,4,7,8-PeCDF	3.7	4.3	-	0.6 (≤2.8)	-	-
1,2,3,4,7,8-HxCDF	13	13	-	0 (≤2.8)	-	-
1,2,3,6,7,8-HxCDF	8.6	10	-	1.4 (≤2.8)	-	-
2,3,4,6,7,8-HxCDF	1.8	1.7	-	0.1 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	1.7	1.7	-	0 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	37	44	17 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	17	22	26 (≤50)	-	-	-
OCDF	89	120	30 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-1-S-20-14BPC**	BDT-1-S-20-14BPC_FD				
2,3,7,8-TCDD	0.44	0.30	-	0.14 (≤0.54)	-	-
1,2,3,7,8-PeCDD	1.3	0.78	-	0.52 (≤2.7)	-	-
1,2,3,4,7,8-HxCDD	0.97	0.46	-	0.51 (≤2.7)	-	-
1,2,3,6,7,8-HxCDD	2.3	1.1	-	1.2 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	1.3	0.71	-	0.59 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	7.0	4.7	-	2.3 (≤2.7)	-	-
OCDD	13	44	-	31 (≤5.4)	J (all detects)	A
2,3,7,8-TCDF	14	8.0	55 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	20	12	-	8 (≤2.7)	J (all detects)	A
2,3,4,7,8-PeCDF	11	6.2	-	4.8 (≤2.7)	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-1-S-20-14BPC**	BDT-1-S-20-14BPC_FD				
1,2,3,4,7,8-HxCDF	32	19	51 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	21	11	-	10 (≤2.7)	J (all detects)	A
2,3,4,6,7,8-HxCDF	4.7	2.2	-	2.5 (≤2.7)	-	-
1,2,3,7,8,9-HxCDF	3.6	1.9	-	1.7 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDF	79	42	61 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	38	18	71 (≤50)	-	J (all detects)	A
OCDF	160	77	70 (≤50)	-	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H310592**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H310592	BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC** BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-4BPC BDT-1-N-5-6BPC BDT-1-S-20-10BPC BDT-1-S-20-12BPC BDT-1-S-20-14BPC** BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-14BPC_FD	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310592	BDT-1-N-5-2BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310592	BDT-1-S-20-2BPC	All TCL compounds	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310592	BDT-1-S-20-8BPC	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H310592	BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC** BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-2BPC BDT-1-N-5-4BPC BDT-1-N-5-6BPC BDT-1-S-20-10BPC BDT-1-S-20-12BPC BDT-1-S-20-14BPC** BDT-1-S-20-2BPC BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-8BPC BDT-1-S-20-14BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H310592	BDT-1-N-5-10BPC BDT-1-N-5-12BPC BDT-1-N-5-14BPC** BDT-1-N-5-8BPC_FD BDT-1-N-5-8BPC BDT-1-N-5-2BPC BDT-1-N-5-4BPC BDT-1-N-5-6BPC BDT-1-S-20-10BPC BDT-1-S-20-12BPC BDT-1-S-20-14BPC** BDT-1-S-20-2BPC BDT-1-S-20-4BPC BDT-1-S-20-6BPC BDT-1-S-20-8BPC BDT-1-S-20-14BPC_FD	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H310592	BDT-1-S-20-14BPC** BDT-1-S-20-14BPC_FD	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)	A	Field duplicates (RPD) (fd)
G0H310592	BDT-1-S-20-14BPC** BDT-1-S-20-14BPC_FD	OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,6,7,8-HxCDF	J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H310592**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H310592	BDT-1-S-20-10BPC	1,2,3,4,6,7,8-HpCDD	1.2U pg/g	A	bl
G0H310592	BDT-1-S-20-2BPC	1,2,3,4,6,7,8-HpCDD 1,2,3,7,8,9-HxCDF	0.96U pg/g 0.39U pg/g	A	bl
G0H310592	BDT-1-S-20-4BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.59U pg/g 2.6U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H310592**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24340E21  
 SDG #: G0H310592  
 Laboratory: Test America

VALIDATION COMPLETENESS WORKSHEET  
 Stage 2B/4

Date: 11/20/10  
 Page: 1 of 1  
 Reviewer: FJ  
 2nd Reviewer: FJ

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: 8/27/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/CA	A	
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, 5 11, 16
XV.	Field blanks	N	

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

1	BDT-1-N-5-10BPC	11	BDT-1-S-20-14BPC** D	21	0259192	31	
2	BDT-1-N-5-12BPC	12	BDT-1-S-20-2BPC	22		32	
3	BDT-1-N-5-14BPC**	13	BDT-1-S-20-4BPC	23		33	
4	BDT-1-N-5-8BPC FD D	14	BDT-1-S-20-6BPC	24		34	
5	BDT-1-N-5-8BPC D	15	BDT-1-S-20-8BPC	25		35	
6	BDT-1-N-5-2BPC	16	BDT-1-S-20-14BPC FD D	26		36	
7	BDT-1-N-5-4BPC	17	BDT-1-S-20-4BPCMS	27		37	
8	BDT-1-N-5-6BPC	18	BDT-1-S-20-4BPCMSD	28		38	
9	BDT-1-S-20-10BPC	19		29		39	
10	BDT-1-S-20-12BPC	20		30		40	

Notes: 16



LDC #: 24340E21  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
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 < 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?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	/			
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?	/			
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
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?	/			
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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?	/			
<b>VII. Laboratory control samples</b>				
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 #: 24340E2  
 SDG #: pu cans

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: J

<b>VIII: Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?			/
Were the performance evaluation (PE) samples within the acceptance limits?			/
<b>IX: Internal standards</b>			
Were internal standard recoveries within the 40-135% criteria?			/
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	/		
<b>X: Target compound identification</b>			
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 $\pm 2$ 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?	/		
<b>XI: Compound quantitation/CRQLs</b>			
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?	/		
<b>XII: System performance</b>			
System performance was found to be acceptable.	/		
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	/		
<b>XIV: Field duplicates</b>			
Field duplicate pairs were identified in this SDG.	/		
Target compounds were detected in the field duplicates.	/		
<b>XV: Field blanks</b>			
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 PeCDD
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:

**Blanks**

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 all samples associated with a method blank?

N N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

N N/A

Was the method blank contaminated?

Blank extraction date: 9/16/10 Blank analysis date: 9/27/10

Conc. units: pg/g

Associated samples: AV

A E MYC  
(bl)

Compound		Blank ID	Sample Identification			
		0259192-MB	5X	9	12	13
F	0.28	1.4	1.2/U	0.96/U	0.59*/U	
G	0.53	2.65			2.6/U	
L	0.063*	0.315				
M	0.071*	0.355				
N	0.093*	0.465		0.39*/U		
B	0.15	0.75				
P	0.11*	6.55				
Q	0.21	1.05				

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



**VALIDATION FINDINGS WORKSHEET**  
**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".

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?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)		Qualifications
				(	)	
		3	H	19	( 40-135 )	J/W/S/P Qual F
			I	7.2	( )	G, R
			G	16	( )	Q, P
		4	H	32	( )	
			I	20	( )	
			G	27	( )	
		5	H	22	( )	
			I	11	( )	
			G	19	( )	
		6	I	31	( )	
		7	H	30	( )	
			I	13	( )	
			G	27	( )	
					( )	
					( )	

  

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF	K	<sup>13</sup> C-1,2,3,4-TCDD	
<sup>13</sup> C-2,3,7,8-TCDD	L	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
<sup>13</sup> C-1,2,3,7,8-PeCDE	M		
<sup>13</sup> C-1,2,3,7,8-PeCDD	N		
<sup>13</sup> C-1,2,3,6,7,8-HxCDFE	O		
<sup>13</sup> C-1,2,3,6,7,8-HxCDD	P		
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDE	Q		
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	R		
<sup>13</sup> C-OCDD	T		



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  $\geq$  10?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		12	B	27 (40-135)	JWP All TEL
			D	29 ( )	
			F	27 ( )	
			H	14 ( )	
			I	9.6 ( )	
			A	26 ( )	
			C	29 ( )	
			E	28 ( )	
			G	15 ( )	↓
		14	H	33 ( )	JWP QUAL F
			I	18 ( )	G, Q
			G	30 ( )	O, P
		15	F	39 ( )	QUAL C, D, F
			H	23 ( )	F
			I	13 ( )	G, Q
			A	39 ( )	O, P, H
			G	21 ( )	O, P
				( )	↓
				( )	

  

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



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".

Y/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	H	23 (40-135)	↓ MS   P QUAL F
			I	14	
			G	21	g, R 8   P
		Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
A		<sup>13</sup> C-2,3,7,8-TCDF			K
B		<sup>13</sup> C-2,3,7,8-TCDD			L
C		<sup>13</sup> C-1,2,3,7,8-PeCDF			M
D		<sup>13</sup> C-1,2,3,7,8-PeCDD			N
E		<sup>13</sup> C-1,2,3,6,7,8-HxCDF			O
F		<sup>13</sup> C-1,2,3,6,7,8-HxCDD			P
G		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			Q
H		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			R
I		<sup>13</sup> 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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	4	5				
B	0.53	0.55		0.02	≤2.8	
C	0.38	0.33		0.05	≤2.8	
D	0.74	0.85		0.11	≤2.8	
E	0.57	0.43		0.14	≤2.8	
F	2.5	2.9		0.4	≤2.8	
G	3.0	5.7		2.7	≤5.6	
H	4.2	4.9		0.7	≤2.8	
I	7.2	8.4		1.2	≤2.8	
J	3.7	4.3		0.6	≤2.8	
K	13	13		0	≤2.8	
L	8.6	10		1.4	≤2.8	
M	1.8	1.7		0.1	≤2.8	
N	1.7	1.7		0	≤2.8	
O	37	44	17			
P	17	22	26			
Q	89	120	30			

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

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?

(fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	11	16				
A	0.44	0.30		0.14	≤0.54	
B	1.3	0.78		0.52	≤2.7	
C	0.97	0.46		0.51	≤2.7	
D	2.3	1.1		1.2	≤2.7	
E	1.3	0.71		0.59	≤2.7	
F	7.0	4.7		2.3	≤2.7	
G	13	44		31	≤5.4	J/A det
H	14	8.0	55			J/A det
I	20	12		8	≤2.7	J/A det
J	11	6.2		4.8	≤2.7	J/A det
K	32	19	51			<i>J/A det</i>
L	21	11		10	≤2.7	J/A det
M	4.7	2.2		2.5	≤2.7	
N	3.6	1.9		1.7	≤2.7	
O	79	42	61			J/A det
P	38	18	71			J/A det
Q	160	77	70			J/A det

**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,  
 $C_s$  = 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 (<S> std)	RRF (<S> std)	%RSD	%RSD			
1	ICAL DB225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.020	1.020	3.32	3.32	3.32	3.32	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)									
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)									
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)									
2	ICAL PB5	9/14/10	OCDF ( <sup>13</sup> C-OCDF)									
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.784	0.784	1.05	1.05	11.8	11.8	11.8	11.8	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8	10.8	10.8	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.7	12.7	12.7	12.7	
3			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3	12.3	12.3	
			OCDF ( <sup>13</sup> C-OCDF)	3.118	3.118	1.52	1.52	15.3	15.3	15.3	15.3	
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)									
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)									
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)									
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)									
			OCDF ( <sup>13</sup> C-OCDF)									
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)									

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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$  Where: ave. RRF = initial calibration average RRF  
 RRF =  $(A_s)(C_{is}) / (A_{is})(C_s)$  RRF = continuing calibration RRF  
 $A_s$  = 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	295E105D2-519 DB 225	9/29/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	0.97	7.7	0.97	7.7
			<del>2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</del>					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
	DB - 225		1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
	295E105D2-541	9/29/10	<del>OCDF (<sup>13</sup>C-OCDF)</del> 2,3,7,8-TCDF	1.056	0.94	10.7	0.94	10.7
2	CVN 52	9/28/10 9:57	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.981	1.13	14.7	1.13	14.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.11	7.3	1.11	7.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.26	10.5	1.26	10.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.27	12.1	1.27	12.1
			OCDF ( <sup>13</sup> C-OCDF)	2.118	2.12	0.1	2.12	0.1
3	CVN 52	9/27/10 10:11	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	<del>0.981</del>	1.05	6.5	1.05	6.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.05	1.6	1.05	1.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		1.26	10.7	1.26	10.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)		1.26	11.3	1.26	11.3
			OCDF ( <sup>13</sup> C-OCDF)	↓	2.21	4.6	2.21	4.6

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 =  $100 * MSR - MSDR / (MSR + MSDR)$  MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 17 + 18

Compound	Spike Added (1818)		Sample Concentration (1818)	Spiked Sample Concentration (1818)		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	210	215	0.18	21.8	23.6	103	103	109	109	8.0	8.0
1,2,3,7,8-PeCDD	105	107	0.26	107	109	101	101	101	101	1.9	1.9
1,2,3,4,7,8-HxCDD	105	107	0.21	107	112	102	102	104	104	4.3	4.3
1,2,3,4,7,8,9-HpCDF	105	107	7.0	146	146	133	133	130	130	0.060	0.060
OCDF	210	215	3.1	242	228	101	101	92	92	5.9	5.9

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.

LDC #: T 240004  
 SDG #: for com

**VALIDATION FINDINGS WORKSHEET I**  
**Laboratory Control Sample Results Verification**

Page: 1 of 1  
 Reviewer: FJ  
 2nd Reviewer: R

**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: 0259107-107

Compound	Spike Added (ppb)		Spiked Sample Concentration (ppb)		LCS		LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.
2,3,7,8-TCDD	20	NA	20.4	NA	102	102		
1,2,3,7,8-PeCDD	100		103		103	103		
1,2,3,4,7,8-HxCDD	100		105		105	105		
1,2,3,4,7,8,9-HpCDF	100		103		103	103		
OCDF	200		184		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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDPE		
	[354.9792]	LOCK	C <sub>8</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>8</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDFE							
[354.9792]		LOCK	C <sub>8</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> O	HxCDF (S)						
	385.8610	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDD (S)							
	[430.9728]	LOCK	C <sub>8</sub> F <sub>17</sub>	OCDFE							
				PFK							

(e) The following nucleic masses were used:

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

S = internal/recovery standard

LDC #: 24340E2/  
 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<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = 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. #3, OCDF:

$$\text{Conc.} = \frac{(10181130)(4000)}{21730100(2.12)(10.09)(0.928)}$$

= 94.41 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		# 3 2, 3, 7, 8 TCDF			
		= 7073/22 (2000)			
		253917472 (1.056) (10.09) (0.928)			
		= 5.6 pg/g			

**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 23, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I010541

**Sample Identification**

BDT-1-S-5-10BPC  
BDT-1-S-5-12BPC  
BDT-1-S-5-14BPC\*\*  
BDT-1-S-5-8BPC  
BDT-1-S-5-2BPC  
BDT-1-S-5-4BPC  
BDT-1-S-5-6BPC  
SSAQ5-03-1BPC  
SSAQ5-03-2BPC\*\*  
SSAQ6-01-1BPC  
SSAQ6-01-2BPC  
BDT-1-S-5-14BPCMS  
BDT-1-S-5-14BPCMSD  
SSAQ5-03-1BPCMS  
SSAQ5-03-1BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 15 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.
- UU 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 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
0260233-MB	9/17/10	2,3,7,8-TCDD 1,2,3,4,7,8-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-PeCDF 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	0.054 pg/g 0.037 pg/g 0.17 pg/g 0.87 pg/g 0.032 pg/g 0.035 pg/g 0.049 pg/g 0.045 pg/g 0.076 pg/g 0.034 pg/g 0.041 pg/g 0.13 pg/g 0.10 pg/g 0.092 pg/g	All samples in SDG G01010541

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-5-10BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.37 pg/g 4.3 pg/g 0.11 pg/g 0.12 pg/g	0.37U pg/g 4.3U pg/g 0.11U pg/g 0.12U pg/g
BDT-1-S-5-12BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.32 pg/g 3.9 pg/g 0.12 pg/g	0.32U pg/g 3.9U pg/g 0.12U pg/g
BDT-1-S-5-14BPC**	OCDD	4.3 pg/g	4.3U pg/g
BDT-1-S-5-8BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.30 pg/g 2.2 pg/g 0.24 pg/g 0.16 pg/g 0.19 pg/g	0.30U pg/g 2.2U pg/g 0.24U pg/g 0.16U pg/g 0.19U pg/g
BDT-1-S-5-2BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.063 pg/g 0.077 pg/g 0.69 pg/g	0.063U pg/g 0.077U pg/g 0.69U pg/g
BDT-1-S-5-4BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.059 pg/g 0.064 pg/g 0.30 pg/g 1.2 pg/g 0.19 pg/g	0.059U pg/g 0.064U pg/g 0.30U pg/g 1.2U pg/g 0.19U pg/g
BDT-1-S-5-6BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.074 pg/g 0.079 pg/g 0.80 pg/g	0.074U pg/g 0.079U pg/g 0.80U pg/g
SSAQ5-03-1BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF	0.67 pg/g 0.12 pg/g 0.20 pg/g 0.15 pg/g	0.67U pg/g 0.12U pg/g 0.20U pg/g 0.15U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAQ5-03-2BPC**	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.079 pg/g 0.099 pg/g 0.53 pg/g	0.079U pg/g 0.099U pg/g 0.53U pg/g
SSAQ6-01-1BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.12 pg/g 0.10 pg/g 0.65 pg/g	0.12U pg/g 0.10U pg/g 0.65U pg/g
SSAQ6-01-2BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.53 pg/g 3.3 pg/g 0.21 pg/g 0.16 pg/g 0.13 pg/g	0.53U pg/g 3.3U pg/g 0.21U pg/g 0.16U pg/g 0.13U pg/g

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) were not within QC limits for some 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-1-S-5-10BPC	<sup>13</sup> C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
BDT-1-S-5-12BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 39 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P



Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAQ5-03-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 17 (40-135) 29 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAQ5-03-2BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 9.9 (40-135) 23 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAQ6-01-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 12 (40-135) 37 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAQ6-01-2BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 17 (40-135) 36 (40-135)	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	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
SSAQ5-03-1BPC SSAQ6-01-2BPC	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 G01010541	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 G0I010541	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

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I010541**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I010541	BDT-1-S-5-10BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I010541	BDT-1-S-5-12BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I010541	SSAQ5-03-1BPC SSAQ5-03-2BPC** SSAQ6-01-1BPC SSAQ6-01-2BPC	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I010541	SSAQ5-03-1BPC SSAQ6-01-2BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (no 2 <sup>nd</sup> column confirmation) (o)
G0I010541	BDT-1-S-5-10BPC BDT-1-S-5-12BPC BDT-1-S-5-14BPC** BDT-1-S-5-8BPC BDT-1-S-5-2BPC BDT-1-S-5-4BPC BDT-1-S-5-6BPC SSAQ5-03-1BPC SSAQ5-03-2BPC** SSAQ6-01-1BPC SSAQ6-01-2BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I010541	BDT-1-S-5-10BPC BDT-1-S-5-12BPC BDT-1-S-5-14BPC** BDT-1-S-5-8BPC BDT-1-S-5-2BPC BDT-1-S-5-4BPC BDT-1-S-5-6BPC SSAQ5-03-1BPC SSAQ5-03-2BPC** SSAQ6-01-1BPC SSAQ6-01-2BPC	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  
G0I010541**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I010541	BDT-1-S-5-10BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.37U pg/g 4.3U pg/g 0.11U pg/g 0.12U pg/g	A	bl
G0I010541	BDT-1-S-5-12BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.32U pg/g 3.9U pg/g 0.12U pg/g	A	bl
G0I010541	BDT-1-S-5-14BPC**	OCDD	4.3U pg/g	A	bl
G0I010541	BDT-1-S-5-8BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.30U pg/g 2.2U pg/g 0.24U pg/g 0.16U pg/g 0.19U pg/g	A	bl
G0I010541	BDT-1-S-5-2BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.063U pg/g 0.077U pg/g 0.69U pg/g	A	bl
G0I010541	BDT-1-S-5-4BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.059U pg/g 0.064U pg/g 0.30U pg/g 1.2U pg/g 0.19U pg/g	A	bl
G0I010541	BDT-1-S-5-6BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.074U pg/g 0.079U pg/g 0.80U pg/g	A	bl
G0I010541	SSAQ5-03-1BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF	0.67U pg/g 0.12U pg/g 0.20U pg/g 0.15U pg/g	A	bl
G0I010541	SSAQ5-03-2BPC**	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.079U pg/g 0.099U pg/g 0.53U pg/g	A	bl
G0I010541	SSAQ6-01-1BPC	2,3,7,8-TCDD 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD	0.12U pg/g 0.10U pg/g 0.65U pg/g	A	bl
G0I010541	SSAQ6-01-2BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.53U pg/g 3.3U pg/g 0.21U pg/g 0.16U pg/g 0.13U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01010541.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 24340F21

SDG #: G01010541

Laboratory: Test America

Date: 11/20/10

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#GV-	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
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	SIA	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	N	
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-1-S-5-10BPC	11	SSAQ6-01-2BPC	21	0260233	31	
2	BDT-1-S-5-12BPC	12	BDT-1-S-5-14BPCMS	22		32	
3	BDT-1-S-5-14BPC**	13	BDT-1-S-5-14BPCMSD	23		33	
4	BDT-1-S-5-8BPC	14	SSAQ5-03-1BPCMS	24		34	
5	BDT-1-S-5-2BPC	15	SSAQ5-03-1BPCMSD	25		35	
6	BDT-1-S-5-4BPC	16		26		36	
7	BDT-1-S-5-6BPC	17		27		37	
8	SSAQ5-03-1BPC	18		28		38	
9	SSAQ5-03-2BPC**	19		29		39	
10	SSAQ6-01-1BPC	20		30		40	

Notes: 16

LDC #: 24340f21  
 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
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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"/>	
<b>III. Initial calibration</b>				
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 $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
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 #: 24340F21  
 SDG #: pu cones

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: J

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 ≥ 2.5, at ± 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

Blanks

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 all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 9/17/10

Associated samples: All

Conc. units: ~~ppb~~

\* EMPC

(bl)

Compound	Blank ID	Sample Identification																			
		1	2	3	4	5	6	7	8												
	02602321	MP SX																			
A	0.054 *	0.27								0.063*/u	0.059*/u	0.074*/u									
C	0.037*	0.185								0.077*/u	0.064*/u	0.079*/u									
F	0.17	0.85		0.37/u						0.69/u	0.30/u	0.80/u									
G	0.87*	4.35		4.3/u		4.3/u					1.2/u										
H	0.032	0.16																			0.12/u
I	0.035 *	0.175																			
J	0.049	0.245							0.24/u												
K	0.045	0.225																			
L	0.076	0.38																			
M	0.034	0.17		0.11/u																	
N	0.041	0.205		0.12*/u					0.16/u												
O	0.13	0.65							0.19/u												
P	0.10	0.5																			
Q	0.092 *	0.46																			

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

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 all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 9/17/10 Blank analysis date: 9/29/10 Associated samples: AU

Conc. units: ppb

\* E M P L

(bl)

Compound	Blank ID	MB	SX	9	10	11	Sample Identification
	0260233						
A	0.054*	0.27		0.070*/u	0.12/u		
C	0.037*	0.185		0.099/u	0.10*/u		
F	0.17	0.85		0.53*/u <del>0.087*/u</del>	0.65/u	0.53*/u	
G	0.87*	4.35		0.53*/u	-	3.3/u	
H	0.032	0.16					
I	0.035*	0.175					
J	0.049	0.245				0.21/u	
K	0.045	0.225					
L	0.074	0.38					
M	0.034	0.17				0.16/u	
N	0.041	0.205				0.13*/u	
O	0.13	0.65					
P	0.10	0.5					
Q	0.092*	0.46					

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

**VALIDATION FINDINGS WORKSHEET**  
**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".  
Y N N/A  
Y N N/A  
Y 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.

Was a MS/MSD analyzed every 20 samples of each matrix?  
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
		12 + 13	H	63 (79-137)	75 (79-137)	( )	3	no qual reqs in
			P	( )	150 (79-139)	( )		
				( )	( )	( )		
		14 + 15	E	( )	78 (80-143)	( )	8	↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

**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 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
		12	H	30 (40-135)	no qual MS
			I	20	
			G	28	
		13	H	26	MSD
			I	18	
			G	23	
		14	H	26	MS
			I	12	
			G	27	
		15	H	33	MSD
			I	19	
			G	33	
		1	I	38	JMSIP equal G, Q
		2	I	32	JMSIP G, Q
			G	39	O, P
Internal Standards			Check Standard Used	Recovery Standards	Check Standard Used
A.	<sup>13</sup> C-2,3,7,8-TCDF			K.	<sup>13</sup> C-1,2,3,4-TCDD
B.	<sup>13</sup> C-2,3,7,8-TCDD			L.	
C.	<sup>13</sup> C-1,2,3,7,8-PeCDF			M.	<sup>13</sup> C-1,2,3,7,8,9-HxCDD
D.	<sup>13</sup> C-1,2,3,7,8-PeCDD			N.	
F.	<sup>13</sup> C-1,2,3,6,7,8-HxCDF			O.	
F.	<sup>13</sup> C-1,2,3,6,7,8-HxCDD			P.	
G.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			Q.	
H.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			R.	
I.	<sup>13</sup> C-OCDD			T.	

**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".

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

N/A Was the S/N ratio all internal standard peaks  $\geq 10$ ?

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		8	H	28 ( 40-135 )	J/WJ/P OVAL F
			I	17 ( )	G, O
			G	29 ( )	O, P
		9	H	30 ( )	P
			I	9.9 ( )	
			G	23 ( )	
		10	H	37 ( )	
			I	12 ( )	
			G	37 ( )	
		11	H	34 ( )	
			I	17 ( )	
			G	36 ( )	✓
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF		K.	<sup>13</sup> C-1,2,3,4-TCDD
<sup>13</sup> C-2,3,7,8-TCDD		L.	<sup>13</sup> C-1,2,3,7,8,9-HxCDD
<sup>13</sup> C-1,2,3,7,8-PeCDF		M.	
<sup>13</sup> C-1,2,3,7,8-PeCDD		N.	
<sup>13</sup> C-1,2,3,6,7,8-HxCDF		O.	
<sup>13</sup> C-1,2,3,6,7,8-HxCDD		P.	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		Q.	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		R.	
<sup>13</sup> 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	Compd Sample ID	Finding	Associated Samples	Qualifications
		#	no 2nd column confirmation was performed	8, 11	none / p (e)

Comments: See sample calculation verification worksheet for recalculations

**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,  $A_{is}$  = Area of associated internal standard  
 $C_x$  = 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)	Average RRF (initial)	RRF ( <S>3 std)	RRF ( <S>3 std)	%RSD	%RSD		
1	ICAL DB223	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.022	1.022	3.32	3.32	3.32	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2	ICAL DB5	9/14/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.984	0.984	1.05	1.05	11.8	11.8	11.8	11.8
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8	10.8	10.8
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.7	12.7	12.7	12.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3	12.3	12.3
			OCDF ( <sup>13</sup> C-OCDF)	2.118	2.118	1.52	1.52	15.3	15.3	15.3	15.3
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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



**VALIDATION FINDINGS WORKSHEET**  
**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$  Where: ave. RRF = initial calibration average RRF  
 RRF = continuing calibration RRF  
 $\text{RRF} = (A_x)(C_b) / (A_b)(C_x)$   $A_x$  = Area of associated internal standard  
 $A_b$  = Area of compound,  $C_x$  = 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 516	9/28/10 19:58	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.984	1.03	1.03	5.1	5.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.08	1.08	4.4	4.4
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.27	1.27	11.5	11.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.25	1.25	9.8	9.8
			OCDF ( <sup>13</sup> C-OCDF)	2.118	2.0	2.0	5.6	5.6
2	cen 52	9/29/10 9:56	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.05	1.05	1.05	6.7	6.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.13	1.13	1.13	9.3	9.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.23	1.23	1.23	7.8	7.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.24	1.24	1.24	9.5	9.5
			OCDF ( <sup>13</sup> C-OCDF)	2.04	2.04	2.04	3.7	3.7
3	cen 52 DB225	10/5/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	0.98	0.98	7.0	7.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

**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 =  $100 * MSR - MSDR / (MSR + MSDR)$  MSR = Matrix spike percent recovery MS DR = Matrix spike duplicate percent recovery

MS/MSD samples: 12, 13

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	210	207	0.53	23.2	21.7	108	108	102	102	6.4	6.4
1,2,3,7,8-PeCDD	105	103	0.92	113	109	107	107	104	104	3.4	3.4
1,2,3,4,7,8-HxCDD	105	103	0.43	112	98.3	98.3	107	95	95	13	13
1,2,3,4,7,8,9-HpCDF	105	103	1.2	143	167	125	125	150	150	16	16
OCDF	210	207	50	271	291	105	105	116	116	7.2	7.2

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.



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte			
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF			
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>3</sub> O	HpCDF			
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	HpCDF (S)			
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF			
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO	HpCDD			
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD			
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)			
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)			
	375.6364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD (S)			
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	OCDDPE			
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	OCDF	
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF	
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	OCDD	
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD	
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)			
357.8516		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)			
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)			
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	[422.9278]	M+4		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)			
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDFE		LOCK		C <sub>9</sub> F <sub>17</sub>	DCDDPE			
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		LOCK			PFK			
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF							
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> O	HxCDF (S)							
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	391.8127	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)								
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)								
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	OCDDPE								
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK								

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24346F21  
 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?

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>s</sub> = 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. # 3 OCDD:

$$\text{Conc.} = \frac{(1298620)(4000)}{(91694500)(1.37)(10.11)(0.948)}$$

= 4.3 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	<u>DB225</u>	<u>2, 3, 7, 8-TCDF</u>			
		<u>= 4144200 (2000)</u>			
		<u>418647000 (1.056) (10.11) (0.948)</u>			
		<u>= 20 pg/g</u>			

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** August 31, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I020531

### Sample Identification

SSAQ5-04-1BPC  
SSAQ5-04-2BPC  
SSAQ5-07-1BPC  
SSAQ5-07-2BPC  
RSAK2-1BPC  
RSAK2-3BPC  
RSAK2-5BPC  
RSAK2-8BPC  
RSAK2-10BPC\*\*  
SSAI3-07-1BPC  
SSAI3-07-3BPC  
SSAI3-07-5BPC  
SSAI3-07-8BPC  
SSAI3-07-8BPC\_FD  
SSAI3-07-10BPC  
SSAK4-02-0.00BPC  
SSAQ5-07-1BPCMS  
SSAQ5-07-1BPCMSD  
RSAK2-5BPCMS  
RSAK2-5BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 20 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.
- UU 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 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
0262056-MB	9/19/10	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.18 pg/g 0.15 pg/g 0.053 pg/g 0.089 pg/g 0.11 pg/g	All samples in SDG G01020531

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
SSAQ5-04-1BPC	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	0.36 pg/g 0.11 pg/g 0.14 pg/g 0.32 pg/g	0.36U pg/g 0.11U pg/g 0.14U pg/g 0.32U pg/g
SSAQ5-04-2BPC	2,3,7,8-TCDF	0.11 pg/g	0.11U pg/g
RSAK2-8BPC	2,3,7,8-TCDF	1.0 pg/g	1.0U pg/g

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
SSAQ5-04-2BPC	<sup>13</sup> C-OCDD	29 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAQ5-07-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 25 (40-135) 38 (40-135)	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	J (all detects) UJ (all non-detects)	P
RSAK2-1BPC	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	22 (40-135) 38 (40-135) 35 (40-135) 23 (40-135) 19 (40-135) 34 (40-135) 34 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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-PeCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
RSAK2-5BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
RSAK2-8BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 25 (40-135) 34 (40-135)	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	J (all detects) UJ (all non-detects)	P
RSAK2-3BPC	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	18 (40-135) 31 (40-135) 29 (40-135) 19 (40-135) 12 (40-135) 26 (40-135) 32 (40-135) 27 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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-PeCDF 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) UJ (all non-detects)	P
RSAK2-10BPC**	<sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 18 (40-135) 13 (40-135) 31 (40-135) 17 (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 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) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAI3-07-1BPC	<sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 25 (40-135) 17 (40-135) 25 (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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAI3-07-5BPC	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-2,3,7,8-TCDF	39 (40-135) 39 (40-135) 33 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 2,3,7,8-TCDF	J (all detects) UJ (all non-detects)	P
SSAI3-07-8BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 21 (40-135) 37 (40-135) 36 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 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) UJ (all non-detects)	P
SSAI3-07-10BPC	<sup>13</sup> C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAK4-02-0.00BPC	<sup>13</sup> C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) 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
RSAK2-5BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAI3-07-1BPC	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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAI3-07-3BPC SSAI3-07-8BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-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) J (all detects)	P
SSAI3-07-5BPC SSAI3-07-8BPC_FD	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) J (all detects)	P
SSAK4-02-0.00BPC	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 G0I020531	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 G0I020531	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 SSAI3-07-8BPC and SSAI3-07-8BPC\_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
	SSAI3-07-8BPC	SSAI3-07-8BPC_FD				
2,3,7,8-TCDD	50	20	86 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDD	180	69	89 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDD	130	49	91 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDD	270	100	92 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	230	97	81 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	910	390	80 (≤50)	-	J (all detects)	A
OCDD	840	380	75 (≤50)	-	J (all detects)	A
2,3,7,8-TCDF	1200	420	96 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	2600	1000	89 (≤50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	1400	550	87 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	5700	2300	85 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	3700	1500	85 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	790	340	80 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	620	250	85 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	13000	5500	81 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	5700	2400	81 (≤50)	-	J (all detects)	A
OCDF	38000	18000	71 (≤50)	-	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I020531**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I020531	SSAQ5-04-2BPC RSAK2-5BPC SSAI3-07-10BPC SSAK4-02-0.00BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	SSAQ5-07-1BPC RSAK2-8BPC	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	RSAK2-1BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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-PeCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	RSAK2-3BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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-PeCDF 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) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	RSAK2-10BPC**	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 OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	SSAI3-07-1BPC	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I020531	SSAI3-07-5BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 2,3,7,8-TCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	SSAI3-07-8BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD 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) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I020531	RSAK2-5BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I020531	SSAI3-07-1BPC	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)
G0I020531	SSAI3-07-3BPC SSAI3-07-8BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-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) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I020531	SSAI3-07-5BPC SSAI3-07-8BPC_FD	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) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I020531	SSAK4-02-0.00BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I020531	SSAQ5-04-1BPC SSAQ5-04-2BPC SSAQ5-07-1BPC SSAQ5-07-2BPC RSAK2-1BPC RSAK2-3BPC RSAK2-5BPC RSAK2-8BPC RSAK2-10BPC** SSAI3-07-1BPC SSAI3-07-3BPC SSAI3-07-5BPC SSAI3-07-8BPC SSAI3-07-8BPC_FD SSAI3-07-10BPC SSAK4-02-0.00BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)



SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I020531	SSAQ5-04-1BPC SSAQ5-04-2BPC SSAQ5-07-1BPC SSAQ5-07-2BPC RSAK2-1BPC RSAK2-3BPC RSAK2-5BPC RSAK2-8BPC RSAK2-10BPC** SSAI3-07-1BPC SSAI3-07-3BPC SSAI3-07-5BPC SSAI3-07-8BPC SSAI3-07-8BPC_FD SSAI3-07-10BPC SSAK4-02-0.00BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0I020531	SSAI3-07-8BPC SSAI3-07-8BPC_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-PeCDF 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)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0I020531**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I020531	SSAQ5-04-1BPC	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	0.36U pg/g 0.11U pg/g 0.14U pg/g 0.32U pg/g	A	bl
G0I020531	SSAQ5-04-2BPC	2,3,7,8-TCDF	0.11U pg/g	A	bl
G0I020531	RSAK2-8BPC	2,3,7,8-TCDF	1.0U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I020531.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340G21

SDG #: G01020531

Laboratory: Test America

Stage 2B/4

Date: 10/20/10

Page: 1 of 7

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: 8/31/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration <del>AGV</del>	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	ics
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 = 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	SSAQ5-04-1BPC	11	SSAI3-07-3BPC	21	0202056	31	
2	SSAQ5-04-2BPC	12	SSAI3-07-5BPC	22		32	
3	SSAQ5-07-1BPC	13	SSAI3-07-8BPC	23		33	
4	SSAQ5-07-2BPC	14	SSAI3-07-8BPC_FD	24		34	
5	RSAK2-1BPC	15	SSAI3-07-10BPC	25		35	
6	RSAK2-3BPC	16	SSAK4-02-0.00BPC	26		36	
7	RSAK2-5BPC	17	SSAQ5-07-1BPCMS	27		37	
8	RSAK2-8BPC	18	SSAQ5-07-1BPCMSD	28		38	
9	RSAK2-10BPC**	19	RSAK2-5BPCMS	29		39	
10	SSAI3-07-1BPC	20	RSAK2-5BPCMSD	30		40	

Notes: JB

LDC #: 24340621  
 SDG #: mu cones

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
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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 < 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"/>	
<b>III. Initial calibration</b>				
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) ≤ 20% for unlabeled standards and < 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"/>	
<b>IV. Continuing calibration</b>				
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) ≤ 20% for unlabeled standards and ≤ 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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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 checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
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 #: 2434062  
 SDG #: PC CONES

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: A  
 2nd Reviewer: K

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDF 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

Blanks

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 all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 9/19/10 Blank analysis date: 9/30/10 Associated samples: All

Conc. units: pg/g

AEMPC  
(bl)

Compound	Blank ID	Sample Identification				
	0262036	4B	5X	1	2	8
G	0.18	0.9	0.36*/M	-	-	-
H	0.15*	1.35	0.11*/M	0.11*/M	1.0/M	-
K	0.053*	0.265	0.14*/M	0.14*/M	0.14*/M	-
Q	0.089*	0.445	0.32*/M	0.32*/M	0.32*/M	-
R	0.11*	0.55	-	-	-	-

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

**VALIDATION FINDINGS WORKSHEET**  
**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".  
 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.  
 Was a MS/MSD analyzed every 20 samples of each matrix?  
 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
		17 + 18	perennial	( ) + ( )	( ) ( )	( ) ( )	3	no qual var 1 M
		19 + 20		( ) ( )	( ) ( )	( ) ( )	7	↓

VALIDATION FINDINGS WORKSHEET

Reviewer: FT

2nd Reviewer: [Signature]

(i)

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?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		2	I	( 40-135 )	JW/P qual G, Q
		3	H	( )	F
			I	( )	G, Q
			G	( )	O, P
		5	B	( )	A
			P	( )	B
			H	( )	F
			I	( )	G, Q
			A	( )	H
			C	( )	I, J
			G	( )	O, P
		7	I	( )	
		8	H	( )	F
			I	( )	G, Q
			G	( )	O, P

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF	K	<sup>13</sup> C-1,2,3,4-TCDD	
<sup>13</sup> C-2,3,7,8-TCDD	L	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
<sup>13</sup> C-1,2,3,7,8-PeCDE	M		
<sup>13</sup> C-1,2,3,7,8-PeCDD	N		
<sup>13</sup> C-1,2,3,6,7,8-HxCDE	O		
<sup>13</sup> C-1,2,3,6,7,8-HxCDD	P		
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDE	Q		
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	R		
<sup>13</sup> C-AGDD	T		



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".

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  $\geq 10$ ?

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		6	B	18 ( 40-135 )	JMS/P qual A
			D	31 ( )	B
			H	29 ( )	F
			I	19 ( )	G, Q
			A	12 ( )	H
			C	26 ( )	I, J
			E	32 ( )	K, L, M, N
			G	27 ( )	O, P
		9	F	35 ( )	C, P, E
			H	18 ( )	F
			I	13 ( )	G, Q
			E	31 ( )	K, L, M, N
			G	17 ( )	O, P
		10	F	39 ( )	C, P, E
			H	28 ( )	F
			I	17 ( )	G, Q
			G	25 ( )	O, P

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF			
<sup>13</sup> C-2,3,7,8-TCDD		<sup>13</sup> C-1,2,3,4-TCDD	K
<sup>13</sup> C-1,2,3,7,8-PeCDF		<sup>13</sup> C-1,2,3,7,8,9-HxCDD	L
<sup>13</sup> C-1,2,3,7,8-PeCDD			M
<sup>13</sup> C-1,2,3,6,7,8-HxCDF			N
<sup>13</sup> C-1,2,3,6,7,8-HxCDD			O
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			P
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			Q
<sup>13</sup> C-OCDF			R
<sup>13</sup> C-OCDD			T

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 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
		12	B	39 (40-135)	J/MS/P qual A
			D	39	B
			A	33	H
		14	H	33	F
			I	21	G, Q
			A	37	H
			G	36	O, P
		15	I	26	G, Q
		16	I	33	↓
		17	H	38	no qual MS
			I	26	↓
			G	38	no qual MS
		18	H	37	no qual MS
			I	20	↓
			G	35	↓

  

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF		K, <sup>13</sup> C-1,2,3,4-TCDD	
<sup>13</sup> C-2,3,7,8-TCDD		L, <sup>13</sup> C-1,2,3,7,8,9-HxCDD	
<sup>13</sup> C-1,2,3,7,8-PeCDF		M	
<sup>13</sup> C-1,2,3,7,8-PeCDD		N	
<sup>13</sup> C-1,2,3,6,7,8-HxCDF		O	
<sup>13</sup> C-1,2,3,6,7,8-HxCDD		P	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		Q	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		R	
<sup>13</sup> C-OCDD		T	

## 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".  
 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  $\geq$  10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		19	I	26 ( 40-135 )	no qual MS
			A	37 ( )	
		20	H	36 ( )	MSD
			T	25 ( )	
			G	37 ( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF			
<sup>13</sup> C-2,3,7,8-TCDD			
<sup>13</sup> C-1,2,3,7,8-PeCDF			
<sup>13</sup> C-1,2,3,7,8-PeCDD			
<sup>13</sup> C-1,2,3,6,7,8-HxCDF			
<sup>13</sup> C-1,2,3,6,7,8-HxCDD			
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			
<sup>13</sup> C-OCDF			
	K	<sup>13</sup> C-1,2,3,4-TCDD	
	L	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
	M		
	N		
	O		
	P		
	Q		
	R		
	T		

LDC #: 2434062

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

Page: 1 of 1  
 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".

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? (e)  
 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
		Q	x'd cal Range	7	J/P det
		H, K, e, P, Q	↓	10	
		H, I, J, K, L, e, P, Q	↓	11, 13	
		H, I, K, L, e, P, Q	↓	12, 14	
		e, Q	↓	16	↓

Comments: See sample calculation verification worksheet for recalculations

**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	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)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24340G21

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
Reviewer: FT  
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	Qualifications (Parent Only)
	13	14		
A	50	20	86	J/A det
B	180	69	89	J/A det
C	130	49	91	J/A det
D	270	100	92	J/A det
E	230	97	81	J/A det
F	910	390	80	J/A det
G	840	380	75	J/A det
H	1200	420	96	J/A det
I	2600	1000	89	J/A det
J	1400	550	87	J/A det
K	5700	2300	85	J/A det
L	3700	1500	85	J/A det
M	790	340	80	J/A det
N	620	250	85	J/A det
O	13000	5500	81	J/A det
P	5700	2400	81	J/A det
Q	38000	18000	71	J/A det

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**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 (initial)	(%RSD)	RRF (initial)	(%RSD)
1	ICA PB 225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.0526		1.0526	1.02	1.02	3.32	1.02	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2	ICA L	9/29/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.99702		0.99702	1.06040	1.06040	5.52065	1.06040	5.52065
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.07464		1.07464	1.06344	1.06344	3.66087	1.06344	3.66087
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09507		1.09507	1.20053	1.20053	8.30063	1.20053	8.30063
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.05453		1.05453	1.17327	1.17327	7.25898	1.17327	7.25898
			OCDF ( <sup>13</sup> C-OCDF)	1.486110		1.486110	1.60304	1.60304	9.45734	1.60304	9.45734
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

**VALIDATION FINDINGS WORKSHEET**  
**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$  Where: ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_s) / (A_s)(C_x)$  RRF = continuing calibration RRF  
 $A_x$  = Area of compound,  $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_s$  = 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 PB225	10/4/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.0526	1.0526	1.15	8.5	8.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
2	010e1010PS_1	10/11/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.99702	1.08221	1.08221	8.5	8.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03464	1.15815	1.15815	11.9	11.9
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09301	1.23983	1.23983	13.2	13.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.05453	1.24107	1.24107	17.7	17.7
			OCDF ( <sup>13</sup> C-OCDF)	1.48611	1.66632	1.66632	12.1	12.1
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.



**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 =  $100 * MSR - MSDR / (MSR + MSDR)$  MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 16 of 17

Compound	Spike Added ( <u>100 ug</u> )		Sample Concentration ( <u>100 ug</u> )	Spiked Sample Concentration ( <u>100 ug</u> )		Matrix Spike		Matrix Spike Duplicate		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	21.5	21.5	0.24	21.0	21.3	96	96	98	98	1.5	1.5
1,2,3,7,8-PeCDD	108	108	0.42	107	111	99	99	103	103	4.4	4.4
1,2,3,4,7,8-HxCDD	108	108	0.26	109	112	101	101	104	104	2.8	2.8
1,2,3,4,7,8,9-HpCDF	108	108	2.3	155	169	123	123	135	135	8.2	8.2
OCDF	215	215	220	492	540	126	126	148	148	9.3	9.3

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 I**  
**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: 0262056 W

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	NA	18.2	NA	91	91	91	91						
1,2,3,7,8-PeCDD	100		91.1		91	91								
1,2,3,4,7,8-HxCDD	100		54.9		85	85								
1,2,3,4,7,8,9-HpCDF	100		94.1		94	94								
OCDF	200		155		93	93								

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

(a) The following nuclidic masses were used:

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

S = Internal/recovery standard

LDC #: 2434067  
 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 Were all reported results recalculated and verified for all level IV samples?  
Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>s</sub> = 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. #9 OCDF:

$$\text{Conc.} = \frac{(9871.47)(4000)}{(126184.68)(1.486)(10.06)(0.936)}$$

= 22.36 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#9 2,3,7,8-TCDF			
		= 3182660 (2000)			
		427040000 (1.056)(10.06)(0.936)			
			1.5 pg/g		

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

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

**Collection Date:** September 1, 2010

**LDC Report Date:** November 23, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I030578

**Sample Identification**

SSAJ2-04-10BPC  
SSAJ2-04-1BPC  
SSAJ2-04-5BPC  
SSAI2-03-10BPC\*\*  
SSAI2-03-1BPC  
SSAI2-03-5BPC  
SSAI2-04-10BPC  
SSAI2-04-1BPC  
SSAI2-04-5BPC  
SSAI2-03-1BPC\_FD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 10 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.
- UU 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 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
0262057-MB	9/19/10	1,2,3,4,6,7,8-HpCDD OCDD 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	0.12 pg/g 0.96 pg/g 0.12 pg/g 0.092 pg/g 0.20 pg/g 0.15 pg/g 0.31 pg/g	All samples in SDG G01030578

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

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

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

## 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
SSAJ2-04-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	140 (40-135)	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
SSAJ2-04-1BPC	<sup>13</sup> C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAI2-03-10BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 28 (40-135) 30 (40-135)	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	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAI2-03-1BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 38 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAI2-03-5BPC	<sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	142 (40-135) 139 (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	J (all detects) UJ (all non-detects)	P
SSAI2-04-10BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	31 (40-135) 38 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAI2-04-1BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAI2-04-5BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 158 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAI2-03-1BPC_FD	<sup>13</sup> C-OCDD	35 (40-135)	OCDD  OCDF	J (all detects) UJ (all non-detects) 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
SSAJ2-04-10BPC	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SSAJ2-04-1BPC	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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAI2-03-1BPC	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
SSAI2-04-10BPC SSAI2-04-1BPC SSAI2-03-1BPC_FD	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
SSAI2-04-5BPC	OCDD 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	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

For samples SSAJ2-04-10BPC and SSAI2-03-5BPC the results were reported from the DB-5 column instead of the DB-225 column confirmation run. Due to extremely high concentrations in the samples, they were diluted to 50X, causing the <sup>13</sup>C-2,3,7,8-TCDF internal standard to be diluted out in the DB-225 column analysis. Therefore, the 2,3,7,8-TCDF could not be quantitated for these samples.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0I030578	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 G0I030578	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 SSAI2-03-1BPC and SSAI2-03-1BPC\_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
	SSAI2-03-1BPC	SSAI2-03-1BPC_FD				
2,3,7,8-TCDD	8.9	6.4	33 (≤50)	-	-	-
1,2,3,7,8-PeCDD	29	21	32 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	23	16	36 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	45	34	28 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	37	27	31 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	160	110	37 (≤50)	-	-	-
OCDD	200	130	42 (≤50)	-	-	-
2,3,7,8-TCDF	220	160	32 (≤50)	-	-	-
1,2,3,7,8-PeCDF	440	310	35 (≤50)	-	-	-
2,3,4,7,8-PeCDF	270	210	25 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	790	480	49 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	580	380	42 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	150	81	60 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	110	62	56 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	2300	1500	42 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1000	660	41 (≤50)	-	-	-
OCDF	5900	4000	38 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I030578**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I030578	SSAJ2-04-10BPC	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	Internal standards (%R) (i)
G0I030578	SSAJ2-04-1BPC SSAI2-04-1BPC SSAI2-03-1BPC_FD	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I030578	SSAI2-03-10BPC**	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I030578	SSAI2-03-1BPC SSAI2-04-10BPC SSAI2-04-5BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I030578	SSAI2-03-5BPC	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I030578	SSAJ2-04-10BPC	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I030578	SSAJ2-04-1BPC	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)
G0I030578	SSAI2-03-1BPC	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)
G0I030578	SSAI2-04-10BPC SSAI2-04-1BPC SSAI2-03-1BPC_FD	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0I030578	SSAI2-04-5BPC	OCDD 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	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)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I030578	SSAJ2-04-10BPC SSAJ2-04-1BPC SSAJ2-04-5BPC SSAI2-03-10BPC** SSAI2-03-1BPC SSAI2-03-5BPC SSAI2-04-10BPC SSAI2-04-1BPC SSAI2-04-5BPC SSAI2-03-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I030578	SSAJ2-04-10BPC SSAJ2-04-1BPC SSAJ2-04-5BPC SSAI2-03-10BPC** SSAI2-03-1BPC SSAI2-03-5BPC SSAI2-04-10BPC SSAI2-04-1BPC SSAI2-04-5BPC SSAI2-03-1BPC_FD	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0I030578	SSAI2-03-1BPC SSAI2-03-1BPC_FD	2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	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  
G0I030578**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I030578**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340H21  
 SDG #: G01030578  
 Laboratory: Test America

Stage 2B/4

Date: 11/20/10  
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 Reviewer: FJ  
 2nd Reviewer: Tg

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: 9/1/10
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	N	client specified
VII.	Laboratory control samples	A	ics
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 = 5 + 10
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

1011

1	SSAJ2-04-10BPC	11	0262057	21		31	
2	SSAJ2-04-1BPC	12		22		32	
3	SSAJ2-04-5BPC	13		23		33	
4	SSAI2-03-10BPC**	14		24		34	
5	SSAI2-03-1BPC	15		25		35	
6	SSAI2-03-5BPC	16		26		36	
7	SSAI2-04-10BPC	17		27		37	
8	SSAI2-04-1BPC	18		28		38	
9	SSAI2-04-5BPC	19		29		39	
10	SSAI2-03-1BPC_FD	20		30		40	

Notes: J6

LDC #: 24340H 2/  
 SDG #: per cover

VALIDATION FINDINGS CHECKLIST

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 Reviewer: FJ  
 2nd Reviewer: [Signature]

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
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 < 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?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and < 30% for labeled standards?	/			
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?	/			
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
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?	/			
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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?			/	
<b>VII. Laboratory control samples</b>				
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 #: 24340H21  
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VALIDATION FINDINGS CHECKLIST

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<b>VIII: Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?			/
Were the performance evaluation (PE) samples within the acceptance limits?			✓
<b>IX: Internal standards</b>			
Were internal standard recoveries within the 40-135% criteria?	-	/	
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	/		
<b>X: Target compound identification</b>			
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 $\geq 2.5$ ?		✓	
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	/		
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDF channel?	/		
Was an acceptable lock mass recorded and monitored?	/		
<b>XI: Compound quantitation/CRQLs</b>			
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?	/		
<b>XII: System performance</b>			
System performance was found to be acceptable.	/		
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	/		
<b>XIV: Field duplicates</b>			
Field duplicate pairs were identified in this SDG.	/		
Target compounds were detected in the field duplicates.	/		
<b>XV: Field blanks</b>			
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**  
**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
		H, Q	xld cal Range	1	1/Part (e)
		H, Q, Q		2	
		H, Q, P, Q		5	
		Q, Q		7, 8, 10	
		G, H, I, K, L, Q, P	✓	9	↓
2, 3, 7, 8-TCDF		# 1, 6 The DB225 levels, the sample were diluted 50X in the DB225 column; therefore	reported from DB5 confirmation run. Due to extremely high	analysis rather than to extremely high	Text'
		PC - 2, 3, 7, 8 TCDF	in internal standard	To be diluted out.	
		in the DB225 column; therefore		the analyte could not be quantitated	

Comments: See sample calculation verification worksheet for recalculations



LDC#: 24340H21

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

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**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?

(fd)

*RPD*

Compound	Concentration (pg/g)		%RSD ≤50	Qualifications (Parent Only)
	5	10		
A	8.9	6.4	33	
B	29	21	32	
C	23	16	36	
D	45	34	28	
E	37	27	31	
F	160	110	37	
G	200	130	42	
H	220	160	32	
I	440	310	35	
J	270	210	25	
K	790	480	49	
L	580	380	42	
M	150	81	60	J/A det
N	110	62	56	J/A det
O	2300	1500	42	
P	1000	660	41	
Q	5900	4000	38	

V:\FIELD DUPLICATES\24340H21.wpd



**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:

$RRF = (A_s)(C_p)/(A_p)(C_s)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_s$  = Area of compound,  
 $A_p$  = Area of associated internal standard  
 $C_s$  = Concentration of compound,  
 $C_p$  = 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)	Average RRF (initial)	Average RRF (initial)	RRF (C.S. 3 sta)	RRF (C.S. 3 sta)	RRF (C.S. 3 sta)	%RSD	%RSD
1	ICAL	8/30/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.169	1.2609	1.2609	5.52	5.52	5.52	5.52
	PBS		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.252	1.2587	1.2587	4.0	4.0	4.0	4.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.165	1.2152	1.2152	6.20	6.20	6.20	6.20
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.180	1.2654	1.2654	5.62	5.62	5.62	5.62
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.892	1.9979	1.9979	6.95	6.95	6.95	6.95
2	ICAL	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.050	1.050	1.020	1.020	3.32	3.32	3.32	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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.

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SDG #: see conts

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration Results Verification**

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2nd Reviewer: [Signature]

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

Where: ave. RRF = initial calibration average RRF  
 RRF = continuing calibration RRF

$A_x$  = Area of compound,  $A_{is}$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_{is}$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	cevl 20:110	10/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.05	10.4	1.05	10.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.14	8.8	1.14	8.8
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.20	2.6	1.20	2.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.21	3.0	1.21	3.0
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.74	7.8	1.74	7.8
2	cevl DB225	10/7/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.00	5.5	1.00	5.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

**VALIDATION FINDINGS WORKSHEET I**  
**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 =  $100 * SSC/SA$  Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $100 * (LCS - LCSD) / 2(LCS + LCSD)$  LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0267057-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		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	17.7	NA	88	88							
1,2,3,7,8-PeCDD	100		91.8		92	92								
1,2,3,4,7,8-HxCDD	100		117		117	117								
1,2,3,4,7,8,9-HpCDF	100		102		102	102								
OCDF	200		194		97	97			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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>1</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> O	HpCDF (S)		
	317.9399	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDD (S)		435.8169	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
369.8919		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HxCDFE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> O	HxCDF (S)						
	385.8610	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> O	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	401.8559	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24340H2/  
 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 Were all reported results recalculated and verified for all level IV samples?  
Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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. #4 TCDF  
 Conc. =  $\frac{(571418)(2000)}{266329000(10.51)(1.25)(0.915)}$   
 = 0.365 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8-TCDF			
		= $\frac{20118946(2000)}{(532796400)(1.056)(10.51)(0.915)}$			
		= <u>7.4 pg/g</u>			

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

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

**Collection Date:** September 2 through September 3, 2010

**LDC Report Date:** December 2, 2010

**Matrix:** Soil/Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G01040477

**Sample Identification**

SSAM5-04-10BPC  
SSAM5-04-1BPC  
SSAM5-04-5BPC  
SSAM5-04-5BPC\_FD  
SSAK6-05-4BPC  
SSAK6-05-4BPC\_FD  
SSAK6-05-6BPC  
SSAK6-05-8BPC  
SSAK6-05-10BPC\*\*  
EB-09022010

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 9 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.
- UU 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 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
0262057-MB	9/19/10	1,2,3,4,6,7,8-HpCDD OCDD 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	0.12 pg/g 0.96 pg/g 0.12 pg/g 0.092 pg/g 0.20 pg/g 0.15 pg/g 0.31 pg/g	All soil samples in SDG G01040477
0251243-MB	9/8/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.7 pg/L 39 pg/L 4.2 pg/L	All water samples in SDG G01040477

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-09022010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	1.8 pg/L 19 pg/L 3.3 pg/L	1.8U pg/L 19U pg/L 3.3U pg/L

Sample EB-09022010 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-09022010	9/2/10	1,2,3,4,6,7,8-HpCDD OCDD 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	1.8 pg/L 19 pg/L 1.3 pg/L 1.6 pg/L 3.3 pg/L 1.6 pg/L 3.9 pg/L	All soil samples in SDG G01040477

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0251243-LCS	2,3,4,6,7,8-HxCDF	138 (80-137)	All water samples in SDG G01040477	J+ (all detects)	P

### 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
SSAM5-04-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 21 (40-135) 23 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAM5-04-5BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 38 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAM5-04-5BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 26 (40-135) 32 (40-135)	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	J (all detects) UJ (all non-detects)	P
SSAK6-05-4BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 37 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAK6-05-4BPC_FD	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 39 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAK6-05-8BPC	<sup>13</sup> C-OCDD	36 (40-135)	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAK6-05-10BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	23 (40-135) 15 (40-135) 20 (40-135)	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	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
SSAM5-04-1BPC SSAK6-05-6BPC	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
SSAK6-05-4BPC SSAK6-05-4BPC_FD	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01040477	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 G01040477	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 SSAM5-04-5BPC and SSAM5-04-5BPC\_FD and samples SSAK6-05-4BPC and SSAK6-05-4BPC\_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
	SSAM5-04-5BPC	SSAM5-04-5BPC_FD				
2,3,7,8-TCDD	0.23	0.49	-	0.26 (≤0.54)	-	-
1,2,3,7,8-PeCDD	0.85	1.9	-	1.05 (≤2.7)	-	-
1,2,3,4,7,8-HxCDD	0.86	1.6	-	0.74 (≤2.7)	-	-
1,2,3,6,7,8-HxCDD	1.5	2.6	-	1.1 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	1.2	2.5	-	1.3 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	4.9	9.6	-	4.7 (≤2.7)	J (all detects)	A
OCDD	6.9	15	-	8.1 (≤5.4)	J (all detects)	A
2,3,7,8-TCDF	6.2	12	64 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	11	22	-	11 (≤2.7)	J (all detects)	A
2,3,4,7,8-PeCDF	5.1	12	-	6.9 (≤2.7)	J (all detects)	A
1,2,3,4,7,8-HxCDF	16	33	69 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	14	28	67 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	3.4	7.1	-	3.7 (≤2.7)	J (all detects)	A
1,2,3,7,8,9-HxCDF	2.6	5.7	-	3.1 (≤2.7)	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	60	120	67 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	27	57	71 (≤50)	-	J (all detects)	A
OCDF	140	290	70 (≤50)	-	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK6-05-4BPC	SSAK6-05-4BPC_FD				
2,3,7,8-TCDD	37	33	11 (≤50)	-	-	-
1,2,3,7,8-PeCDD	120	110	9 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	99	95	4 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	200	190	5 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	130	120	8 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	660	620	6 (≤50)	-	-	-
OCDD	700	680	3 (≤50)	-	-	-
2,3,7,8-TCDF	990	930	6 (≤50)	-	-	-
1,2,3,7,8-PeCDF	1800	1700	6 (≤50)	-	-	-
2,3,4,7,8-PeCDF	970	900	7 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	1500	2200	38 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	2000	1900	5 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	600	440	31 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	430	420	2 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	9700	9400	3 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	4600	4800	4 (≤50)	-	-	-
OCDF	25000	25000	0 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01040477**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01040477	EB-09022010	2,3,4,6,7,8-HxCDF	J+ (all detects)	P	Laboratory control samples (%R) (l)
G01040477	SSAM5-04-10BPC SSAM5-04-5BPC_FD SSAK6-05-10BPC**	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (l)
G01040477	SSAM5-04-5BPC SSAK6-05-4BPC SSAK6-05-4BPC_FD	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (l)
G01040477	SSAK6-05-8BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (l)
G01040477	SSAM5-04-1BPC SSAK6-05-6BPC	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)
G01040477	SSAK6-05-4BPC SSAK6-05-4BPC_FD	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)
G01040477	SSAM5-04-10BPC SSAM5-04-1BPC SSAM5-04-5BPC SSAM5-04-5BPC_FD SSAK6-05-4BPC SSAK6-05-4BPC_FD SSAK6-05-6BPC SSAK6-05-8BPC SSAK6-05-10BPC** EB-09022010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01040477	SSAM5-04-10BPC SSAM5-04-1BPC SSAM5-04-5BPC SSAM5-04-5BPC_FD SSAK6-05-4BPC SSAK6-05-4BPC_FD SSAK6-05-6BPC SSAK6-05-8BPC SSAK6-05-10BPC** EB-09022010	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 (Code)
G01040477	SSAM5-04-5BPC SSAM5-04-5BPC_FD	2,3,7,8-TCDF 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)	A	Field duplicates (RPD) (fd)
G01040477	SSAM5-04-5BPC SSAM5-04-5BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G01040477**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G01040477	EB-09022010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	1.8U pg/L 19U pg/L 3.3U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01040477.**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 24340121

SDG #: G01040477

Laboratory: Test America

Date: 11/20/10

Page: 6/7

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: 9/2 - 9/3/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/IGV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	SW	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 = 3, 4      5, 6
XV.	Field blanks	SW	EB = 10

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 & WATER

1	SSAM5-04-10BPC	S	11	0262057	21	31
2	SSAM5-04-1BPC		12	0251243	22	32
3	SSAM5-04-5BPC		13		23	33
4	SSAM5-04-5BPC_FD		14		24	34
5	SSAK6-05-4BPC		15		25	35
6	SSAK6-05-4BPC_FD		16		26	36
7	SSAK6-05-6BPC		17		27	37
8	SSAK6-05-8BPC		18		28	38
9	SSAK6-05-10BPC**	✓	19		29	39
10	EB-09022010	✓	20		30	40

Notes: 16

LDC #: 21340 I21  
 SDG #: per 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
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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 < 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"/>	
<b>III. Initial calibration</b>				
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) ≤ 20% for unlabeled standards and ≤ 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"/>	
<b>IV. Continuing calibration</b>				
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) ≤ 20% for unlabeled standards and ≤ 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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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 type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
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 type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

LDC #: 24340I21  
 SDG #: mu con

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: A  
 2nd Reviewer: Q

<b>VIII: Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>
<b>IX: Internal standards</b>			
Were internal standard recoveries within the 40-135% criteria?			<input checked="" type="checkbox"/>
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?			<input checked="" type="checkbox"/>
<b>X: Target compound identification</b>			
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 $\geq 2.5$ ?			<input checked="" type="checkbox"/>
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ 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 PCDF channel?			<input checked="" type="checkbox"/>
Was an acceptable lock mass recorded and monitored?			<input checked="" type="checkbox"/>
<b>XI: Compound quantitation/CRQLs</b>			
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"/>
<b>XII: System performance</b>			
System performance was found to be acceptable.			<input checked="" type="checkbox"/>
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.			<input checked="" type="checkbox"/>
<b>XIV: Field duplicates</b>			
Field duplicate pairs were identified in this SDG.			<input checked="" type="checkbox"/>
Target compounds were detected in the field duplicates.			<input checked="" type="checkbox"/>
<b>XV: Field blanks</b>			
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:

LDC #: 243412/ VALIDATION FINDINGS WORKSHEET

Blanks

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 all samples associated with a method blank?

Y  N  N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y  N  N/A

Was the method blank contaminated?

Blank extraction date: 9/19/10 Blank analysis date: 9/30/10

Associated samples: All soils 75X

Conc. units: pg/g

Compound	Blank ID	Sample Identification									
	<u>0262057-4B</u>										
F	<u>0.12*</u>										
G	<u>0.96</u>										
K	<u>0.12*</u>										
L	<u>0.092</u>										
O	<u>0.20*</u>										
P	<u>0.15</u>										
Q	<u>0.31*</u>										

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

**Blanks**

Reviewer: FT

2nd Reviewer: 2

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

Y  N  N/A

Y  N  N/A

Were all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Was the method blank contaminated?

Blank extraction date: 9/8/10

Blank analysis date: 9/21/10

Associated samples: All water

(bl)

Conc. units: pg/l

Compound	Blank ID	Sample Identification																				
	0251243	B/K	10																			
F	3.7		1.8/4																			
G	3.9		1.9/4																			
E	4.2		3.3/4																			

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

LDC #: 24340121

### VALIDATION FINDINGS WORKSHEET Field Blanks

Page: 1 of 2

Reviewer: FT

2nd Reviewer: R

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

Y N/A Were field blanks identified in this SDG?

Blank units: pg/L Associated sample units: pg/kg

Sampling date: 01/27/10

Field blank type: (circle one) Field Blank / Rinsate / Other: EB

Associated Samples: All Soils 75X

Compound		Blank ID	Sample Identification							
F		<u>1.0</u>								
G		<u>1.8</u>								
K		<u>1.9</u>								
L		<u>1.3</u>								
Ø		<u>1.6</u>								
P		<u>3.3</u>								
Ø		<u>1.6</u>								
		<u>3.9*</u>								
CRQL										

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:  
Samples with compound concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "U".

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Samples (LCS)**

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)  
 Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N N/A Was a LCS required?  
 N N/A Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?  
 N N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

#	Date	Lab ID/Reference	Compound	LCS %R (Limits)	LCSB %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		0251243-103	M	( )	38 (8-137)	( )	all water	IT Part (1) no MS/ID no LCS/D
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		



**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?

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

(L)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
			H	26 ( 40-135 )	JMS JP QUAL F
			I	21 ( )	G, Q
			G	23 ( )	E, P
			I	33 ( )	G, Q
			G	38 ( )	E, P
			H	34 ( )	F
			I	26 ( )	G, Q
			G	32 ( )	E, P
			I	29 ( )	G, Q
			G	37 ( )	E, P
			I	28 ( )	G, Q
			G	39 ( )	E, P
			I	36 ( )	G, Q
Internal Standards			Check Standard Used	Recovery Standards	Check Standard Used
A	<sup>13</sup> C-2,3,7,8-TCDF			K	<sup>13</sup> C-1,2,3,4-TCDD
B	<sup>13</sup> C-2,3,7,8-TCDD			L	<sup>13</sup> C-1,2,3,7,8,9-HxCDD
C	<sup>13</sup> C-1,2,3,7,8-PeCDF			M	
D	<sup>13</sup> C-1,2,3,7,8-PeCDD			N	
E	<sup>13</sup> C-1,2,3,6,7,8-HxCDF			O	
F	<sup>13</sup> C-1,2,3,6,7,8-HxCDD			P	
G	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF			Q	
H	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			R	
I	<sup>13</sup> 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".

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
		H, O, P, Q	x'd cal Range	2, 7	1 part
		H, I, K, L, R, P, Q	↓	5, 4	1 part

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24340121

# VALIDATION FINDINGS WORKSHEET

## Compound Quantitation and Reported CRQLs

Page: 1 of 1  
Reviewer: FT  
2nd Reviewer: S

**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  
X 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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24340I21

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 Were field duplicate pairs identified in this SDG?

Y/N/NA Were target analytes detected in the field duplicate pairs? (fd)

Compound	Concentration (pg/g)		%BBD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	4				
A	0.23	0.49		0.26	≤0.54	
B	0.85	1.9		1.05	≤2.7	
C	0.86	1.6		0.74	≤2.7	
D	1.5	2.6		1.1	≤2.7	
E	1.2	2.5		1.3	≤2.7	
F	4.9	9.6		4.7	≤2.7	J/A det
G	6.9	15		8.1	≤5.4	J/A det
H	6.2	12	64			J/A det
I	11	22		11	≤2.7	J/A det
J	5.1	12		6.9	≤2.7	J/A det
K	16	33	69			J/A det
L	14	28	67			J/A det
M	3.4	7.1		3.7	≤2.7	J/A det
N	2.6	5.7		3.1	≤2.7	J/A det
O	60	120	67			J/A det
P	27	57	71			J/A det
Q	140	290	70			J/A det

V:\FIELD DUPLICATES\24340I21.wpd

LDC#: 24340I21

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

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

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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	Qualifications (Parent Only)
	5	6		
A	37	33	11	
B	120	110	9	
C	99	95	4	
D	200	190	5	
E	130	120	8	
F	660	620	6	
G	700	680	3	
H	990	930	6	
I	1800	1700	6	
J	970	900	7	
K	1500	2200	38	
L	2000	1900	5	
M	600	440	31	
N	430	420	2	
O	9700	9400	3	
P	4600	4800	4	
Q	25000	25000	0	

V:\FIELD DUPLICATES\24340I21.wpd

## 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,

$C_s$  = 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 (<5.3 std)	RRF (<5.3 std)	%RSD	%RSD		
1	1 CAL DB225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.020	1.020	3-32	3-32		
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2	1 CAL DB5	9/14/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.784	0.784	1.05	1.05	11.8	11.8		
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8		
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.7	12.7		
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3		
			OCDF ( <sup>13</sup> C-OCDF)	2.118	2.118	1.52	1.52	15.3	15.3		
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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 #: 24340121  
 SDG #: see conts

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration Results Verification**

Page: 1 of 1  
 Reviewer: F7  
 2nd Reviewer: R

**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 = 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	ceV 7:59	10/8/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.924	0.92	6.4	0.92	6.4
	S 29		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.04	0.6	1.04	0.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.09	4.8	1.09	4.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.22	7.9	1.22	7.9
2	ceV 9:22	10/7/10 PB 225	OCDF ( <sup>13</sup> C-OCDF)	2.118	2.20	3.9	2.20	3.9
	S 2		2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.00	5.5	1.00	5.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
3			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.



VALIDATION FINDINGS WORKSHEET I  
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 =  $100 \cdot \text{SSC/SA}$       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: 0262057-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	17.7	NA	88	88				
1,2,3,7,8-PeCDD	100		91.8		92	92				
1,2,3,4,7,8-HxCDD	100		117		117	117				
1,2,3,4,7,8,9-HpCDF	100		102		102	102				
OCDF	200		194		97	97	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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> O <sub>2</sub>	TODD		423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TODD		425.7737	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> O <sub>2</sub>	TODD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TODD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFPE		479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDFPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>12</sub> H <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFPE		
409.7974		M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>5</sub> <sup>37</sup> ClO	HpCDFPE				C <sub>9</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>16</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>13</sub> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF (S)						
	385.8610	M+2	C <sub>12</sub> H <sub>13</sub> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDF (S)							
	389.8156	M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>13</sub> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>13</sub> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>13</sub> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFPE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24340121  
 SDG #: pu 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 Were all reported results recalculated and verified for all level IV samples?  
Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>i</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>e</sub> = 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. # 9, OCDF:

$$\text{Conc.} = \frac{(7696352)(4000)}{38934100(2.12)(9.65)(0.907)}$$

= 43 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8 - TCDF			
		= 6628120 (2000)			
		( 667035000 ) (1056) (9.65) (0.907)			
		= 2.15 pg/g			

## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** September 2, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I070430

### Sample Identification

SSAN7-04-2BPC  
SSAN7-04-3BPC  
SSAM7-07-1BPC  
SSAM7-07-2BPC\*\*  
SSAM7-07-3BPC  
SSAL8-03-1BPC  
SSAL8-03-3BPC  
SSAK8-08-1BPC  
SSAK8-08-3BPC  
SSAK8-08-3BPC\_FD  
SSAN7-04-1BPC  
SSAM7-07-3BPC\_FD  
SSAM7-06-1BPC  
SSAM7-06-2BPC  
SSAM7-06-3BPC  
SSAN7-05-1BPC  
SSAN7-05-2BPC  
SSAN7-05-3BPC  
SSAN7-05-1BPC\_FD  
SSAM7-06-3BPCMS

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 21 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.

Cooler temperatures were not provided and therefore were not reviewed.

## **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 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
0263135-MB	9/20/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.17 pg/g 0.89 pg/g 0.46 pg/g 0.15 pg/g 0.072 pg/g 0.20 pg/g 0.47 pg/g	All samples in SDG G01070430

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

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
SSAM7-07-3BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 36 (40-135) 39 (40-135)	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 OCDF	J (all detects) UJ (all non-detects)	P
SSAM7-06-3BPC	<sup>13</sup> C-OCDD	35 (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
SSAN7-05-3BPC	<sup>13</sup> C-OCDD	32 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) 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
SSAN7-04-2BPC SSAN7-04-3BPC	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAM7-07-1BPC SSAM7-07-2BPC** SSAM7-06-3BPC SSAN7-05-1BPC_FD	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SSAM7-07-3BPC SSAL8-03-1BPC SSAN7-05-2BPC	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
SSAK8-08-1BPC	2,3,7,8-TCDF 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)	P
SSAK8-08-3BPC	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
SSAN7-04-1BPC	1,2,3,7,8-PeCDF 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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAM7-06-1BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAM7-06-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
SSAN7-05-3BPC	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01070430	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 G01070430	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 SSAK8-08-3BPC and SSAK8-08-3BPC\_FD, samples SSAM7-07-3BPC and SSAM7-07-3BPC\_FD, and samples SSAN7-05-1BPC and SSAN7-05-1BPC\_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
	SSAN7-05-1BPC	SSAN7-05-1BPC_FD				
2,3,7,8-TCDD	94	66	35 (≤50)	-	-	-
1,2,3,7,8-PeCDD	340	150	78 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDD	190	80	81 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDD	490	220	76 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	350	130	92 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	1400	630	76 (≤50)	-	J (all detects)	A
OCDD	1300	1000	26 (≤50)	-	-	-
2,3,7,8-TCDF	4300	4000	7 (≤50)	-	-	-
1,2,3,7,8-PeCDF	5800	2900	67 (≤50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	2600	1300	67 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	7200	3200	77 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	5000	2500	67 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	1300	480	92 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	630	350	57 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	16000	9800	48 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	5400	3400	45 (≤50)	-	-	-
OCDF	29000	26000	11 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAM7-07-3BPC	SSAM7-07-3BPC_FD				
2,3,7,8-TCDD	22	25	13 (≤50)	-	-	-
1,2,3,7,8-PeCDD	75	92	20 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	38	47	21 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAM7-07-3BPC	SSAM7-07-3BPC_FD				
1,2,3,6,7,8-HxCDD	110	130	17 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	80	88	10 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	310	370	18 (≤50)	-	-	-
OCDD	390	470	19 (≤50)	-	-	-
2,3,7,8-TCDF	1700	2000	16 (≤50)	-	-	-
1,2,3,7,8-PeCDF	1100	1300	17 (≤50)	-	-	-
2,3,4,7,8-PeCDF	430	610	35 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	1400	1800	25 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	1100	1300	17 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	240	260	8 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	170	180	6 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	3700	4600	22 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1600	1700	6 (≤50)	-	-	-
OCDF	19000	22000	15 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK8-08-3BPC	SSAK8-08-3BPC_FD				
2,3,7,8-TCDD	13	4.6	95 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDD	52	13	-	39 (≤2.7)	J (all detects)	A
1,2,3,4,7,8-HxCDD	40	7.9	-	32.1 (≤2.7)	J (all detects)	A
1,2,3,6,7,8-HxCDD	78	18	125 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	56	13	-	43 (≤2.7)	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	250	57	126 (≤50)	-	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK8-08-3BPC	SSAK8-08-3BPC_FD				
OCDD	270	62	125 (≤50)	-	J (all detects)	A
2,3,7,8-TCDF	290	77	116 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	540	130	122 (≤50)	-	J (all detects)	A
2,3,4,7,8-PeCDF	300	68	126 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	820	180	128 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	720	180	120 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	150	47	105 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	110	24	128 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	2700	620	125 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	1100	260	124 (≤50)	-	J (all detects)	A
OCDF	6000	1500	120 (≤50)	-	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01070430**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01070430	SSAM7-07-3BPC	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 OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01070430	SSAM7-06-3BPC SSAN7-05-3BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01070430	SSAN7-04-2BPC SSAN7-04-3BPC	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G01070430	SSAM7-07-1BPC SSAM7-07-2BPC** SSAM7-06-3BPC SSAN7-05-1BPC_FD	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G01070430	SSAM7-07-3BPC SSAL8-03-1BPC SSAN7-05-2BPC	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)
G01070430	SSAK8-08-1BPC	2,3,7,8-TCDF 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)	P	Project Quantitation Limit (exceeded range) (e)
G01070430	SSAK8-08-3BPC	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)
G01070430	SSAN7-04-1BPC	1,2,3,7,8-PeCDF 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)
G01070430	SSAM7-06-1BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G01070430	SSAM7-06-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 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 (Code)
G01070430	SSAN7-05-3BPC	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)
G01070430	SSAN7-04-2BPC SSAN7-04-3BPC SSAM7-07-1BPC SSAM7-07-2BPC** SSAM7-07-3BPC SSAL8-03-1BPC SSAL8-03-3BPC SSAK8-08-1BPC SSAK8-08-3BPC SSAK8-08-3BPC_FD SSAN7-04-1BPC SSAM7-07-3BPC_FD SSAM7-06-1BPC SSAM7-06-2BPC SSAM7-06-3BPC SSAN7-05-1BPC SSAN7-05-2BPC SSAN7-05-3BPC SSAN7-05-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01070430	SSAN7-04-2BPC SSAN7-04-3BPC SSAM7-07-1BPC SSAM7-07-2BPC** SSAM7-07-3BPC SSAL8-03-1BPC SSAL8-03-3BPC SSAK8-08-1BPC SSAK8-08-3BPC SSAK8-08-3BPC_FD SSAN7-04-1BPC SSAM7-07-3BPC_FD SSAM7-06-1BPC SSAM7-06-2BPC SSAM7-06-3BPC SSAN7-05-1BPC SSAN7-05-2BPC SSAN7-05-3BPC SSAN7-05-1BPC_FD	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G01070430	SSAN7-05-1BPC SSAN7-05-1BPC_FD	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 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 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	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)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01070430	SSAK8-08-3BPC SSAK8-08-3BPC_FD	2,3,7,8-TCDD 1,2,3,6,7,8-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-PeCDF 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)	A	Field duplicates (RPD) (fd)
G01070430	SSAK8-08-3BPC SSAK8-08-3BPC_FD	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD	J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G01070430**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01070430**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24340J21

SDG #: G01070430

Laboratory: Test America

Stage 2B/4

Date: 11/20/10

Page: 1 of 1

Reviewer: FJ

2nd Reviewer: FJ

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: 9/2/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	A	
IV.	Routine calibration/IEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LC7
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	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 9, 10    5, 12    16, 19
XV.	Field blanks	N	

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

30/1

1	SSAN7-04-2BPC	11	SSAN7-04-1BPC	21	SSAM7-06-3BPCMSD	31	
2	SSAN7-04-3BPC	12	SSAM7-07-3BPC FD D <sub>1</sub>	22		32	
3	SSAM7-07-1BPC	13	SSAM7-06-1BPC	23		33	
4	SSAM7-07-2BPC**	14	SSAM7-06-2BPC	24	02 63135	34	
5	SSAM7-07-3BPC D <sub>1</sub>	15	SSAM7-06-3BPC	25		35	
6	SSAL8-03-1BPC	16	SSAN7-05-1BPC R <sub>2</sub>	26		36	
7	SSAL8-03-3BPC	17	SSAN7-05-2BPC	27		37	
8	SSAK8-08-1BPC	18	SSAN7-05-3BPC	28		38	
9	SSAK8-08-3BPC D	19	SSAN7-05-1BPC FD D <sub>2</sub>	29		39	
10	SSAK8-08-3BPC FD D	20	SSAM7-06-3BPCMS	30		40	

Notes: 16

LDC #: 24340 J2  
 SDG #: per 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
<b>I. Technical holding times</b>				
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 checked="" type="checkbox"/>	
<b>II. GC/MS Instrument performance check</b>				
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 < 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"/>	
<b>III. Initial calibration</b>				
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) ≤ 20% for unlabeled standards and < 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"/>	
<b>IV. Continuing calibration</b>				
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) ≤ 20% for unlabeled standards and ≤ 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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike/duplicates</b>				
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"/>	
<b>VII. Laboratory control samples</b>				
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 #: 2434052  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: g

<b>VIII: Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>		
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>		
<b>IX: Internal standards</b>			
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"/>		
<b>X: Target compound identification</b>			
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 $\pm 2$ 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 PCDF channel?	<input checked="" type="checkbox"/>		
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>		
<b>XI: Compound quantitation/CRQLs</b>			
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"/>		
<b>XII: System performance</b>			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>		
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>		
<b>XIV: Field duplicates</b>			
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>		
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>		
<b>XV: Field blanks</b>			
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:



LDC #: 24340J2

### VALIDATION FINDINGS WORKSHEET

Page: 1 of 1

Reviewer: FT

2nd Reviewer: RE

#### Blanks

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 all samples associated with a method blank?

Y N N/A  
Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A  
Was the method blank contaminated?

Blank extraction date: 7/20/10 Blank analysis date: 10/01/10

Associated samples: All 7 SX

\* EMPC

Compound	Blank ID	Sample Identification									
	0263135-	M13	SX								
F	0.17*	0.85									
G	0.89	4.45									
H	0.46*	2.3									
K	0.15	0.75									
L	0.072*	0.36									
B	0.20*	1.0									
Q	0.47	2.35									

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

**VALIDATION FINDINGS WORKSHEET**  
**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".  
 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.  
 Was a MS/MSD analyzed every 20 samples of each matrix?  
 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
		20+2	several were	100% +/- outside	% RPD limits	( )	15	no qual les in





**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). (1)

#	Date	compd - Sample ID	Finding	Associated Samples	Qualifications
		H	x'd cal Range	1, 2	JIP det
		H, Q		3, 4, 15, 19	
		H, K, Q, P, Q		5, 6, 17	
		H, K, L, Q, P, Q		8	
		H, Q, P, Q		9	
		I, K, Q, P, Q		11	
		Q		13	
		H, I, Q		14	

Comments: See sample calculation verification worksheet for recalculations

# 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/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?  
Y 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
		H, E, S	x'd cal range	18	2/P det (E)

Comments: See sample calculation verification worksheet for recalculations

# 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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

**VALIDATION FINDINGS WORKSHEET**  
Field Duplicates

**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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	Qualifications (Parent Only)
	16	19		
A	94	66	35	
B	340	150	78	J/A det
C	190	80	81	J/A det
D	490	220	76	J/A det
E	350	130	92	J/A det
F	1400	630	76	J/A det
G	1300	1000	26	
H	4300	4000	7	
I	5800	2900	67	J/A det
J	2600	1300	67	J/A det
K	7200	3200	77	J/A det
L	5000	2500	67	J/A det
M	1300	480	92	J/A det
N	630	350	57	J/A det
O	16000	9800	48	
P	5400	3400	45	
Q	29000	26000	11	

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LDC#: 24340J21

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 2 of 3  
Reviewer: FT  
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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	Qualifications (Parent Only)
	5	12		
A	22	25	13	
B	75	92	20	
C	38	47	21	
D	110	130	17	
E	80	88	10	
F	310	370	18	
G	390	470	19	
H	1700	2000	16	
I	1100	1300	17	
J	430	610	35	
K	1400	1800	25	
L	1100	1300	17	
M	240	260	8	
N	170	180	6	
O	3700	4600	22	
P	1600	1700	6	
Q	19000	22000	15	

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**VALIDATION FINDINGS WORKSHEET**  
Field Duplicates

**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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	9	10				
A	13	4.6	95			J/A det
B	52	13		39	≤2.7	J/A det
C	40	7.9		32.1	≤2.7	J/A det
D	78	18	125			J/A det
E	56	13		43	≤2.7	J/A det
F	250	57	126			J/A det
G	270	62	125			J/A det
H	290	77	116			J/A det
I	540	130	122			J/A det
J	300	68	126			J/A det
K	820	180	128			J/A det
L	720	180	120			J/A det
M	150	47	105			J/A det
N	110	24	128			J/A det
O	2700	620	125			J/A det
P	1100	260	124			J/A det
Q	6000	1500	120			J/A det

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**VALIDATION FINDINGS WORKSHEET I**  
**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,  
 $A_{is}$  = Area of associated internal standard  
 $C_x$  = 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)	Average RRF (initial)	RRF (C.S. 3 std)	RRF (C.S. 3 std)	%RSD	%RSD	RRF (C.S. 3 std)	%RSD
1	ICAL PBS	8/30/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.169	1.2609	1.2609	5.52	5.52	5.52	5.52
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.252	1.2887	1.2887	4.0	4.0	4.0	4.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.165	1.2152	1.2152	6.20	6.20	6.20	6.20
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.180	1.2654	1.2654	5.62	5.62	5.62	5.62
			OCDF ( <sup>13</sup> C-OCDF)	1.892	1.892	1.9979	1.9979	6.95	6.95	6.95	6.95
2	ICAL PBS	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.052	1.052	1.020	1.020	3.32	3.32	3.32	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3	ICAL PBS		2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

**VALIDATION FINDINGS WORKSHEET**  
**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$  Where: ave. RRF = initial calibration average RRF  
 RRF =  $(A_x) / (C_x) / (A_{is}) / (C_{is})$  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	10/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	0.98	7.4	0.98	7.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					
2	cen s31	10/19/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	1.10	6.1	1.10	6.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.02	18.7	1.02	18.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.165	1.12	4.3	1.12	4.3
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.180	1.06	9.9	1.06	9.9
			OCDF ( <sup>13</sup> C-OCDD)	1.892	1.57	17.1	1.57	17.1
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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: 20 + 2

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported	Recalculated
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	22.0	22.0	3.0	21.6	22.6	84	84	89	89	4.6	4.6
1,2,3,7,8-PeCDD	110	110	9.3	110	111	92	92	92	92	0.68	0.68
1,2,3,4,7,8-HxCDD	110	110	5.2	102	101	88	88	87	87	1.0	1.0
1,2,3,4,7,8,9-HpCDF	110	110	43.0	51.0	70.2	80	80	250	250	31	31
OCDF	220	220	1600.0	8050	8640	0	0	0	0	0	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.

LDC #: 2434221

SDG #: per copy

**VALIDATION FINDINGS WORKSHEET I**  
**Laboratory Control Sample Results Verification**

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Reviewer: FL

2nd Reviewer: g

**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 =  $100 * SSC/SA$       Where: SSC = Spiked sample concentration  
SA = Spike added

$RPD = |LCS - LCSD| * 2 / (LCS + LCSD)$

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

LCS ID: 0263135-117

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	20.6	NA	17.6	NA	86	86	93	93						
1,2,3,7,8-PeCDD	100		89.2		89	89	100	100						
1,2,3,4,7,8-HxCDD	100		10.0		100	100	93	93						
1,2,3,4,7,8,9-HpCDF	20.0		18.5		93	93	NA	NA						
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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O	TCDF (S)		419.8250	M	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF (S)		
	317.9389	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		423.7767	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD		425.7737	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		435.8169	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O <sub>2</sub>	TCDD (S)		437.8140	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		479.7165	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	HxCDF		[430.9728]	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	NCDPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK			LOCK			PFK	
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		353.8970	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	471.7750	M+2		C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
369.8919		M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	[422.9278]	M+4		C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF		LOCK			PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> O	HxCDF (S)						
		385.8610	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF (S)						
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF (S)							
	391.8127	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	401.8559	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	403.8529	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>13</sub>	OCDF							

(e) The following nucleic masses were used:

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

S = Internal/recovery standard

LDC #: 24340J2  
 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 Were all reported results recalculated and verified for all level IV samples?  
 Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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

- $A_s$  = Area of the characteristic ion (EICP) for the compound to be measured  
 $A_i$  = Area of the characteristic ion (EICP) for the specific internal standard  
 $I_s$  = Amount of internal standard added in nanograms (ng)  
 $V_s$  = 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:

1, 2, 3, 7, 8

Sample I.D. # 4 ~~TEQ~~ PE: CDF

$$\text{Conc.} = \frac{(197305900)(2000)}{(7236390)(1.22)(10.15)(0.90)}$$

$$= 4900 \text{ pg/g}$$

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		<u>2, 3, 7, 8 - TEQ</u>	<u>DB225</u>		
		<u>= 811904000</u>	<u>(2000)</u>		
		<u>26769400</u>	<u>(1036)(10.15)(0.901)</u>		
		<u>6300</u>	<u>pg/g</u>		

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

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

**Collection Date:** September 8, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I100609

**Sample Identification**

SSAO8-07-0BPC

## Introduction

This data review covers one soil 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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.
- UU 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% .

## **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.

## **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
0263138-MB	9/20/10	1,2,3,4,7,8-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 OCDF	0.063 pg/g 0.11 pg/g 0.64 pg/g 0.062 pg/g 0.058 pg/g 0.040 pg/g 0.048 pg/g 0.14 pg/g 0.13 pg/g	All samples in SDG G01100609

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
SSA08-07-0BPC	2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.14 pg/g 0.10 pg/g	0.14U pg/g 0.10U pg/g

Sample EB-09082010-01 (from SDG G01100610) 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-09082010-01	9/8/10	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	4.0 pg/L 1.7 pg/L 2.6 pg/L	All samples in SDG G01100609

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within 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
SSAO8-07-0BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 27 (40-135) 29 (40-135)	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	J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAO8-07-0BPC	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 G01100609	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01100609	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

## XII. System Performance

Raw data were not reviewed for this SDG.

### **XIII. Overall Assessment of Data**

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

### **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I100609**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I100609	SSAO8-07-0BPC	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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I100609	SSAO8-07-0BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (no 2 <sup>nd</sup> column confirmation(o))
G0I100609	SSAO8-07-0BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I100609	SSAO8-07-0BPC	All compounds reported as 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  
G0I100609**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I100609	SSAO8-07-0BPC	2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.14 pg/g 0.10 pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0I100609**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 24340K21  
 SDG #: G01100609  
 Laboratory: Test America

Date: 11/20/10  
 Page: 1 of 1  
 Reviewer: EJ  
 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: 9/8/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration/CEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	LC5
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	EB = EB - 0908 2010 - 01

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

SDG # G01100610

Validated Samples:  
 5011

1	SSAO8-07-0BPC	11	0263138	21		31	
2		12		22		32	
3		13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: JR

# 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 PeCDD
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:

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### VALIDATION FINDINGS WORKSHEET

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

Y/N N/A

Y/N N/A

Were all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Was the method blank contaminated?

Blank extraction date: 9/20/10

Blank analysis date: 10/6/10

Associated samples: A11

Conc. units: psg

EMPC

(bl)

Compound	Blank ID	Sample Identification				
	0263138-	MD	SY			
C	0.063	0.315	.			
F	0.11*	0.55	.			
G	0.64	3.2	.			
K	0.062	0.31	.			
L	0.058*	0.29	.			
M	0.040*	0.20	0.14*/u			
N	0.048*	0.24	0.10*/u			
Ø	0.14*	0.70				
Q	0.13	0.65				

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











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

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

**Collection Date:** September 8, 2010

**LDC Report Date:** November 23, 2010

**Matrix:** Soil/Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I100610

**Sample Identification**

SSAQ3-02-1BPC\*\*  
SSAQ3-02-1BPC\_FD  
EB-09082010\_01

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 2 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.
- UU 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 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
0263138-MB	9/20/10	1,2,3,4,7,8-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 OCDF	0.063 pg/g 0.11 pg/g 0.64 pg/g 0.062 pg/g 0.058 pg/g 0.040 pg/g 0.048 pg/g 0.14 pg/g 0.13 pg/g	All soil samples in SDG G01100610
0257259-MB	9/14/10	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 OCDF	0.56 pg/L 3.2 pg/L 0.36 pg/L 0.32 pg/L 0.29 pg/L 0.55 pg/L	All water samples in SDG G01100610

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
SSAQ3-02-1BPC**	1,2,3,4,7,8-HxCDD	0.17 pg/g	0.17U pg/g
SSAQ3-02-1BPC_FD	1,2,3,4,7,8-HxCDD	0.22 pg/g	0.22U pg/g
EB-09082010_01	OCDD OCDF	4.0 pg/L 2.6 pg/L	4.0U pg/L 2.6U pg/L

Sample EB-09082010\_01 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-09082010_01	9/8/10	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	4.0 pg/L 1.7 pg/L 2.6 pg/L	All soil samples in SDG G01100610

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

## VI. Matrix Spike/Matrix Spike Duplicates

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



## 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
SSAQ3-02-1BPC**	<sup>13</sup> C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAQ3-02-1BPC_FD	<sup>13</sup> C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) 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 for samples on which Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01100610	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 G01100610	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 SSAQ3-02-1BPC and SSAQ3-02-1BPC\_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
	SSAQ3-02-1BPC	SSAQ3-02-1BPC_FD				
2,3,7,8-TCDD	0.13	0.11	-	0.02 ( $\leq 0.55$ )	-	-
1,2,3,7,8-PeCDD	0.33	0.36	-	0.03 ( $\leq 2.8$ )	-	-
1,2,3,4,7,8-HxCDD	0.17	0.22	-	0.05 ( $\leq 2.8$ )	-	-
1,2,3,6,7,8-HxCDD	0.51	0.55	-	0.04 ( $\leq 2.8$ )	-	-
1,2,3,7,8,9-HxCDD	0.47	0.54	-	0.07 ( $\leq 2.8$ )	-	-
1,2,3,4,6,7,8-HpCDD	2.6	2.4	-	0.2 ( $\leq 2.8$ )	-	-
OCDD	15	8.3	-	6.7 ( $\leq 5.5$ )	-	-
2,3,7,8-TCDF	2.8	2.8	0 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDF	5.1	5.1	-	0 ( $\leq 2.8$ )	-	-
2,3,4,7,8-PeCDF	2.2	2.3	-	0.1 ( $\leq 2.8$ )	-	-
1,2,3,4,7,8-HxCDF	10	11	-	1 ( $\leq 2.8$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAQ3-02-1BPC	SSAQ3-02-1BPC_FD				
1,2,3,6,7,8-HxCDF	7.2	7.6	-	0.4 (≤2.8)	-	-
2,3,4,6,7,8-HxCDF	1.3	1.7	-	0.4 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	1.2	1.5	-	0.3 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	34	34	0 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	16	16	0 (≤50)	-	-	-
OCDF	120	120	0 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01100610**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01100610	SSAQ3-02-1BPC** SSAQ3-02-1BPC_FD	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01100610	SSAQ3-02-1BPC** SSAQ3-02-1BPC_FD EB-09082010_01	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01100610	SSAQ3-02-1BPC** SSAQ3-02-1BPC_FD EB-09082010_01	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 G01100610**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G01100610	SSAQ3-02-1BPC**	1,2,3,4,7,8-HxCDD	0.17U pg/g	A	bl
G01100610	SSAQ3-02-1BPC_FD	1,2,3,4,7,8-HxCDD	0.22U pg/g	A	bl
G01100610	EB-09082010_01	OCDD OCDF	4.0U pg/L 2.6U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01100610.**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 24340L21  
 SDG #: G01100610  
 Laboratory: Test America

Date: 11/20/10  
 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	Δ	Sampling dates: 9/8/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration/CEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	ics
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	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	SW	P = 1, 2
XV.	Field blanks	SW	EB = 3

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	SSAQ3-02-1BPC** S	11	0263138-MB	21		31	
2	SSAQ3-02-1BPC_FD ↓	12		22		32	
3	EB-09082010_01 W	13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: 16

LDC #: 24340L21  
 SDG #: mu cones

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: FJ  
 2nd Reviewer: J

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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"/>	
<b>III. Initial calibration</b>				
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 $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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 type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
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 #: 24340221  
 SDG #: mu cones

VALIDATION FINDINGS CHECKLIST

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

<b>VIII: Regional Quality Assurance and Quality Control</b>			
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"/>
<b>IX: Internal standards</b>			
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"/>
<b>X: Target compound identification</b>			
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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE 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"/>
<b>XI: Compound quantitation/CRQLs</b>			
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"/>
<b>XII: System performance</b>			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIII: Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIV: Field duplicates</b>			
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"/>
<b>XV: Field blanks</b>			
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:



LDC #: 21340221

**VALIDATION FINDINGS WORKSHEET**

Page: 1 of 1

Reviewer: FT

2nd Reviewer: R

**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 all samples associated with a method blank?

Y  N  N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y  N  N/A

Was the method blank contaminated?

Blank extraction date: 9/20/10

Blank analysis date: 10/6/10

Associated samples: A11 801/5

Conc. units: ~~pg/g~~

(68)

Compound	Blank ID	Sample Identification				
		1	2	3	4	5
	0263138	MB	SX			
C	0.063	0.315	0.55	0.17*/4	0.22/4	
F	0.11*	3.2				
G	0.64	0.31				
K	0.062	0.29				
L	0.058*	0.20				
M	0.040*	0.24				
N	0.048*	0.70				
Ø	0.14*	0.65				
Ø	0.13					

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





# VALIDATION FINDINGS WORKSHEET

**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".

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?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	I	30 (40-135)	JUS/P out 9, 8
		2	I	28 (40-135)	↓

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

LDC #: 24340 L 21

### VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: 1 of 1  
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".

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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

**VALIDATION FINDINGS WORKSHEET**  
Field Duplicates

**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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	2				
A	0.13	0.11		0.02	≤0.55	
B	0.33	0.36		0.03	≤2.8	
C	0.17	0.22		0.05	≤2.8	
D	0.51	0.55		0.04	≤2.8	
E	0.47	0.54		0.07	≤2.8	
F	2.6	2.4		0.2	≤2.8	
G	15	8.3		6.7	≤5.5	
H	2.8	2.8	0			
I	5.1	5.1		0	≤2.8	
J	2.2	2.3		0.1	≤2.8	
K	10	11		1	≤2.8	
L	7.2	7.6		0.4	≤2.8	
M	1.3	1.7		0.4	≤2.8	
N	1.2	1.5		0.3	≤2.8	
O	34	34	0			
P	16	16	0			
Q	120	120	0			

**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,  
 $A_{is}$  = Area of associated internal standard  
 $C_x$  = 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)	RRF (initial)	Average RRF (initial)	RRF (initial)	RRF (CS 2-std)	%RSD	RRF (CS 3-std)	%RSD
1	1 CAL	7/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	0.9849	0.995	0.9849	0.9849	3.68	0.9849	3.68
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	0.9681	0.983	0.9681	0.9681	3.24	0.9681	3.24
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.1014	1.163	1.1014	1.1014	5.17	1.1014	5.17
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.1128	1.072	1.1128	1.1128	2.61	1.1128	2.61
			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.3500	1.370	1.3500	1.3500	1.98	1.3500	1.98
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

**VALIDATION FINDINGS WORKSHEET**  
**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:

$RRF = (A_s)(C_{is}) / (A_i)(C_s)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_s$  = Area of compound,  
 $C_s$  = 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 (std)	(%RSD)	RRF (std)	(%RSD)
1	ICAL DB225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.0579		1.0579		1.020		1.020	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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



**VALIDATION FINDINGS WORKSHEET**  
**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       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	CEV 15	10/5/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	1.05	1.05	5.3	5.3
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	1.02	1.02	4.1	4.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.15	1.15	1.3	1.3
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.12	1.12	4.9	4.9
			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.26	1.26	7.7	7.7
2	OEN D1322 S19	10/7/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	6.97	0.97	8.5	8.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

# VALIDATION FINDINGS WORKSHEET 1

## 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 =  $100 * SSC/SA$  Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $100 * |LCS - LCS_{D1}| / 2(LCS + LCS_{D1})$  LCS = Laboratory control sample percent recovery LCS<sub>D</sub> = Laboratory control sample duplicate percent recovery

LCS ID: LCS 0263138

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCS <sub>D</sub> Percent Recovery		RPD	
	LCS	LCS <sub>D</sub>	LCS	LCS <sub>D</sub>	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	20.7	NA	104	104				
1,2,3,7,8-PeCDD	100		111		111	111				
1,2,3,4,7,8-HxCDD	100		102		102	102				
1,2,3,4,7,8,9-HpCDF	100		109		109	109				
OCDF	200		176		88	88	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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDF (S)		
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	TCDD		425.7737	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	TCDD (S)		437.8140	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDD (S)		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	NCDPE		
	2	339.8597	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>		PeCDF		443.7399	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDF
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>		PeCDF (S)		459.7348	M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDF
355.8546		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDD		
357.8516		M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	OCDD (S)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDFE				C <sub>10</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O	HxCDF (S)						
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	HxCDF (S)						
	389.8156	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HxCDD							
	391.8127	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> O <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub>	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) The following nucleic masses were used:

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

S = internal/recovery standard

LDC #: 2434022/  
 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 Were all reported results recalculated and verified for all level IV samples?  
Y/N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>s</sub> = 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. # 1 OCDF:

$$\text{Conc.} = \frac{(21553246)(4000)}{(56275104)(1.37)(9.85)(0.92)} = 123 \text{ pg/g}$$

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8 - TCDF			
		= $\frac{8703430(2000)}{643467000(1.056)(9.85)(0.92)}$			
		= 2.8 pg/g			

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

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

**Collection Date:** August 13, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G01130476

**Sample Identification**

SSAL4-04-1BPC

## Introduction

This data review covers one soil 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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.
- UU 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% .

## **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.

## **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
02456381-MB	9/13/10	1,2,3,4,6,7,8-HpCDD OCDD 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	0.18 pg/g 0.84 pg/g 0.067 pg/g 0.056 pg/g 0.13 pg/g 0.12 pg/g 0.19 pg/g	All samples in SDG G01130476

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

No field blanks were identified in this SDG.

#### VI. Matrix Spike/Matrix Spike Duplicates

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

#### VII. Laboratory Control Samples (LCS)

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

#### VIII. Regional Quality Assurance and Quality Control

Not applicable.

#### IX. Internal Standards

All internal standard recoveries were within QC limits.

#### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

#### 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
SSAL4-04-1BPC	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01130476	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01130476	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

## **XII. System Performance**

Raw data were not reviewed for this SDG.

## **XIII. Overall Assessment of Data**

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

## **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
 Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01130476**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01130476	SSAL4-04-1BPC	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)
G01130476	SSAL4-04-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01130476	SSAL4-04-1BPC	All compounds reported as 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 G01130476**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
 Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01130476**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24340M21  
 SDG #: G01130476  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Stage 2B

Date: 11/20/10  
 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: 8/13/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/CEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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:  
 30 IL

1	SSAL4-04-1BPC	11	0256381-MB	21		31	
2		12		22		32	
3		13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: no IS

# 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:

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**Blanks**

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

\* EMPK

Blank extraction date: 9/13/10 Blank analysis date: 9/28/10 Associated samples: All 75X

Conc. units: pg/g

Compound	Blank ID	Sample Identification									
	0256381 MB										
F	0.18										
G	0.84 *										
K	0.067 *										
L	0.056 *										
O	0.13 *										
P	0.12										
Q	0.19 *										

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

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported 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".

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).

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

#	Date	Compound Sample-ID	Finding	Associated Samples	Qualifications
		H, O, P, Q	K'd cal Rangy	1	1 / Pcht (e)

Comments: See sample calculation verification worksheet for recalculations

**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 X/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	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)

Comments: See sample calculation verification worksheet for recalculations



## Laboratory Data Consultants, Inc. Data Validation Report

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

**Collection Date:** September 16 through September 17, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G01180492

### Sample Identification

SSAM6-06-4\_01\_BPC  
SSAM6-06-1\_01\_BPC  
SSAM6-05-4\_01\_BPC  
SSAM6-05-1\_01\_BPC  
SSAM5-05-4\_01\_BPC\*\*  
SSAM5-05-1\_01\_BPC  
SSAM5-05-1\_01\_BPC\_FD  
SSAM6-06-0\_01\_BPC  
SSAM6-05-0\_01\_BPC  
SSAM5-05-0\_01\_BPC  
SSAM6-06-1\_01\_BPCMS  
SSAM6-06-1\_01\_BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 12 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.
- UU 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 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
0265175-MB	9/22/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.73 pg/g 0.058 pg/g 0.14 pg/g 0.069 pg/g 0.26 pg/g	All samples in SDG G01180492

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
SSAM6-06-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.41 pg/g 3.6 pg/g	0.41U pg/g 3.6U pg/g
SSAM6-05-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.27 pg/g 1.7 pg/g	0.27U pg/g 1.7U pg/g
SSAM5-05-4_01_BPC**	1,2,3,4,6,7,8-HpCDD OCDD	0.56 pg/g 3.3 pg/g	0.56U pg/g 3.3U pg/g

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
SSAM6-06-1_01_BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	23 (40-135) 36 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAM6-05-4_01_BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 37 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	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
SSAM6-06-1_01_BPC	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
SSAM6-05-1_01_BPC SSAM6-06-0_01_BPC	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
SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM6-05-0_01_BPC	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
SSAM5-05-0_01_BPC	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01180492	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 G01180492	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 SSAM5-05-1\_01\_BPC and SSAM5-05-1\_01\_BPC\_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
	SSAM5-05-1_01_BPC	SSAM5-05-1_01_BPC_FD				
2,3,7,8-TCDD	44	47	7 (≤50)	-	-	-
1,2,3,7,8-PeCDD	160	180	12 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	97	120	21 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	190	210	10 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	180	210	15 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	640	700	9 (≤50)	-	-	-
OCDD	840	880	5 (≤50)	-	-	-
2,3,7,8-TCDF	930	1100	17 (≤50)	-	-	-
1,2,3,7,8-PeCDF	1700	1900	11 (≤50)	-	-	-
2,3,4,7,8-PeCDF	890	1000	12 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAM5-05-1_01_BPC	SSAM5-05-1_01_BPC_FD				
1,2,3,4,7,8-HxCDF	3100	3500	12 ( $\leq 50$ )	-	-	-
1,2,3,6,7,8-HxCDF	2200	2300	4 ( $\leq 50$ )	-	-	-
2,3,4,6,7,8-HxCDF	510	430	17 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDF	460	480	4 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDF	7700	8400	9 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	3800	3800	0 ( $\leq 50$ )	-	-	-
OCDF	21000	21000	0 ( $\leq 50$ )	-	-	-



**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01180492**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01180492	SSAM6-06-1_01_BPC SSAM6-05-4_01_BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01180492	SSAM6-06-1_01_BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G01180492	SSAM6-05-1_01_BPC SSAM6-06-0_01_BPC	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)
G01180492	SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM6-05-0_01_BPC	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)
G01180492	SSAM5-05-0_01_BPC	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G01180492	SSAM6-06-4_01_BPC SSAM6-06-1_01_BPC SSAM6-05-4_01_BPC SSAM6-05-1_01_BPC SSAM5-05-4_01_BPC** SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM6-06-0_01_BPC SSAM6-05-0_01_BPC SSAM5-05-0_01_BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01180492	SSAM6-06-4_01_BPC SSAM6-06-1_01_BPC SSAM6-05-4_01_BPC SSAM6-05-1_01_BPC SSAM5-05-4_01_BPC** SSAM5-05-1_01_BPC SSAM5-05-1_01_BPC_FD SSAM6-06-0_01_BPC SSAM6-05-0_01_BPC SSAM5-05-0_01_BPC	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  
G01180492**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G01180492	SSAM6-06-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.41U pg/g 3.6U pg/g	A	bl
G01180492	SSAM6-05-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.27U pg/g 1.7U pg/g	A	bl
G01180492	SSAM5-05-4_01_BPC**	1,2,3,4,6,7,8-HpCDD OCDD	0.56U pg/g 3.3U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01180492**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24340N21  
 SDG #: G01180492  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Stage 2B/4

Date: 11/20/10  
 Page: 1 of 7  
 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: 9/16 - 9/17/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration <del>ACV</del>	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
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 = 6 + 7
XV.	Field blanks	N	

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

1	SSAM6-06-4_01_BPC	11	SSAM6-06-1_01_BPCMS	21	0265175	31	
2	SSAM6-06-1_01_BPC	12	SSAM6-06-1_01_BPCMSD	22		32	
3	SSAM6-05-4_01_BPC	13		23		33	
4	SSAM6-05-1_01_BPC	14		24		34	
5	SSAM5-05-4_01_BPC**	15		25		35	
6	SSAM5-05-1_01_BPC	16		26		36	
7	SSAM5-05-1_01_BPC FD	17		27		37	
8	SSAM6-06-0_01_BPC	18		28		38	
9	SSAM6-05-0_01_BPC	19		29		39	
10	SSAM5-05-0_01_BPC	20		30		40	

Notes: JQ

LDC #: 24340N2  
 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
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/MS Instrument performance check</b>				
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?	/			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	/			
<b>IV. Continuing calibration</b>				
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?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
<b>V. Blanks</b>				
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?	/			
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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?		/		
<b>VII. Laboratory control samples</b>				
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 #: 24340N21  
 SDG #: pu com

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: FA  
 2nd Reviewer: Q

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDF 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 PeCDD
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:

LDC #: 24340122

**VALIDATION FINDINGS WORKSHEET**

Blanks

Page: 1 of 1

Reviewer: FT

2nd Reviewer: R

\* E M P C

(bp)

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 all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 9/22/10

Associated samples: All

Blank analysis date: 10/6/10

Conc. units: 1017

Compound	Blank ID	Blank ID	5X	Sample Identification							
	0265173	MB		1	3	5					
F	0.14	0.70		0.41/4	0.27/4	0.56/4					
G	0.73	3.65		3.68/4	1.7/4	3.3/4					
K	0.058*	0.29									
Q	0.14*	0.70									
P	0.069*	0.345									
R	0.26	1.3									

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

VALIDATION FINDINGS WORKSHEET  
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".  
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 a MS/MSD analyzed every 20 samples of each matrix?  
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
		11 + 12	Around outside	100 R ( ) limit	+ % RPD ( )	were ( )	2	no good resin



# 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".

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

Y/N/N/A Was the SIN ratio all internal standard peaks  $\geq$  10?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		2	I	23 ( 40-135 )	1/w/p OMA G.R
			G	36 ( )	↓ O.P
		3	I	29 ( )	↓
			G	37 ( )	↓
				( )	
				( )	
		11	I	36 ( )	no qual MS
				( )	
				( )	
		12	H	34 ( )	no qual MS D
			I	20 ( )	
			E	39 ( )	
			G	30 ( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
<sup>13</sup> C-2,3,7,8-TCDF		K	<sup>13</sup> C-1,2,3,4-TCDD
<sup>13</sup> C-2,3,7,8-TCDD		L	
<sup>13</sup> C-1,2,3,7,8-PeCDF		M	<sup>13</sup> C-1,2,3,7,8,9-HxCDD
<sup>13</sup> C-1,2,3,7,8-PeCDD		N	
<sup>13</sup> C-1,2,3,6,7,8-HxCDF		O	
<sup>13</sup> C-1,2,3,6,7,8-HxCDD		P	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		Q	
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		R	
<sup>13</sup> 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".

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

#	Date	comp -Sample-ID-	Finding	Associated Samples	Qualifications
		<u>B, Q</u>	<u>xld cal and Range</u>	<u>2</u>	<u>J   P det (C)</u>
		<u>H, G, Q</u>	↓	<u>4, 8</u>	↓
		<u>H, I, K, L, P, Q</u>	↓	<u>6, 7, 9</u>	↓
		<u>H, I, Y, L</u> <u>H</u>	<u>↓</u>	<u>10</u>	↓

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24340N21

# 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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24340N21

**VALIDATION FINDINGS WORKSHEET**  
**Field Duplicates**

Page: 1 of 1  
 Reviewer: TJ  
 2nd Reviewer: f

**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? (fd)

Compound	Concentration (pg/g)		%RSD ≤50	(pg/g) Limits	Qualifications (Parent Only)
	6	7			
A	44	47	7		
B	160	180	12		
C	97	120	21		
D	190	210	10		
E	180	210	15		
F	640	700	9		
G	840	880	5		
H	930	1100	17		
I	1700	1900	11		
J	890	1000	12		
K	3100	3500	12		
L	2200	2300	4		
M	510	430	17		
N	460	480	4		
O	7700	8400	9		
P	3800	3800	0		
Q	21000	21000	0		

V:\FIELD DUPLICATES\24340N21.wpd

**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:

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	(C.S. 2-std)	Average RRF (Initial)	(C.S. 3-std)	RRF	%RSD	RRF	%RSD
1	1 CAL	7/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	0.9847	0.995	0.9847	3.68	3.68	0.9847	3.68
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	0.9687	0.983	0.9687	3.24	3.24	0.9687	3.24
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.1014	1.163	1.1014	5.17	5.17	1.1014	5.17
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.1128	1.072	1.1128	2.67	2.67	1.1128	2.67
2			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.3500	1.370	1.3500	1.98	1.98	1.3500	1.98
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
3			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								

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**  
**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:

$RRF = (A_x/C_x)/(A_{is}/C_{is})$   
 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 (initial)	%RSD	RRF (initial)	%RSD
1	ICAL PBDFs	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.0576		1.0576		1.020	3.32	1.020	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration Results Verification**

8290

**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  
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$  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	eev 52	10/6/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	1.01	1.8	1.01	1.8
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	1.06	7.3	1.06	7.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.14	2.3	1.14	2.3
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.10	3.1	1.10	3.1
			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.29	5.5	1.29	5.5
2	eev 37	10/9/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.02	3.9	1.02	3.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

**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 =  $100 * (MSR - MSDR) / 2 * (MSR + MSDR)$  MSR = Matrix spike percent recovery MS DR = Matrix spike duplicate percent recovery

MS/MSD samples: 11 & 12

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported	Recalculated
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	20.9	21.5	6.8	23.1	23.4	78	78	77	77	1.3	1.3
1,2,3,7,8-PeCDD	105	108	24	115	117	87	87	86	86	2.0	2.0
1,2,3,4,7,8-HxCDD	105	108	16	103	116	83	83	92	92	12	12
1,2,3,4,7,8,9-HpCDF	105	108	800	362	470	0	0	0	0	0	0
OCDF	209	215	4500	1520	2070	0	0	0	0	0	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 I**  
**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 =  $|\text{LCS} - \text{LCSD}| \cdot 2 / (\text{LCS} + \text{LCSD})$  LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 6265 175

Compound	Spike Added (ppb)		Spiked Sample Concentration (ppb)		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	18.5	NA	92	92								
1,2,3,7,8-PeCDD	100		98.2		98	98								
1,2,3,4,7,8-HxCDD	100		90.2		90	90								
1,2,3,4,7,8,9-HpCDF	100		97.0		97	97								
OCDF	200	↓	162	↓	81	81								

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

(a) The following nucleic masses were used:

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

S = internal/recovery standard

LDC #: 24340N2  
 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)

N N/A Were all reported results recalculated and verified for all level IV samples?  
 Y N N/A 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<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = 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. ~~#15~~ #5, 1, 2, 3, 4, 7, 8 - HxCDF  
 Conc. =  $\frac{(791237)(2000)}{(8530700)(1.22)(10.07)(0.919)}$   
 = 1.6 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8-TCDF			
		= 1191823 (2000)			
		557372000 (1.056)(10.07)(0.919)			
		=		0.44 pg/g	

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

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

**Collection Date:** September 22, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I230576

**Sample Identification**

SB01-25.0\_01\_BPC  
SB02-28.5\_01\_BPC\*\*  
SB02-28.5\_01\_BPC\_FD  
SB03-28.5\_01\_BPC  
SB01-25.0\_01\_BPCMS  
SB01-25.0\_01\_BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 6 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.
- UU 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 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
0269019-MB	9/26/10	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 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.38 pg/g 0.33 pg/g 0.20 pg/g 0.18 pg/g 0.51 pg/g 0.30 pg/g 1.1 pg/g	All samples in SDG G01230576

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
SB03-28.5_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD 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	0.47 pg/g 0.70 pg/g 1.1 pg/g 0.75 pg/g 2.2 pg/g 0.89 pg/g	0.47U pg/g 0.70U pg/g 1.1U pg/g 0.75U pg/g 2.2U pg/g 0.89U pg/g

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
SB02-28.5_01_BPC_FD	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	25 (40-135) 33 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	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 for samples on which Stage 4 review was performed.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0I230576	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 G0I230576	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 SB02-28.5\_01\_BPC\*\* and SB02-28.5\_01\_BPC\_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
	SB02-28.5_01_BPC**	SB02-28.5_01_BPC_FD				
2,3,7,8-TCDD	1.1	0.72U	-	0.38 (≤0.72)	-	-
1,2,3,7,8-PeCDD	3.2	3.6U	-	0.4 (≤3.6)	-	-
1,2,3,4,7,8-HxCDD	2.0	0.14	-	1.86 (≤3.6)	-	-
1,2,3,6,7,8-HxCDD	3.6	0.39	-	3.21 (≤3.6)	-	-
1,2,3,7,8,9-HxCDD	3.2	0.36	-	2.84 (≤3.6)	-	-
1,2,3,4,6,7,8-HpCDD	11	1.3	-	9.7 (≤3.6)	J (all detects)	A
OCDD	9.0	2.6	-	6.4 (≤7.2)	-	-
2,3,7,8-TCDF	24	1.4	-	22.6 (≤0.72)	J (all detects)	A
1,2,3,7,8-PeCDF	46	2.8	-	43.2 (≤3.6)	J (all detects)	A
2,3,4,7,8-PeCDF	22	1.1	-	20.9 (≤3.6)	J (all detects)	A
1,2,3,4,7,8-HxCDF	72	5.3	-	66.7 (≤3.6)	J (all detects)	A
1,2,3,6,7,8-HxCDF	56	4.5	-	51.5 (≤3.6)	J (all detects)	A
2,3,4,6,7,8-HxCDF	13	1.3	-	11.7 (≤3.6)	J (all detects)	A
1,2,3,7,8,9-HxCDF	9.7	0.89	-	8.81 (≤3.6)	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	170	16	-	154 (≤3.6)	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	71	7.1	-	63.9 (≤3.6)	J (all detects)	A
OCDF	290	31	-	259 (≤7.2)	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I230576**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I230576	SB02-28.5_01_BPC_FD	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (l)
G0I230576	SB01-25.0_01_BPC SB02-28.5_01_BPC** SB02-28.5_01_BPC_FD SB03-28.5_01_BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I230576	SB01-25.0_01_BPC SB02-28.5_01_BPC** SB02-28.5_01_BPC_FD SB03-28.5_01_BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0I230576	SB02-28.5_01_BPC** SB02-28.5_01_BPC_FD	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 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)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0I230576**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I230576	SB03-28.5_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD 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	0.47U pg/g 0.70U pg/g 1.1U pg/g 0.75U pg/g 2.2U pg/g 0.89U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I230576**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24340021

VALIDATION COMPLETENESS WORKSHEET

SDG #: G01230576

Stage 2B/4

Laboratory: Test America

Date: 11/20/10

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: 9/22/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/REV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LES
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 = 2, 3
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	SB01-25.0_01_BPC	11	0269019	21		31	
2	SB02-28.5_01_BPC**	12		22		32	
3	SB02-28.5_01_BPC_FD	13		23		33	
4	SB03-28.5_01_BPC	14		24		34	
5	SB01-25.0_01_BPCMS	15		25		35	
6	SB01-25.0_01_BPCMSD	16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: 16

LDC #: 24340021  
 SDG #: pu cones

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
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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"/>	
<b>III. Initial calibration</b>				
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 $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike, duplicates</b>				
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 checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
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 #: 24340 021  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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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?	X	✓	
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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDF 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:

LDC #: 2434002

**VALIDATION FINDINGS WORKSHEET**

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

Blanks

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 all samples associated with a method blank?

Y N N/A  
 Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A  
 Was the method blank contaminated?

\* **EMPS** (bl)

Blank extraction date: 7/26/10 Blank analysis date: 10/07/10 Associated samples: All

Conc. units: ppb

Compound	Blank ID	MP	5X	Sample Identification																																									
F	0.12*	0.6	4	0.47/u																																									
G	0.38	1.9		0.70*/u																																									
K	0.33	1.65		1.1/u																																									
L	0.20	1.0		0.75*/u																																									
M	0.18*	0.9																																											
<del>N</del>																																													
O	0.51*	2.55		2.2*/u																																									
P	0.30	1.5		0.89/u																																									
Q	1.1	5.5																																											

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







LDC #: 24340027

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

Page: 1 of 1  
 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".

(1)  
 Y N N/A  
 Y N N/A

Were the correct internal standard (IS), quantifications 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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	J/K detects (k)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24340021

**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? (fd)

Compound	Concentration (pg/g)		(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	3			
A	1.1	0.72U	0.38	≤0.72	
B	3.2	3.6U	0.4	≤3.6	
C	2.0	0.14	1.86	≤3.6	
D	3.6	0.39	3.21	≤3.6	
E	3.2	0.36	2.84	≤3.6	
F	11	1.3	9.7	≤3.6	J/A det
G	9.0	2.6	6.4	≤7.2	
H	24	1.4	22.6	≤0.72	J/A det
I	46	2.8	43.2	≤3.6	J/A det
J	22	1.1	20.9	≤3.6	J/A det
K	72	5.3	66.7	≤3.6	J/A det
L	56	4.5	51.5	≤3.6	J/A det
M	13	1.3	11.7	≤3.6	J/A det
N	9.7	0.89	8.81	≤3.6	J/A det
O	170	16	154	≤3.6	J/A det
P	71	7.1	63.9	≤3.6	J/A det
Q	290	31	259	≤7.2	J/A det

V:\FIELD DUPLICATES\24340021.wpd

**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:

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	(%SD)	Average RRF (Initial)	(%SD)	RRF	(%SD)	RRF	(%SD)
1	CAL	7/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.995	0.985	0.9849	0.9849	3.68	3.68	3.68	3.68
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.983	0.983	0.9681	0.9681	3.24	3.24	3.24	3.24
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.163	1.1014	1.1014	5.17	5.17	5.17	5.17
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.072	1.1128	1.1128	2.61	2.61	2.61	2.61
2			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.370	1.3500	1.3500	1.98	1.98	1.98	1.98
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
3			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								

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**  
**Initial Calibration Calculation Verification**

LDC #: 24340021  
 SDG #: see coms

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 Reviewer: F7  
 2nd Reviewer: [Signature]

**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,  
 $C_s$  = 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)	RRF (initial)	Average RRF (initial)	RRF (initial)	%RSD	%RSD	RRF (std)	%RSD
1	1012 DBMS	7/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.036	1.020	1.032	1.020	3.32	3.32	1.020	3.32
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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 #: 24340021  
 SDG #: see com

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration Results Verification**

Page: 1 of 1  
 Reviewer: F7  
 2nd Reviewer: R

**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 =  $(A_x)(C_b) / (A_b)(C_x)$       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	eww 515	10/6/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	6.795	1.02	2.9	1.02	2.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.783	1.03	4.9	1.03	4.9
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.12	4.0	1.12	4.0
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.05	1.7	1.05	1.7
			OCDF ( <sup>13</sup> C-OCDF)	1.370	1.27	7.2	1.27	7.2
2	eww PB225 537	10/9/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.02	3.9	1.02	3.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

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 =  $100 * MSR - MSDR$  MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 546

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported	Recalculated
	MS	MSD		MS	MSD	Percent Recovery	Recalc.	Percent Recovery	Recalc.		
2,3,7,8-TCDD	21.6	21.5	0.87	24.4	25.7	109	109	115	115	5.2	5.2
1,2,3,7,8-PeCDD	108	108	3.6	126	128	113	113	115	115	1.6	1.6
1,2,3,4,7,8-HxCDD	108	108	2.6	126	131	114	114	119	119	4.1	4.1
1,2,3,4,7,8,9-HpCDF	108	108	100	212	351	102	102	232	232	49	49
OCDF	216	215	450	577	1010	61	61	261	261	54	54

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.





Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

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

(e) The following nucleidic masses were used:

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

S = internal/recovery standard

LDC #: 24340021  
 SDG #: pel 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?

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

- A<sub>s</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>i</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>s</sub> = 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. #2 2, 3, 7, 8-TCDF

$$\text{Conc.} = \frac{(685730)(2000)}{(13414670)(0.98)(10.32)(0.919)}$$

$$= 1.1 \text{ pg/g}$$

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8-TCDF			
		= 25326569 (2000)			
		207585232 (1.056)(10.32)(0.919)			
		= 24 pg/g			

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

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

**Collection Date:** September 24, 2010

**LDC Report Date:** November 22, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I250521

**Sample Identification**

SSAQ3-03-0.00BPC  
SSAQ3-03-0.00BPCMS  
SSAQ3-03-0.00BPCMSD

## Introduction

This data review covers 3 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was 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.
- UU 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% .

## 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.

## 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
0284345-MB	10/11/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.32 pg/g 0.64 pg/g 0.15 pg/g 0.16 pg/g	All samples in SDG G01250521

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

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. Percent recoveries (%R) were within 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
SSAQ3-03-0.00BPC	<sup>13</sup> C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01250521	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:



Sample	Finding	Flag	A or P
All samples in SDG G0I250521	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

## **XII. System Performance**

Raw data were not reviewed for this SDG.

## **XIII. Overall Assessment of Data**

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

## **XIV. Field Duplicates**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I250521**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I250521	SSAQ3-03-0.00BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0I250521	SSAQ3-03-0.00BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I250521	SSAQ3-03-0.00BPC	All compounds reported as 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  
G0I250521**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I250521**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 24340P21  
 SDG #: G0I250521  
 Laboratory: Test America

Date: 11/20/10  
 Page: 1 of 1  
 Reviewer: PJ  
 2nd Reviewer: PJ

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: 9/24/10
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	LC>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
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:

2011

1	SSAQ3-03-0.00BPC	11	02 84345-MB	21		31	
2	SSAQ3-03-0.00BPCMS	12		22		32	
3	SSAQ3-03-0.00BPCMSD	13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes: JG

# 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:

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LDC #: 24 340P2)

**VALIDATION FINDINGS WORKSHEET**

**Blanks**

Page: 1 of 1

Reviewer: FT

2nd Reviewer: R

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 all samples associated with a method blank?

Y/N N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y/N N/A

Was the method blank contaminated?

Blank extraction date: 10/11/10

Blank analysis date: 10/13/10

Associated samples: All 75X

Conc. units: pg/g

Compound	Blank ID	Sample Identification
F	0284345	MB
G	0.32	
B	0.64	
Q	0.15 *	
	0.16	

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

**VALIDATION FINDINGS WORKSHEET**  
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 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
		2 + 3	G	150 (80-137)	( ) ( )	( ) ( )	1	no qual lesion
			H	( ) ( )	48 (79-137)	44 (30)		
			B	156 (81-137)	( ) ( )	38 (30)		
			P	140 (79-137)	( ) ( )	( ) ( )		
			Q	194 (75-141)	( ) ( )	46 (30)	↓	
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
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				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		

**VALIDATION FINDINGS WORKSHEET**

Reviewer: FT

2nd Reviewer: 2

**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".

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  $\geq 10$ ?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		/	<u>I</u>	<u>38</u> ( <u>40-135</u> )	<u>J/US/P</u> <u>9,9</u>
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
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				( )	

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			$^{13}C$ -1,2,3,7,8,9-HxCDD	
C.	$^{13}C$ -1,2,3,7,8-PeCDF				
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.	$^{13}C$ -OCDF				

LDC #: 24340P

**VALIDATION FINDINGS WORKSHEET**  
**Compound Quantitation and Reported CRQLs**

Page: 1 of 1  
Reviewer: FT  
2nd Reviewer: R

**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
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)

Comments: See sample calculation verification worksheet for recalculations