



## 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 8, 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 27, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 24445:**

**SDG #**

**Fraction**

G0H120523, G0H280490, G0I020523, G0I140549

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 #: 24445  
 SDG #: G0H120523, G0H280490, G0I020523, G0I140549

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_LDC24445_120710.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 9, 2010

**LDC Report Date:** December 7, 2010

**Matrix:** Soil/Water

**Parameters:** Dioxins/Dibenzofurans

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

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H120523

**Sample Identification**

SSAQ4-08-10BPC\*\*  
SSAQ4-08-10BPC\_FD  
SSAQ4-08-1BPC  
SSAQ4-08-5BPC  
EB-08092010

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 4 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
0228254-MB	8/16/10	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 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	2.5 pg/L 2.0 pg/L 2.6 pg/L 7.7 pg/L 2.5 pg/L 1.9 pg/L 1.9 pg/L 1.5 pg/L 2.6 pg/L 2.7 pg/L 2.0 pg/L 3.7 pg/L	All water samples in SDG G0H120523
0228363-MB	8/16/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.096 pg/g 0.80 pg/g 0.064 pg/g 0.26 pg/g 0.087 pg/g 0.16 pg/g	All soil samples in SDG G0H120523

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-08092010	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	1.6 pg/L 1.2 pg/L 2.4 pg/L 4.3 pg/L 11 pg/L 2.6 pg/L 2.5 pg/L 10 pg/L	1.6U pg/L 1.2U pg/L 2.4U pg/L 4.3U pg/L 11U pg/L 2.6U pg/L 2.5U pg/L 10U pg/L

Sample EB08092010 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
EB08092010	8/9/10	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 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	1.1 pg/L 1.6 pg/L 1.2 pg/L 2.4 pg/L 4.3 pg/L 5.7 pg/L 11 pg/L 6.9 pg/L 14 pg/L 11 pg/L 2.6 pg/L 2.5 pg/L 2.7 pg/L 10 pg/L 48 pg/L	All soil samples in SDG G0H120523



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

### VI. Matrix Spike/Matrix Spike Duplicates

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
SSAQ4-08-1BPC	<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
SSAQ4-08-5BPC	<sup>13</sup> C-OCDD	21 (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
SSAQ4-08-10BPC**	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
SSAQ4-08-10BPC_FD SSAQ4-08-1BPC	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
SSAQ4-08-5BPC	OCDF	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 G0H120523	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 G0H120523	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 SSAQ4-08-10BPC\*\* and SSAQ4-08-10BPC\_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
	SSAQ4-08-10BPC	SSAQ4-08-10BPC_FD				
2,3,7,8-TCDD	3.6	2.7	29 (≤50)	-	-	-
1,2,3,7,8-PeCDD	23	16	36 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	15	12	22 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	13	25	63 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	17	12	34 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	120	110	9 (≤50)	-	-	-
OCDD	190	180	5 (≤50)	-	-	-
2,3,7,8-TCDF	120	99	19 (≤50)	-	-	-
1,2,3,7,8-PeCDF	450	380	17 (≤50)	-	-	-
2,3,4,7,8-PeCDF	220	190	15 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	670	650	3 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	670	590	13 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	150	150	0 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	110	98	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	2700	2700	0 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1100	1100	0 (≤50)	-	-	-
OCDF	12000	13000	8(≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H120523**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H120523	SSAQ4-08-1BPC SSAQ4-08-5BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (l)
G0H120523	SSAQ4-08-10BPC**	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)
G0H120523	SSAQ4-08-10BPC_FD SSAQ4-08-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-5BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H120523	SSAQ4-08-10BPC SSAQ4-08-10BPC_FD	1,2,3,6,7,8-HxCDD	J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H120523**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H120523	EB-08092010	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	1.6U pg/L 1.2U pg/L 2.4U pg/L 4.3U pg/L 11U pg/L 2.6U pg/L 2.5U pg/L 10U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H120523**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24445A21

SDG #: G0H120523

Laboratory: Test America

Stage 2B/4

Date: 12/01/10

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: 8/9/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration#CV	Δ	
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	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	Δ	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 = 1, 2
XV.	Field blanks	SW	EB = 5

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

3	1	SSAQ4-08-10BPC**	11	0230135	21	31
4	2	SSAQ4-08-10BPC_FD	122	0228254	22	32
5	3	SSAQ4-08-1BPC ✓	13		23	33
9	4	SSAQ4-08-5BPC ✓	14		24	34
14	5	EB-08092010	15		25	35
	6		16		26	36
	7		17		27	37
	8		18		28	38
	9		19		29	39
	10		20		30	40

Notes: \_\_\_\_\_

LDC #: 24445 A21  
 SDG #: pu cones

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.	<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 type="checkbox"/>	<input checked="" 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 #: 24445 A21  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

VIII: Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>		
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>		
IX: Internal standards			
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>		
Was the minimum S/N ratio of all internal standard peaks > 10?	<input checked="" type="checkbox"/>		
X: Target compound identification			
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	<input checked="" type="checkbox"/>		
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	<input checked="" type="checkbox"/>		
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	<input checked="" type="checkbox"/>		
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>		
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>		
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	<input checked="" type="checkbox"/>		
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ 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"/>		
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:





LDC #: 2445A21

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

Page: 1 of 1  
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/g

Sampling date: 8/9/10

Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: All soils 75X

Compound	Blank ID	Sample Identification									
	<u>5</u>										
C	<u>1.1</u>										
D	<u>1.6</u>										
E	<u>1.2</u>										
F	<u>2.4</u>										
G	<u>4.3</u>										
H	<u>5.7</u>										
I	<u>11</u>										
J	<u>6.9</u>										
K	<u>14</u>										
L	<u>11</u>										
M	<u>2.6</u>										
N	<u>2.5</u>										
O	<u>2.7</u>										
P	<u>10</u>										
Q	<u>4X</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".



LDC #: 24445 A21

### 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 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	Compd 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)
		0, 1, 2	x'd and range	1	J/A detects (e)
		0, 2	↓	2, 3	↓
		0	↓	4	↓

Comments: See sample calculation verification worksheet for recalculations

LDC#:24445A21

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

Page: 1 of 1  
Reviewer:     
2nd Reviewer:   

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)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	2				
A	3.6	2.7	29			
B	23	16	36			
C	15	12	22			
D	13	25	63			J/A dot
E	17	12	34			
F	120	110	9			
G	190	180	5			
H	120	99	19			
I	450	380	17			
J	220	190	15			
K	670	650	3			
L	670	590	13			
M	150	150	0			
N	110	98	12			
O	2700	2700	0			
P	1100	1100	0			
Q	12000	13000	8			

V:\FIELD DUPLICATES\24445A21.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)	%RSD	Average RRF (initial)	%RSD	RRF (L.S. std)	%RSD	RRF (L.S. std)	%RSD
1	ICAL	7/20/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.003	3.13	1.003	3.13	1.0326	3.13	1.0326	3.13
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.142	11.9	1.0790	11.9	1.0790	11.9	1.0790	11.9
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.130	6.08	1.0831	6.08	1.0831	6.08	1.0831	6.08
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.079	1.38	1.073	1.38	1.073	1.38	1.073	1.38
			OCDF ( <sup>13</sup> C-OCDF)	1.467	3.12	1.4726	3.12	1.4726	3.12	1.4726	3.12
2	ICAL	7/27/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.875	14.2	0.87	14.2	0.87	14.2	0.87	14.2
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.957	13.5	0.93	13.5	0.93	13.5	0.93	13.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.107	12.5	1.13	12.5	1.13	12.5	1.13	12.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.026	13.6	1.07	13.6	1.07	13.6	1.07	13.6
			OCDF ( <sup>13</sup> C-OCDF)	1.445	18.1	1.55	18.1	1.55	18.1	1.55	18.1
3	ICAL	8/16/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.00187	4.19	1.00395	4.19	1.00395	4.19	1.00395	4.19
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.16617	8.10	1.15763	8.10	1.15763	8.10	1.15763	8.10
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.14627	8.30	1.16196	8.30	1.16196	8.30	1.16196	8.30
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.08648	6.01	1.10879	6.01	1.10879	6.01	1.10879	6.01
			OCDF ( <sup>13</sup> C-OCDF)	1.59850	8.41	1.58313	8.41	1.58313	8.41	1.58313	8.41

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:

$RRF = (A_x/C_x)/(A_s/C_s)$   
 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,  
 $A_s$  = Area of associated internal standard  
 $C_s$  = 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 (std)	RRF (std)	%RSD	%RSD	
1	1CAL	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	
	DB225		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
2			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)								
3			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	ave 17:33 DB225	8/30/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.052	0.96	8.7	0.96	8.7
			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	ave 8:40	8/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.875	0.88	0	0.88	0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.957	0.87	9.5	0.87	9.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.107	1.03	7.0	1.03	7.0
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.026	1.07	4.2	1.07	4.2
			OCDF ( <sup>13</sup> C-OCDF)	1.445	1.58	9.2	1.58	9.2
3	ave 21:35	8/20/8	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.00187	0.90044	10.1	0.90044	10.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.16617	1.0963	6.0	1.0963	6.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.14627	1.16495	1.6	1.16495	1.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.08648	1.08079	0.5	1.0879	0.5
			OCDF ( <sup>13</sup> C-OCDF)	1.54852	1.44162	6.9	1.44162	6.9

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.





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	407.7618	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>3</sub> <sup>35</sup> Cl <sub>5</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	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 <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	C <sub>12</sub> H <sub>3</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 <sup>35</sup> 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>4</sub> O <sub>2</sub>	TCDD		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>3</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 <sup>35</sup> 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	<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	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (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>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <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		[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>5</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>5</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	C <sub>12</sub> <sup>35</sup> Cl <sub>5</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>4</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> ClO <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>5</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>5</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>6</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		C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
369.8919		M+4	<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)	[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>5</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>5</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	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	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>6</sub> O	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	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							
	391.8127	M+2	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+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							
	403.8529	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)							
	445.7555	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)							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>13</sub>	OCDFE							
				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



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

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

**Collection Date:** August 26, 2010

**LDC Report Date:** December 8, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

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

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0H280490

**Sample Identification**

BDT-2-S-20-10BPC  
BDT-2-S-20-12BPC  
BDT-2-S-20-14BPC\*\*  
BDT-2-S-20-2BPC  
BDT-2-S-20-4BPC  
BDT-2-S-20-6BPC  
BDT-2-S-20-6BPC\_FD  
BDT-2-S-20-8BPC  
BDT-2-S-15-10BPC  
BDT-2-S-15-12BPC  
BDT-2-S-15-14BPC\*\*  
BDT-2-S-15-2BPC  
BDT-2-S-15-4BPC  
BDT-2-S-15-6BPC  
BDT-2-S-15-8BPC  
BDT-2-S-15-2BPC\_FD  
BDT-2-S-20-10BPCMS  
BDT-2-S-20-10BPCMSD

\*\*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.
- 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
0257312-MB	9/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.081 pg/g 1.4 pg/g 0.089 pg/g 0.23 pg/g	All samples in SDG G0H280490

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 recovery (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

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

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

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

Not applicable.

#### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-2-S-20-10BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 37 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-20-14BPC**	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
BDT-2-S-15-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	38 (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

## X. Target Compound Identifications

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

## XI. Project Quantitation Limit

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

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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H280490	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 G0H280490	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

## XII. System Performance

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples BDT-2-S-20-6BPC and BDT-2-S-20-6BPC\_FD and samples BDT-2-S-15-2BPC and BDT-2-S-15-2BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-2-S-20-6BPC	BDT-2-S-20-6BPC_FD				
2,3,7,8-TCDD	1.4	1.6	13 (≤50)	-	-	-
1,2,3,7,8-PeCDD	4.5	5.1	12 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	3.8	4.3	12 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	7.0	7.9	12 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	5.6	6.6	16 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	22	25	13 (≤50)	-	-	-
OCDD	49	64	27 (≤50)	-	-	-
2,3,7,8-TCDF	33	38	14 (≤50)	-	-	-
1,2,3,7,8-PeCDF	53	60	12 (≤50)	-	-	-
2,3,4,7,8-PeCDF	29	33	13 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	96	100	4 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	65	69	6 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	21	25	17 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	9.8	11	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	280	310	10 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	120	120	0 (≤50)	-	-	-
OCDF	590	660	11 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-2-S-15-2BPC	BDT-2-S-15-2BPC_FD				
2,3,7,8-TCDD	12	11	9 (≤50)	-	-	-
1,2,3,7,8-PeCDD	42	40	5 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	41	34	19 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	68	70	3 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	55	56	2 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	230	220	4 (≤50)	-	-	-
OCDD	620	540	14 (≤50)	-	-	-
2,3,7,8-TCDF	300	270	11 (≤50)	-	-	-
1,2,3,7,8-PeCDF	490	440	11 (≤50)	-	-	-
2,3,4,7,8-PeCDF	270	240	12 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	920	870	6 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	550	570	4 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	170	180	6 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	86	100	15 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	2800	2400	15 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1100	1000	10 (≤50)	-	-	-
OCDF	6300	7000	11 (≤50)	-	-	-

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H280490**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-14BPC**	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280490	BDT-2-S-15-10BPC	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)
G0H280490	BDT-2-S-20-2BPC BDT-2-S-20-4BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-2BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-4BPC BDT-2-S-15-2BPC_FD	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC_FD BDT-2-S-20-8BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-2BPC BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC_FD BDT-2-S-20-8BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-2BPC BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	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  
G0H280490**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H280490**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24445B21  
 SDG #: G0H280490  
 Laboratory: Test America

Stage 2B/4

Date: 12/01/10  
 Page: 1 of 1  
 Reviewer: FB  
 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: 8/26/10
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	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 = 6, 7 18, 14
XV.	Field blanks	N	

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

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

soil

1	BDT-2-S-20-10BPC	11	BDT-2-S-15-14BPC**	21	0259197	31
2	BDT-2-S-20-12BPC	12	BDT-2-S-15-2BPC D	22	0257312	32
3	BDT-2-S-20-14BPC**	13	BDT-2-S-15-4BPC	23		33
4	BDT-2-S-20-2BPC	14	BDT-2-S-15-6BPC	24		34
5	BDT-2-S-20-4BPC	15	BDT-2-S-15-8BPC	25		35
6	BDT-2-S-20-6BPC	16	BDT-2-S-15-2BPC_FD D	26		36
7	BDT-2-S-20-6BPC_FD	17	BDT-2-S-20-10BPCMS	27		37
8	BDT-2-S-20-8BPC	18	BDT-2-S-20-10BPCMSD	28		38
9	BDT-2-S-15-10BPC	19		29		39
10	BDT-2-S-15-12BPC	20		30		40

Notes: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

LDC #: 24445 B21  
 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 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 #: 24445 B21  
 SDG #: mu cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: F1  
 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 $\geq 2.5$ ?	✓		
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:











LDC#: 24445B21

**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?

(fd)

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	6	7				
A	1.4	1.6	13	<del>2</del>		
B	4.5	5.1	12			
C	3.8	4.3	12			
D	7.0	7.9	12			
E	5.6	6.6	16			
F	22	25	13			
G	49	64	27			
H	33	38	14			
I	53	60	12			
J	29	33	13			
K	96	100	4			
L	65	69	6			
M	21	25	17			
N	9.8	11	12			
O	280	310	10			
P	120	120	0			
Q	590	660	11			

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LDC#: 23906B4

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

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

**METHOD:** Metals (EPA Method 6020/7000)

Y N NA  
Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	12	16				
A	12	11	9			
B	42	40	5			
C	41	34	19			
D	68	70	3			
E	55	56	2			
F	230	220	4			
G	620	540	14			
H	300	270	11			
I	490	440	11			
J	270	240	12			
K	920	870	6			
L	550	570	4			
M	170	180	6			
N	86	100	15			
O	2800	2400	15			
P	1100	1000	10			
Q	6300	7000	11			

V:\FIELD DUPLICATES\24445B21.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:

$RRF = (A_s)(C_{is}) / (A_{is})(C_s)$   
 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 (CS3 std)	RRF (CS3 std)	%RSD	%RSD	RRF (CS3 std)	%RSD
1	PB225 ICAL	7/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.056	1.02	1.02	3.32	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-OCDD)								
2	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	1.2609	5.52
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.252	1.252	1.2887	1.2887	4.00	4.00	1.2887	4.00
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.202	1.202	1.2152	1.2152	5.27	5.27	1.2152	5.27
			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	1.2654	5.62
			OCDF ( <sup>13</sup> C-OCDD)	1.892	1.892	1.9979	1.9979	6.95	6.95	1.9979	6.95
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 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	00V 2116	10/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.19	12.9	1.19	12.9
	PB 225		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	CON 22:42	9/28/10	OCDF ( <sup>13</sup> C-OCDF)	1.169	1.10	6.3	1.10	6.3
			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.252	1.14	9.3	1.14	9.3
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.202	1.18	1.0	1.18	1.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.180	1.21	2.9	1.21	2.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.892	1.77	6.6	1.77	6.6
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: 17 + 18

Compound	Spike Added (pg)		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	21.1	21.3	0.36	17.3	19.5	80	80	90	90	12	12
1,2,3,7,8-PeCDD	105	106	1.4	86.8	92.4	81	81	86	86	6.3	6.3
1,2,3,4,7,8-HxCDD	105	106	1.1	79.8	89.8	75	75	83	83	12	12
1,2,3,4,7,8,9-HpCDF	105	106	3.7	145	308	102	102	255	255	72	72
OCDF	211	213	16.0	445	1230	135	135	504	504	94	94

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>(b)</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>35</sub> 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>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>8</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>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> 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>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>8</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		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</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	C <sub>12</sub> H <sub>35</sub> Cl <sub>8</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>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>9</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>35</sub> Cl <sub>8</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	HxCDF		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>8</sub> <sup>37</sup> ClO	OCDF			
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	DCDF			
	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> ClO <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	469.7790	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	<sup>13</sup> 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+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</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	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	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> ClO	PeCDD (S)	[422.9278]	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	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	HxCDF		LOCK		C <sub>10</sub> F <sub>17</sub>	DCDF			
[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>3</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	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	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	<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>	HxCDD								
	391.8127	M+4	<sup>13</sup> 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	<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>	HxCDD (S)								
	403.8529	M+4	<sup>13</sup> 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>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDD (S)								
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</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





**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:** December 8, 2010

**Matrix:** Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0I020523

**Sample Identification**

EB-08312010

## 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.

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.
- 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% .

## 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
02651243-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 samples in SDG G0I020523

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-08312010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.5 pg/L 34 pg/L 7.8 pg/L	3.5U pg/L 34U pg/L 7.8U pg/L

Sample EB-8312010 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-08312010	8/31/10	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 1,2,3,7,8-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	1.2 pg/L 2.3 pg/L 2.2 pg/L 3.5 pg/L 34 pg/L 2.9 pg/L 4.2 pg/L 4.2 pg/L 3.0 pg/L 2.4 pg/L 7.8 pg/L 2.8 pg/L 12 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 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 samples in SDG G01020523	J+ (all detects)	P

## 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 CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01020523	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 G01020523	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 G0I020523**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I020523	EB-08312010	2,3,4,6,7,8-HxCDF	J+ (all detects)	P	Laboratory control samples (%R) (l)
G0I020523	EB-08312010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I020523	EB-08312010	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 G0I020523**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I020523	EB08312010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.5U pg/L 34U pg/L 7.8U pg/L	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0I020523**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24445C21

SDG #: G01020523

Laboratory: Test America

Stage 2B

Date: 12/01/10

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: 8/31/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	6L sample
VII.	Laboratory control samples	SW	LES
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
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 = Rinsate  
FB = Field blank

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples:  
water

1	EB-08312010	11	8/25/10	21	31
2		12	0251243	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: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



# 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:** September 10, 2010

**LDC Report Date:** December 8, 2010

**Matrix:** Soil/Water

**Parameters:** Dioxins/Dibenzofurans

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

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G01140549

**Sample Identification**

SSAJ8-03-1BPC  
SSAJ8-03-3BPC  
SSAJ8-03-3BPC\_FD  
SSAJ8-03-5BPC  
SSAJ8-03-8BPC  
SSAJ8-03-10BPC\*\*  
SSAO7-07-0BPC\*\*  
EB-09102010  
SSAO7-07-0BPCMS  
SSAO7-07-0BPCMSD

\*\*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.
- 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
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 G01140549

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
SSAJ8-03-1BPC	OCDD	3.1 pg/g	3.1U pg/g
SSAJ8-03-3BPC	OCDD	3.2 pg/g	3.2U pg/g
SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD OCDD	0.11 pg/g 0.84 pg/g	0.11U pg/g 0.84U pg/g
SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.057 pg/g 1.0 pg/g 0.16 pg/g 0.14 pg/g	0.057U pg/g 1.0U pg/g 0.16U pg/g 0.14U pg/g
SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.55 pg/g 0.93 pg/g 0.10 pg/g 0.076 pg/g	0.55U pg/g 0.93U pg/g 0.10U pg/g 0.076U pg/g

Sample EB09102010 was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

## 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
SSAJ8-03-1BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 37 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAJ8-03-3BPC	<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
SSAJ8-03-5BPC	<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
SSAJ8-03-10BPC**	<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
SSAO7-07-0BPC**	<sup>13</sup> C-OCDD	334 (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
SSAJ8-03-10BPC**	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 G01140549	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 G01140549	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 SSAJ8-03-3BPC and SSAJ8-03-3BPC\_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
	SSAJ8-03-3BPC	SSAJ8-03-3BPC_FD				
2,3,7,8-TCDD	0.26	0.091	-	0.169 (≤0.55)	-	-
1,2,3,7,8-PeCDD	0.68	0.21	-	0.47 (≤2.8)	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAJ8-03-3BPC	SSAJ8-03-3BPC_FD				
1,2,3,4,7,8-HxCDD	0.50	0.11	-	0.39 (≤2.8)	-	-
1,2,3,6,7,8-HxCDD	1.0	0.29	-	0.71 (≤2.8)	-	-
1,2,3,7,8,9-HxCDD	0.81	0.17	-	0.64 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDD	3.4	0.87	-	2.53 (≤2.8)	-	-
OCDD	3.2	0.84	-	2.36 (≤5.5)	-	-
2,3,7,8-TCDF	6.0	1.2	-	4.8 (≤0.55)	J (all detects)	A
1,2,3,7,8-PeCDF	11	2.2	-	8.8 (≤2.8)	J (all detects)	A
2,3,4,7,8-PeCDF	6.0	1.1	-	4.9 (≤2.8)	J (all detects)	A
1,2,3,4,7,8-HxCDF	16	3.0	-	13 (≤2.8)	J (all detects)	A
1,2,3,6,7,8-HxCDF	13	2.7	-	10.3 (≤2.8)	J (all detects)	A
2,3,4,6,7,8-HxCDF	2.8	0.63	-	2.17 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	2.5	0.62	-	1.88 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	51	10	-	41 (≤2.8)	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	21	4.6	-	16.4 (≤2.8)	J (all detects)	A
OCDF	110	20	-	90 (≤5.5)	J (all detects)	A

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G01140549**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01140549	SSAJ8-03-1BPC	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01140549	SSAJ8-03-3BPC SSAJ8-03-5BPC SSAJ8-03-10BPC** SSAO7-07-0BPC**	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G01140549	SSAJ8-03-10BPC**	2,3,7,8-TCDF	None	P	Project Quantitation Limit (e)
G01140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G01140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G01140549	SSAJ8-03-3BPC SSAJ8-03-3BPC_FD	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)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G01140549**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G01140549	SSAJ8-03-1BPC	OCDD	3.1U pg/g	A	bl
G01140549	SSAJ8-03-3BPC	OCDD	3.2U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G01140549	SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD OCDD	0.11U pg/g 0.84U pg/g	A	bl
G01140549	SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.057U pg/g 1.0U pg/g 0.16U pg/g 0.14U pg/g	A	bl
G01140549	SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.55U pg/g 0.93U pg/g 0.10U pg/g 0.076U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G01140549**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 24445D21

SDG #: G01140549

Laboratory: Test America

Stage 2B/4

Date: 12/01/10

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: 9/10/10
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	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	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 2, 3
XV.	Field blanks	ND	EB = 8

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

*Call + walk*

1	1	SSAJ8-03-1BPC	11	0263138.	21		31	
2	1	SSAJ8-03-3BPC	12	0281099	22		32	
3	1	SSAJ8-03-3BPC_FD	13		23		33	
4	1	SSAJ8-03-5BPC	14		24		34	
5	1	SSAJ8-03-8BPC	15		25		35	
6	1	SSAJ8-03-10BPC**	16		26		36	
7	1	SSAO7-07-0BPC**	17		27		37	
8	2	EB-09102010	18		28		38	
9		SSAO7-07-0BPCMS	19		29		39	
10		SSAO7-07-0BPCMSD	20		30		40	

Notes: \_\_\_\_\_

LDC #: 24445 D21  
 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 < 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 type="checkbox"/>	<input checked="" 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 #: 24445 D21  
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

<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 ± 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"/>		
<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:





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
			I	27 ( 40-135 )	S/N > 10
			G	37 ( )	9, 10
		2	I	34 ( )	9, 10
			I	35 ( )	9, 10
		6	I	32 ( )	9, 10
		7	I	33 ( )	9, 10
		9	H	29 ( )	no qual MS
			I	20 ( )	
			G	29 ( )	
		10	H	24 ( )	MSD
			I	17 ( )	
			G	24 ( )	

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. <sup>13</sup> C-1,2,3,7,8,9-HxCDD	
D. <sup>13</sup> C-1,2,3,7,8-PeCDD		N. <sup>13</sup> C-1,2,3,7,8,9-HxCDF	
F. <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	

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

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		<i>compd</i>	All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		H	no 2nd column confirmation was performed	6	<del>JK</del> . none / p (e)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24445D21

VALIDATION FINDINGS WORKSHEET  
Field Duplicates

Page: 1 of 1  
Reviewer:         
2nd Reviewer:       

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?

(bd)

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	3				
A	0.26	0.091		0.169	≤0.55	
B	0.68	0.21		0.47	≤2.8	
C	0.50	0.11		0.39	≤2.8	
D	1.0	0.29		0.71	≤2.8	
E	0.81	0.17		0.64	≤2.8	
F	3.4	0.87		2.53	≤2.8	
G	3.2	0.84		2.36	≤5.5	
H	6.0	1.2		4.8	≤0.55	J/A det
I	11	2.2		8.8	≤2.8	↓
J	6.0	1.1		4.9	≤2.8	↓
K	16	3.0		13	≤2.8	↓
L	13	2.7		10.3	≤2.8	↓
M	2.8	0.63		2.17	≤2.8	-
N	2.5 <del>f</del>	0.62		1.88 4.92	≤2.8	-
O	51	10		41	≤2.8	J/A det
P	21	4.6		16.4	≤2.8	↓
Q	110	20		90	≤5.5	↓

**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)	%RSD	Average RRF (initial)	%RSD	RRF (initial)	%RSD	RRF (initial)	%RSD
1	1CAL	9/14/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.984	11.8	0.984	11.8	1.05	11.8	1.05	11.8
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	10.8	1.032	10.8	1.06	10.8	1.06	10.8
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	12.7	1.141	12.7	1.25	12.7	1.25	12.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	12.3	1.134	12.3	1.26	12.3	1.26	12.3
			OCDF ( <sup>13</sup> C-OCDF)	2.118	15.3	2.118	15.3	2.36	15.3	2.36	15.3
2	DB225	7/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	3.32	1.056	3.32	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)								
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 #: 24445 D 21

SDG #: per conts

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

Page: 1 of 1

Reviewer: FJ

2nd Reviewer: JL

**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	ave 19:46	10/5/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.984	1.03	4.5	1.03	4.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.032	1.03	0.1	1.03	0.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.141	1.27	11.6	1.27	11.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.134	1.25	10.2	1.25	10.2
			OCDF ( <sup>13</sup> C-OCDF)	2.118	2.0	5.4	2.0	5.4
2	ave 20:25	10/8/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.056	1.04	1.2	1.04	1.2
	2:11		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 =  $100 * MSR - MSDR / (MSR + MSDR)$  MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 9 + 10

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/g)		Matrix Spike		Matrix Spike Duplicate		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery			
				Reported	Recalc.	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	19.8	20.1	3.1	28.5	26.6	129	129	117	117	6.9	6.9
1,2,3,7,8-PeCDD	98.7	100	8.6	116	113	109	109	104	104	2.6	2.6
1,2,3,4,7,8-HxCDD	↓	↓	5.9	124	126	119	119	119	119	1.9	1.9
1,2,3,4,7,8,9-HpCDF	↓	↓	250	893	460	654	654	212	212	64	64
OCDF	198	201	1200	4800	2010	1800	1800	381	381	82	82

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>2</sub> O	TODF	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	TODF		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	TODF (S)		417.8250	M	C <sub>12</sub> H <sub>35</sub> Cl <sub>7</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	TODF (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>3</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> 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 <sub>2</sub>	TCDD (S)		435.8169	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <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>3</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>6</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>4</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 (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>7</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
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]	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HpCDFE		LOCK		C <sub>10</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>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	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	HxCDF						
		383.8639	M	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> O	HxCDF (S)						
	385.8610	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> O	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	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	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 (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>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDD (S)							
	[430.9728]	LOCK	C <sub>9</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

