



**LABORATORY DATA CONSULTANTS, INC.**

7750 El Camino Real, Suite 2L Carlsbad, CA 92009 Phone: 760/634-0437 Fax: 760/634-0439

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

June 2, 2010

SUBJECT: Tronox LLC Facility, 2010 PCS, Henderson, Nevada,  
Data Validation

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on May 5, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 23125:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
G0D090441, G0D100462, G0D100464, G0D120488, G0D140435	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 Superfund Organic Methods Data Review, June 2008

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

Sincerely,

Erlinda T. Rauto  
Operations Manager/Senior Chemist



**Tronox PCS  
Data Validation Reports  
LDC# 23125**

Dioxins/Dibenzofurans

**LDC**

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 7, 2010  
**LDC Report Date:** May 25, 2010  
**Matrix:** Soil/Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D090441

**Sample Identification**

SSAJ8-02-1BPC\*\*  
SSAJ8-02-1BPC-FD  
SSAK8-01-1BPC\*\*  
SSAK8-01-1BPC-FD  
SA84-5BPC  
SA84-6BPC  
FB-04072010-RZD  
SSAJ8-02-1BPCMS  
SSAJ8-02-1BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 8 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.

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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 with the following exceptions:

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/20/10	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	30.5	SA84-5BPC	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0103408MB	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.040 pg/g 0.13 pg/g 0.71 pg/g 0.033 pg/g 0.048 pg/g 0.033 pg/g 0.13 pg/g 0.068 pg/g 0.32 pg/g	SA84-5BPC SA84-6BPC
0102261MB	4/12/10	OCDD OCDF	0.67 pg/g 0.46 pg/g	SSAJ8-02-1BPC** SSAJ8-02-1BPC-FD SSAK8-01-1BPC** SSAK8-01-1BPC-FD
0103245MB	4/13/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	2.3 pg/L 4.1 pg/L 0.90 pg/L	All water samples in SDG G0D090441

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
FB-04072010-RZD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	2.2 pg/L 8.3 pg/L 1.3 pg/L	2.2U pg/L 8.3U pg/L 1.3U pg/L

Sample FB-04072010-RZD was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:



Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.89 pg/L 1.5 pg/L 2.2 pg/L 8.3 pg/L 1.4 pg/L 1.6 pg/L 1.5 pg/L 1.6 pg/L 1.3 pg/L 1.4 pg/L 4.1 pg/L	All soil samples in SDG G0D090441

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAJ8-02-1BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	30 (40-135) 27 (40-135) 13 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAJ8-02-1BPC-FD	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK8-01-1BPC**	<sup>13</sup> C-OCDD	23 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAK8-01-1BPC-FD	<sup>13</sup> C-OCDD	25 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA84-6BPC	<sup>13</sup> C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a 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 CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
SA84-5BPC	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
SA84-6BPC	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D090441	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 G0D090441	All compounds reported 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 a 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 SSAJ8-02-1BPC\*\* and SSAJ8-02-1BPC-FD and samples SSAK8-01-1BPC\*\* and SSAK8-01-1BPC-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAJ8-02-1BPC**	SSAJ8-02-1BPC-FD				
2,3,7,8-TCDD	0.51U	0.17	-	0.34 ( $\leq 0.51$ )	-	-
1,2,3,4,7,8-HxCDD	0.40	0.28	-	0.12 ( $\leq 2.5$ )	-	-
1,2,3,6,7,8-HxCDD	1.1	0.84	-	0.26 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDD	0.53	0.52	-	0.01 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDD	3.8	2.7	-	1.1 ( $\leq 2.5$ )	-	-
OCDD	12	18	-	6 ( $\leq 5.1$ )	J (all detects)	A
2,3,7,8-TCDF	3.5	2.3	-	1.2 ( $\leq 0.51$ )	J (all detects)	A
1,2,3,7,8-PeCDF	7.9	4.0	-	3.9 ( $\leq 2.5$ )	J (all detects)	A
2,3,4,7,8-PeCDF	4.4	2.0	-	2.4 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDF	15	7.8	-	7.2 ( $\leq 2.5$ )	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAJ8-02-1BPC**	SSAJ8-02-1BPC-FD				
1,2,3,6,7,8-HxCDF	12	6.0	-	6 ( $\leq 2.5$ )	J (all detects)	A
2,3,4,6,7,8-HxCDF	3.4	1.4	-	2 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDF	2.2	1.1	-	1.1 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDF	51	26	65 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	23	8.7	-	14.3 ( $\leq 2.5$ )	J (all detects)	A
OCDF	170	75	78 ( $\leq 50$ )	-	J (all detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK8-01-1BPC**	SSAK8-01-1BPC-FD				
1,2,3,6,7,8-HxCDD	0.76	0.71	-	0.05 ( $\leq 3.0$ )	-	-
1,2,3,7,8,9-HxCDD	0.50	3.0U	-	2.5 ( $\leq 3.0$ )	-	-
1,2,3,4,6,7,8-HpCDD	2.0	2.2	-	0.2 ( $\leq 3.0$ )	-	-
OCDD	7.2	13	-	5.8 ( $\leq 5.9$ )	-	-
2,3,7,8-TCDF	1.9	1.8	-	0.1 ( $\leq 0.59$ )	-	-
1,2,3,7,8-PeCDF	3.8	3.9	-	0.1 ( $\leq 3.0$ )	-	-
2,3,4,7,8-PeCDF	2.0	1.9	-	0.1 ( $\leq 3.0$ )	-	-
1,2,3,4,7,8-HxCDF	7.8	5.0	-	2.8 ( $\leq 3.0$ )	-	-
1,2,3,6,7,8-HxCDF	5.3	5.5	-	0.2 ( $\leq 3.0$ )	-	-
2,3,4,6,7,8-HxCDF	1.7	0.90	-	0.8 ( $\leq 3.0$ )	-	-
1,2,3,7,8,9-HxCDF	0.81	1.1	-	0.29 ( $\leq 3.0$ )	-	-
1,2,3,4,6,7,8-HpCDF	21	18	15 ( $\leq 50$ )	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK8-01-1BPC**	SSAK8-01-1BPC-FD				
1,2,3,4,7,8,9-HpCDF	8.3	6.5	-	1.8 ( $\leq 3.0$ )	-	-
OCDF	75	44	52 ( $\leq 50$ )	-	J (all detects)	A

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D090441**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D090441	SA84-5BPC	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P	Routine calibration (%D) (c)
G0D090441	SSAJ8-02-1BPC**	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D090441	SSAJ8-02-1BPC-FD SSAK8-01-1BPC** SSAK8-01-1BPC-FD SA84-6BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D090441	SA84-5BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D090441	SA84-6BPC	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D090441	SSAJ8-02-1BPC** SSAJ8-02-1BPC-FD SSAK8-01-1BPC** SSAK8-01-1BPC-FD SA84-5BPC SA84-6BPC FB-04072010-RZD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D090441	SSAJ8-02-1BPC** SSAJ8-02-1BPC-FD SSAK8-01-1BPC** SSAK8-01-1BPC-FD SA84-5BPC SA84-6BPC FB-04072010-RZD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D090441	SSAJ8-02-1BPC** SSAJ8-02-1BPC-FD	OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)
G0D090441	SSAJ8-02-1BPC** SSAJ8-02-1BPC-FD	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	A	Field duplicates (RPD) (fd)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D090441	SSAK8-01-1BPC** SSAK8-01-1BPC-FD	OCDF	J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D090441**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D090441	FB-04072010-RZD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	2.2U pg/L 8.3U pg/L 1.3U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D090441**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/A

LDC #: 23125A21  
 SDG #: G0D090441  
 Laboratory: Test America

Date: 5/8/10  
 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: 4/7/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	SW	
IV.	Routine calibration/100	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS/D
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D=1+2, 3+4
XV.	Field blanks	SW	FB=7.

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: \*\*lene IV

1	SSAJ8-02-1BPC ** S	11		21	0102261MB	31
2	SSAJ8-02-1BPC-FD	12		22	0103408MB	32
3	SSAK8-01-1BPC **	13		23	0103245MB	33
4	SSAK8-01-1BPC-FD	14		24		34
5	SA84-5BPC	15		25		35
6	SA84-6BPC	16		26		36
7	FB-04072010-RZD W	17		27		37
8	SSAJ8-02-1BPCMS S	18		28		38
9	SSAJ8-02-1BPCMSD V	19		29		39
10		20		30		40

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



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

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

VALIDATION FINDINGS CHECKLIST

VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?		/		
IX. Internal standards				
Were internal standard recoveries within the 40-135% criteria?		/		
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	/			
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	/			
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	/			
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	/			
Did compound spectra contain all characteristic ions listed in the table attached?	/			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	/			
Was the signal to noise ratio for each target compound and labeled standard $> 2.5$ ?	/			
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	/			
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDPE channel?			/	
Was an acceptable lock mass recorded and monitored?	/			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XII. System performance				
System performance was found to be acceptable.	/			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.	/			
Target compounds were detected in the field duplicates.	/			
XV. Field blanks				
Field blanks were identified in this SDG.	/			
Target compounds were detected in the field blanks.	/			

**VALIDATION FINDINGS WORKSHEET**  
**Routine Calibration**

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

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

N N/A Was a routine calibration performed at the beginning and end of each 12 hour period?

Y N/A Were all percent differences (%D) of RRFs  $\leq$  20% for unlabeled compounds and  $\leq$  30% for labeled?

Y N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq$ 30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications (c)	PCDDs		PCDFs	
								Selected ions (m/z)	Ion Abundance Ratio	Selected ions (m/z)	Ion Abundance Ratio
4/20/10		19AP104D5.4U	13C-K	30.5		5	1+ dots / P (K-N)	M/M+2	0.65-0.89	M/M+2	0.65-0.89
								M+2/M+4	1.32-1.78	M+2/M+4	1.32-1.78
								M+2/M+4	1.05-1.43	M+2/M+4	1.05-1.43
								M/M+2	0.43-0.59	M/M+2	0.43-0.59
								M/M+2	0.37-0.51	M/M+2	0.37-0.51
								M+2/M+4	0.88-1.20	M+2/M+4	0.88-1.20
								M+2/M+4	0.76-1.02	M+2/M+4	0.76-1.02

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

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

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

Were all samples associated with a method blank?  
Was a method blank performed for each matrix and whenever a sample extraction was performed?  
Was the method blank contaminated? If yes, please see qualification below.

Blank extraction date: 4/13/10 Blank analysis date: 4/20/10

Conc. units: pg/g Associated samples: 5-6 (5x)

Compound		Blank ID		Sample Identification								
5X	5 (10X)	6										
0.040	0.13	0.71	0.033	0.048	0.165	0.24	0.165					
0.65	3.55	0.165	0.033	0.048	0.165	0.24	0.165					
0.34	1.6											

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

**VALIDATION FINDINGS WORKSHEET**  
**Blanks**

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

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

- N N/A Were all samples associated with a method blank?
- N N/A Was a method blank analyzed for each matrix?
- N N/A Was the blank contaminated? If yes, please see qualification below.

Blank extraction date: 4/13/10 Blank analysis date: 4/24/10

Conc. units: PS/g Associated Samples: r-f (bl.)

Compound	Blank ID	Sample Identification				
	<u>010326MB</u>	1	2	3	4	
<u>F</u>	<u>0.67</u>	<u>(13)</u>	<u>(18)</u>	<u>(7.2)</u>	<u>(13)</u>	
<u>D</u>	<u>0.46</u>	<u>(170)</u>	<u>(75)</u>	<u>(75)</u>	<u>(44)</u>	

Blank extraction date: 4/13/10 Blank analysis date: 4/24/10

Conc. units: PS/L Associated Samples: Ad Soils All H2O = (bl.)

Compound	Blank ID	Sample Identification				
	<u>0103245MB</u>	1	2	3	4	
<u>F</u>	<u>2.3</u>				<u>7</u>	
<u>G</u>	<u>4.1</u>				<u>2.3/4</u>	
<u>O</u>	<u>0.90</u>				<u>8.3/4</u>	
					<u>1.3/4</u>	

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

### VALIDATION FINDINGS WORKSHEET Field Blanks

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

Q N N/A Were field blanks identified in this SDG?  
Blank units: pg/L Associated sample units: pg/g  
Sampling date: 4/7/10  
Field blank type: (circle one) Field Blank Rinsate / Other: ( > 5x )

Associated Samples: All Soils ( > 5x )

Compound	Blank ID	5X	Sample Identification
	FB-04072010-RZD	5X	
C	0.89	0.00445	
E	1.5	0.0075	
F	2.2	0.011	
G	8.3	0.0415	
K	1.4	0.007	
L	1.6	0.008	
M	1.5	0.0075	
N	1.6	0.008	
O	1.3	0.0065	
P	1.4	0.007	
Q	4.1	0.0205	
CRQL			

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





**VALIDATION FINDINGS WORKSHEET**  
**Internal Standards**

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	G	30	✓ Y/N ✓ (F-E, 0-8)
			H	27	
			I	13	
		2	J	31	(F, 8)
		3	K	23	
		4	L	25	
		6	M	38	
		5(SMS)	N	24	No Qual
		9(MSD)	O	35	
			P	36	
			Q	29	
			R	36	
			S	20	
			T	19	
			U	11	

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

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		5	H.O. &	5	Ident/P (e)
		6	H. &	6	✓
		ml	ZMPC results (Q flag)	ml	✓K (K)

Comments: See sample calculation verification worksheet for recalculations

---



---

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

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

Y N NA  
Y N NA

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

Compound	Concentration (pg/g)		(<=50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	2				
A	0.51U	0.17		0.34	(<=0.51)	
C	0.40	0.28		0.12	(<=2.5)	
D	1.1	0.84		0.26	(<=2.5)	
E	0.53	0.52		0.01	(<=2.5)	
F	3.8	2.7		1.1	(<=2.5)	
G	12	18		6	(<=5.1)	dots/A (fd)
H	3.5	2.3		1.2	(<=0.51)	↓
I	7.9	4.0		3.9	(<=2.5)	
J	4.4	2.0		2.4	(<=2.5)	
K	15	7.8		7.2	(<=2.5)	dots/A (fd)
L	12	6.0		6	(<=2.5)	↓
M	3.4	1.4		2	(<=2.5)	
N	2.2	1.1		1.1	(<=2.5)	
O	51	26	65			dots/A (fd)
P	23	8.7		14.3	(<=2.5)	↓
Q	170	75	78			↓

Compound	Concentration (pg/g)		(<=50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	4				
D	0.76	0.71		0.05	(<=3.0)	
E	0.50	3.0U		2.5	(<=3.0)	
F	2.0	2.2		0.2	(<=3.0)	
G	7.2	13		5.8	(<=5.9)	
H	1.9	1.8		0.1	(<=0.59)	
I	3.8	3.9		0.1	(<=3.0)	
J	2.0	1.9		0.1	(<=3.0)	
K	7.8	5.0		2.8	(<=3.0)	

LDC#: 23125A21

SDG#: See Cover

**VALIDATION FINDINGS WORKSHEET**

**Field Duplicates**

Page: 2 of 2

Reviewer: *[Signature]*

2nd Reviewer: *[Signature]*

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

Y  N  NA

Were field duplicate pairs identified in this SDG?

Y  N  NA

Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		(≤50)	(pg/g)	(pg/g)	Qualifications (Parent Only)
	3	4	RPD	Difference	Limits	
L	5.3	5.5		0.2	(≤3.0)	
M	1.7	0.90		0.8	(≤3.0)	
N	0.81	1.1		0.29	(≤3.0)	
O	21	18	15			
P	8.3	6.5		1.8	(≤3.0)	
Q	75	44	52			<i>data/A (fd)</i>

V:\FIELD DUPLICATES\23125A21.wpd

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	RRF (CS3std)	Average RRF (Initial)	RRF (CS3std)	%RSD	%RSD	RRF (CS3 std)	%RSD
1	1CAR (105)	1/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.87	0.860	0.87	10.4	10.4	0.87	10.6
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.934	0.95	0.934	0.95	10.9	10.9	0.95	12.8
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.058	1.09	1.058	1.09	11.2	11.2	1.09	11.0
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.998	1.05	0.998	1.05	10.2	10.2	1.05	12.2
			OCDF ( <sup>13</sup> C-OCDF)	1.437	1.52	1.437	1.52	14.1	14.1	1.52	14.0
2	1CAR	4/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.10	1.088	1.10	10.9	10.9	1.10	1.20
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

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

LDC #: 23/25A2/  
 SDG #: 26AP10DS

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

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

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

% Difference =  $100 * (\text{ave. RRF} \cdot \text{RRF}) / \text{ave. RRF}$       Where:      ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	24AP10DS	4/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.83	3.9	0.83	4.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.934	0.91	2.2	0.91	2.2
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.058	1.00	5.2	1.00	5.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.998	1.11	11.0	1.11	11.0
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.48	3.1	1.48	3.1
2	26AP10DS	4/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	0.99	8.7	0.99	8.6
			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)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					

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

**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates Results Verification**

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

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

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

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

MS/MSD samples: 8/9

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	20.1	19.8	ND	21.3	21.9	106	106	111	111	8.9	2.8
1,2,3,7,8-PeCDD	101	99.0	↓	107	113	106	106	114	114	5.7	6.3
1,2,3,4,7,8-HxCDD	✓	✓	0.40	93.1	90.6	92	92	91	91	2.1	2.7
1,2,3,4,7,8,9-HpCDF	✓	✓	23	154	180	130	130	158	158	15	16
OCDF	201	198	170	375	436	103	102	136	134	15	15

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

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

**METHOD:** GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

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

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0102261

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		LCS/LCSD		RPD
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	
2,3,7,8-TCDD	20.0	NA	20.2	NA	101	101									
1,2,3,7,8-PeCDD	100	/	106	/	106	106									
1,2,3,4,7,8-HxCDD	✓	/	91.9	/	92	92									
1,2,3,4,7,8,9-HpCDF	✓	/	91.7	/	92	92									
OCDF	200	✓	193	✓	97	97									

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



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>3</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF			
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF		M+4	409.7788	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		M	419.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		M+2	419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</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		M+2	423.7767	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDD		M+4	425.7737	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		M+2	435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDD (S)		M+4	437.8140	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>10</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		M+4	479.7165	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		LOCK	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8587	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	OCDF	
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		M+4	443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		M+2	457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	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)	M+4	459.7348		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDD	M+2	469.7780		M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	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	M+4	471.7750		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
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> Cl <sub>2</sub> O	PeCDD (S)	M+4	513.6775		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>9</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>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	LOCK	[422.9278]		LOCK	C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDFE								
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK								
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF							
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>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+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD								
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD								
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)								
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> 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>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE								
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK								

(e) The following nucleidic masses were used:

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

S = internal/recovery standard

LDC #: 23125A2  
 SDG #: See cover

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification

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

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

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

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

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

Example:

Sample I.D. 1, 2:

Conc. =  $\frac{(10456220)(4000)}{(1758975)(1.437)(10.36)(0.95)}$   
 = 167.4 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 8, 2010  
**LDC Report Date:** May 25, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D100462

**Sample Identification**

SSAK2-01-1BPC  
SSAK3-02-1BPC  
SSAK3-03-1BPC\*\*  
SSAK3-04-1BPC\*\*  
SSAK2-01-1BPC\_FD  
SSAK3-04-1BPC\_FD  
SSAK3-03-1BPCMS  
SSAK3-03-1BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 8 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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 with the following exceptions:

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/20/10	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	30.5	SSAK2-01-1BPC	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0103408MB	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.040 pg/g 0.13 pg/g 0.71 pg/g 0.033 pg/g 0.048 pg/g 0.033 pg/g 0.13 pg/g 0.068 pg/g 0.32 pg/g	SSAK2-01-1BPC SSAK3-02-1BPC SSAK3-03-1BPC** SSAK3-04-1BPC**
0112236MB	4/22/10	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.85 pg/g 0.64 pg/g 1.3 pg/g	SSAK2-01-1BPC_FD SSAK3-04-1BPC_FD

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
SSAK2-01-1BPC	1,2,3,7,8,9-HxCDD	0.19 pg/g	0.19U pg/g
SSAK3-02-1BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.12 pg/g 0.37 pg/g 1.4 pg/g	0.12U pg/g 0.37U pg/g 1.4U pg/g
SSAK3-03-1BPC**	OCDD	1.6 pg/g	1.6U pg/g
SSAK2-01-1BPC_FD	1,2,3,4,6,7,8-HpCDF	0.90 pg/g	0.90U pg/g

Sample FB-04072010-RZD (from SDG G0D090441) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.89 pg/L 1.5 pg/L 2.2 pg/L 8.3 pg/L 1.4 pg/L 1.6 pg/L 1.5 pg/L 1.6 pg/L 1.3 pg/L 1.4 pg/L 4.1 pg/L	All samples in SDG G0D100462

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK2-01-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	30 (40-135) 34 (40-135) 14 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P



Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK3-04-1BPC**	<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
SSAK2-01-1BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	28 (40-135) 26 (40-135) 9.6 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAK3-04-1BPC_FD	<sup>13</sup> C-OCDD	31 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a 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 CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
SSAK3-04-1BPC**	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
SSAK3-04-1BPC_FD	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 GOD100462	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 G0D100462	All compounds reported 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 a 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 SSAK2-01-1BPC and SSAK2-01-1BPC\_FD and samples SSAK3-04-1BPC\*\* and SSAK3-04-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK2-01-1BPC	SSAK2-01-1BPC_FD				
2,3,7,8-TCDD	0.53U	0.081	-	0.449 ( $\leq 0.53$ )	-	-
1,2,3,4,7,8-HxCDD	0.081	2.5U	-	2.419 ( $\leq 2.5$ )	-	-
1,2,3,6,7,8-HxCDD	0.17	2.5U	-	2.33 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDD	0.19	2.5U	-	2.31 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDD	2	2.5U	-	0.5 ( $\leq 2.5$ )	-	-
OCDD	49	5.1U	-	43.9 ( $\leq 5.1$ )	J (all detects) UJ (all non-detects)	A
2,3,7,8-TCDF	0.18	0.2	-	0.02 ( $\leq 0.53$ )	-	-
1,2,3,7,8-PeCDF	0.18	0.2	-	0.02 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDF	0.43	2.5U	-	2.07 ( $\leq 2.5$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK2-01-1BPC	SSAK2-01-1BPC_FD				
1,2,3,6,7,8-HxCDF	0.26	2.5U	-	2.24 ( $\leq 2.5$ )	-	-
2,3,4,6,7,8-HxCDF	0.2	2.5U	-	2.3 ( $\leq 2.5$ )	-	-
1,2,3,7,8,9-HxCDF	0.11	2.5U	-	2.39 ( $\leq 2.5$ )	-	-
1,2,3,4,6,7,8-HpCDF	2.4	0.9	-	1.5 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8,9-HpCDF	0.9	2.5U	-	1.6 ( $\leq 2.5$ )	-	-
OCDF	17	5.1U	-	11.9 ( $\leq 5.1$ )	J (all detects) UJ (all non-detects)	A

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK3-04-1BPC**	SSAK3-04-1BPC_FD				
2,3,7,8-TCDD	6.7	4.4	-	2.3 ( $\leq 2.7$ )	-	-
1,2,3,7,8-PeCDD	25	16	44 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8-HxCDD	19	11	-	8 ( $\leq 2.7$ )	J (all detects)	A
1,2,3,6,7,8-HxCDD	41	26	45 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDD	37	24	43 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDD	140	92	41 ( $\leq 50$ )	-	-	-
OCDD	170	140	19 ( $\leq 50$ )	-	-	-
2,3,7,8-TCDF	160	110	37 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDF	320	200	46 ( $\leq 50$ )	-	-	-
2,3,4,7,8-PeCDF	160	98	48 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8-HxCDF	610	420	37 ( $\leq 50$ )	-	-	-
1,2,3,6,7,8-HxCDF	310	200	43 ( $\leq 50$ )	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK3-04-1BPC**	SSAK3-04-1BPC_FD				
2,3,4,6,7,8-HxCDF	78	52	40 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDF	68	42	47 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDF	1300	840	43 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	630	400	45 ( $\leq 50$ )	-	-	-
OCDF	4700	3000	44 ( $\leq 50$ )	-	-	-

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D100462**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D100462	SSAK2-01-1BPC	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	P	Routine calibration (%D) (c)
G0D100462	SSAK2-01-1BPC SSAK2-01-1BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D100462	SSAK3-04-1BPC** SSAK3-04-1BPC_FD	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D100462	SSAK3-04-1BPC**	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D100462	SSAK3-04-1BPC_FD	OCDF	J (all detects)	P	Compound quantitation and CRQLs (e)
G0D100462	SSAK2-01-1BPC SSAK3-02-1BPC SSAK3-03-1BPC** SSAK3-04-1BPC** SSAK2-01-1BPC_FD SSAK3-04-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D100462	SSAK2-01-1BPC SSAK3-02-1BPC SSAK3-03-1BPC** SSAK3-04-1BPC** SSAK2-01-1BPC_FD SSAK3-04-1BPC_FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D100462	SSAK2-01-1BPC SSAK2-01-1BPC_FD	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Field duplicates (Difference) (fd)
G0D100462	SSAK3-04-1BPC** SSAK3-04-1BPC_FD	1,2,3,4,7,8-HxCDD	J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D100462**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Modified Final Concentration</b>	<b>A or P</b>	<b>Code</b>
G0D100462	SSAK2-01-1BPC	1,2,3,7,8,9-HxCDD	0.19U pg/g	A	bl
G0D100462	SSAK3-02-1BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.12U pg/g 0.37U pg/g 1.4U pg/g	A	bl
G0D100462	SSAK3-03-1BPC**	OCDD	1.6U pg/g	A	bl
G0D100462	SSAK2-01-1BPC_FD	1,2,3,4,6,7,8-HpCDF	0.90U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D100462**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 23125B21  
 SDG #: G0D100462  
 Laboratory: Test America

Date: 5/18/10  
 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: 4/8/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/TV	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 1 + 5. <del>4 + 6</del> 4 + 6
XV.	Field blanks	SW	FB-040T=010-R2D (G0D090441)

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: \*\* 1-eul/v  
 SW soils

1	SSAK2-01-1BPC	11	01034.8MB	21		31	
2	SSAK3-02-1BPC	12	0112236MB	22		32	
3	SSAK3-03-1BPC **	13		23		33	
4	SSAK3-04-1BPC **	14		24		34	
5	SSAK2-01-1BPC_FD	15		25		35	
6	SSAK3-04-1BPC_FD	16		26		36	
7	SSAK3-03-1BPCMS	17		27		37	
8	SSAK3-03-1BPCMSD	18		28		38	
9		19		29		39	
10		20		30		40	

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

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $< 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	<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 checked="" 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"/>	



<b>VIII. Regional Quality Assurance and Quality Control</b>			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>IX. Internal standards</b>			
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the minimum S/N ratio of all internal standard peaks > 10?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>X. Target compound identification</b>			
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
For PCDF identification, was any signal (S/N $\geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDF channel?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XI. Compound quantitation/CRQLs</b>			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XII. System performance</b>			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIII. Overall assessment of data</b>			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XIV. Field duplicates</b>			
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>XV. Field blanks</b>			
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

**VALIDATION FINDINGS WORKSHEET**  
Routine Calibration

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

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

N N/A Was a routine calibration was performed at the beginning and end of each 12 hour period?

Y(N) N/A Were all percent differences (%D) of RRFs  $\leq$  20% for unlabeled compounds and  $\leq$  30% for labeled?

Y(N) N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq$ 30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications (C)
1	4/20/10	19AP104D5 to	BC-K	30.5		1	+ dets / P (K-N)
	PCDDs		Selected Ions (m/z)		Ion Abundance Ratio		
	Tetra-		M/M+2				0.65-0.89
	Penta-		M+2/M+4				1.32-1.78
	Hexa-		M+2/M+4				1.05-1.43
	Hexa- <sup>13</sup> C-HxCDF (IS) only		M/M+2				0.43-0.59
	Hepta- <sup>13</sup> C-HpCDF (IS) only		M/M+2				0.37-0.51
	Hepta-		M+2/M+4				0.88-1.20
	Octa-		M+2/M+4				0.76-1.02
	PCDFs		Selected Ions (m/z)		Ion Abundance Ratio		
	Tetra-		M/M+2				0.65-0.89
	Penta-		M+2/M+4				1.32-1.78
	Hexa-		M+2/M+4				1.05-1.43
	Hexa- <sup>13</sup> C-HxCDF (IS) only		M/M+2				0.43-0.59
	Hepta- <sup>13</sup> C-HpCDF (IS) only		M/M+2				0.37-0.51
	Hepta-		M+2/M+4				0.88-1.20
	Octa-		M+2/M+4				0.76-1.02

# VALIDATION FINDINGS WORKSHEET

## Blanks

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

- Y  N  N/A  Were all samples associated with a method blank?
- Y  N  N/A  Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y  N  N/A  Was the method blank contaminated? if yes, please see qualification below.

Blank extraction date: 4/13/10      Blank analysis date: 4/20/10  
 Conc. units: pg/g      Associated samples: 1-4 (bld)

Compound	Blank ID	Sample Identification				
		5X	1	2	3	
E	0.040	0.2	0.19/U	0.12/U	-	
F	0.13	0.65	-	0.37/U	-	
G	0.71	3.55	-	1.4/U	1.6/U	
H	0.033	0.165	-	-	-	
K	0.048	0.24	-	-	-	
L	0.033	0.165	-	-	-	
O	0.13	0.65	-	-	-	
P	0.068	0.34	-	-	-	
Q	0.32	1.6	-	-	-	

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

### VALIDATION FINDINGS WORKSHEET Blanks

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

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

- N N/A Were all samples associated with a method blank?  
 Y N N/A Was a method blank analyzed for each matrix?  
 Y N N/A Was the blank contaminated? If yes, please see qualification below.

Blank extraction date: 4/27/10 Blank analysis date: 4/27/10

Conc. units:  $\mu\text{g/g}$  Associated Samples: 5-6 (66)

Compound	Blank ID	Sample Identification
	01236MB	
	0.85	5 6
$\mu\text{g}$	0.64	140
0	1.3	0.90 11 (840)
0		3000

Blank extraction date: \_\_\_\_\_ Blank analysis date: \_\_\_\_\_  
Conc. units: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification

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

LDC #: 23125B21  
 SDG #: See Cover

### VALIDATION FINDINGS WORKSHEET

#### Field Blanks

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

**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: 4/7/10

Field blank type: (circle one) Field Blank / Rinsate / Other: All ( > 5 x ) Associated Samples:

Compound	Blank ID	Blank ID	Sample Identification
		EB-04072010-RZD	5X
C	0.89		0.00445
E	1.5		0.0075
F	2.2		0.011
G	8.3		0.0415
K	1.4		0.007
L	1.6		0.008
M	1.5		0.0075
N	1.6		0.008
O	1.3		0.0065
P	1.4		0.007
Q	4.1		0.0205
CRQL			

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

VALIDATION FINDINGS WORKSHEET  
Matrix Spike/Matrix Spike Duplicates

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

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

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

X N N/A Was a MS/MSD analyzed every 20 samples of each matrix?  
Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		T/B	P	147 (79-139)	( )	( )	3	No Qual
			R	162 (75-141)	160 (75-141)	( )		(MSD or CSid)
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 2312521  
 SDG #: 230000

**VALIDATION FINDINGS WORKSHEET**  
**Internal Standards**

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

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	F	30 (40-135)	<del>Y</del> <del>N</del> P (F-E, 0-8)
			H	34	
			I	14	
		4	J	32	(F, 8)
		5	F	28	
			H	26	
			I	9.6	
		6	J	31	
		7(N/S)	J	22	No Qual

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



LDC #: 03125B21  
 SDG #: Per copy

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

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

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

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

- 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).
- N N/A**

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		4	0.2 > calib range		Notes P(e)
		6	0.2 >		↓
		MM	EMPC results (flag)		NR (K)

Comments: See sample calculation verification worksheet for recalculations

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

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

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

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	5				
A	0.53U	0.081		0.449	(≤0.53)	
C	0.081	2.5U		2.419	(≤2.5)	
D	0.17	2.5U		2.33	(≤2.5)	
E	0.19	2.5U		2.31	(≤2.5)	
F	2.0	2.5U		0.5	(≤2.5)	
G	49	5.1U		43.9	(≤5.1)	↓/U↓/A (fd)
H	0.18	0.20		0.02	(≤0.53)	
I	0.18	0.20		0.02	(≤2.5)	
K	0.43	2.5U		2.07	(≤2.5)	
L	0.26	2.5U		2.24	(≤2.5)	
M	0.20	2.5U		2.3	(≤2.5)	
N	0.11	2.5U		2.39	(≤2.5)	
O	2.4	0.90		1.5	(≤2.5)	
P	0.90	2.5U		1.6	(≤2.5)	
Q	17	5.1U		11.9	(≤5.1)	↓/U↓/A (fd)

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	4	6				
A	6.7	4.4		2.3	(≤2.7)	
B	25	16	44			
C	19	11		8	(≤2.7)	↓/U↓/A (fd)
D	41	26	45			
E	37	24	43			
F	140	92	41			
G	170	140	19			
H	160	110	37			
I	320	200	46			

LDC#: 23125B21

SDG#: See Cover

### VALIDATION FINDINGS WORKSHEET

#### Field Duplicates

Page: 2 of 2

Reviewer: [Signature]

2nd Reviewer: [Signature]

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

Y N NA  
Y N NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		(≤50)	(pg/g)	(pg/g)	Qualifications (Parent Only)
	4	6	RPD	Difference	Limits	
J	160	98	48			
K	610	420	37			
L	310	200	43			
M	78	52	40			
N	68	42	47			
O	1300	840	43			
P	630	400	45			
Q	4700	3000	44			

V:\FIELD DUPLICATES\23125B21.wpd

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	RRF (Initial)	Average RRF (Initial)	RRF (CS3 std)	Average RRF (Initial)	RRF (CS3 std)	%RSD	%RSD
1	IC14 (ADS)	4/12/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.98	0.945	0.98	4.44	4.33		
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.04	1.021	1.04	3.03	2.97		
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.19	1.114	1.19	5.33	5.25		
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.11	1.072	1.11	3.60	3.75		
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.51	1.445	1.51	5.85	5.89		
2	IC12	3/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.000	1.00	1.000	1.00	1.36	1.58		
			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)								
3	IC12 (SD2)	4/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.10	1.088	1.10	1.29	1.20		
			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 SW 846 Method 8290)

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	19AP1045 =20	4/19/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	1.00	6.1	1.00	6.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.02	0.2	1.02	0.2
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.19	6.8	1.19	6.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.07	0.1	1.07	0.1
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.51	4.8	1.51	4.8
2	20AP10572	4/20/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.000	1.05	4.8	1.05	4.8
			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)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.080	1.13	3.4	1.13	3.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					

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

LDC #: 2375B2  
 SDG #: 200004

**VALIDATION FINDINGS WORKSHEET**  
**Matrix Spike/Matrix Spike Duplicates Results Verification**

Page: 1 of 1  
 Reviewer: 4  
 2nd Reviewer: [Signature]

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

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

% Recovery =  $100 * (SSR - SR) / SA$

Where: SSR = Spiked sample result, SR = Sample result  
 SA = Spike added

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

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

MS/MSD samples: 7/8

Compound	Spike Added (PS/g)		Sample Concentration (PS/g)	Spiked Sample Concentration (PS/g)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	20.6	20.1	ND	22.0	21.7	107	107	108	108	1.6	1.4
1,2,3,7,8-PeCDD	10.3	10.1	V	10.8	10.5	105	105	105	104	3.0	2.8
1,2,3,4,7,8-HxCDD	V	V	0.11	12.0	11.2	116	116	111	111	6.6	6.9
1,2,3,4,7,8,9-HpCDF	V	V	0.79	15.2	12.8	147	147	126	126	17	17
OCDF	206	201	7.2	346	330	164	164	160	160	4.7	4.7

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

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

**METHOD:** GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

% Recovery =  $100 * \frac{SSC}{SA}$

Where: SSC = Spiked sample concentration  
SA = Spike added

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0103408

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	200	NA	220	NA	110	110						
1,2,3,7,8-PeCDD	100		108		108	108						
1,2,3,4,7,8-HxCDD			116		116	116						
1,2,3,4,7,8,9-HpCDF			118		118	118						
OCDF	200		225		113	113						

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

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

(a) The following nuclidic masses were used:

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

S = internal/recovery standard



LDC #: 23/25B>1  
SDG #: See notes

**VALIDATION FINDINGS WORKSHEET**  
Sample Calculation Verification

Page: 1 of 1  
Reviewer: A  
2nd reviewer: V

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

Y N N/A  
Y N N/A

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

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).
- RRF = Relative Response Factor (average) from the initial calibration
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. 3 F:

$$\text{Conc.} = \frac{(470718)(2000)}{(11653630)(1.072)(10.01)(0.965)}$$

= 0.780 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification

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

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

**Collection Date:** April 8, 2010

**LDC Report Date:** May 25, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D100464

**Sample Identification**

RSAI2-12BPC

RSAI2-12BPCMS

RSAI2-12BPCMSD

## Introduction

This data review covers 3 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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% .

## **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.

Sample FB-04072010-RZD (both from SDG G0D090441) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.89 pg/L 1.5 pg/L 2.2 pg/L 8.3 pg/L 1.4 pg/L 1.6 pg/L 1.5 pg/L 1.6 pg/L 1.3 pg/L 1.4 pg/L 4.1 pg/L	All samples in SDG GOD100464

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within the QC limits. Since the samples were diluted out, no data were qualified.

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
RSAI2-12BPC	<sup>13</sup> C-OCDD	24 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
RSAI2-12BPC	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D100464	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 G0D100464	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, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D100464**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D100464	RSAI2-12BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D100464	RSAI2-12BPC	2,3,7,8-TCDF	J (all detects)	P	Compound quantitation and CRQLs (e)
G0D100464	RSAI2-12BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D100464	RSAI2-12BPC	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D100464**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D100464**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23125C21

SDG #: G0D100464

Laboratory: Test America

Stage 2B

Date: 5/18/10

Page: 6 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: 4/8/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ISV	A	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	W	
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	W	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB-040T2010-R2D(90D090441)

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:

1	RSAI2-12BPC	5	11	0105361 MB	21	31
2	RSAI2-12BPCMS	1	12		22	32
3	RSAI2-12BPCMSD	1	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:

# VALIDATION FINDINGS WORKSHEET Field Blanks

LDC #: 23125C21  
SDG #: See Cover

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

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

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

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

Sampling date: 4/7/10

Field blank type: (circle one) Field Blank / Rinsate / Other: All ( 25x ) Associated Samples:

Compound	Blank ID	Sample Identification
	FB-04072010-RZD	5X
C	0.89	0.00445
E	1.5	0.0075
F	2.2	0.011
G	8.3	0.0415
K	1.4	0.007
L	1.6	0.008
M	1.5	0.0075
N	1.6	0.008
O	1.3	0.0065
P	1.4	0.007
Q	4.1	0.0205
CRQL		

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



# VALIDATION FINDINGS WORKSHEET

## Internal Standards

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	F	24	(40-135) ✓
		2 (Ms)	F H	22 32	No Qual
		3 (MSD)	F H I	15 22 26 9.8	✓
<b>Internal Standards</b>					
A.	<sup>13</sup> C-2,3,7,8-TCDF		Check Standard Used	Internal Standards	Check Standard Used
B.	<sup>13</sup> C-2,3,7,8-TCDD			<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	
C.	<sup>13</sup> C-1,2,3,7,8-PeCDF			<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	
D.	<sup>13</sup> C-1,2,3,7,8-PeCDD			<sup>13</sup> C-OCDD	
E.	<sup>13</sup> C-1,2,3,4,7,8-HxCDF			<sup>13</sup> C-1,2,3,4-TCDD	
F.	<sup>13</sup> C-1,2,3,6,7,8-HxCDD			<sup>13</sup> C-1,2,3,7,8,9-HxCDD	

LDC #: 23125021  
 SDG #: see convey

VALIDATION FINDINGS WORKSHEET  
Compound Quantitation and Reported CRQLs

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

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

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

Y N N/A  
 X N N/A

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
	1		H > calc b raise	1	[Signature]
	aw		ZMPE result (C & Flag)		[Signature]

Comments: See sample calculation verification worksheet for recalculations

---



---



---

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 6, 2010  
**LDC Report Date:** May 25, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D120488

**Sample Identification**

FB04062010-RZB

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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% .

## 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 with the following exceptions:

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/22/10	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	30.1	All samples in SDG G0D120488	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0105215MB	4/15/10	1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.72 pg/L 0.74 pg/L 0.89 pg/L 1.3 pg/L 2.2 pg/L 6.2 pg/L 2.1 pg/L 0.91 pg/L 1.4 pg/L 0.85 pg/L 1.2 pg/L 1.3 pg/L 1.4 pg/L 1.7 pg/L 3.5 pg/L	All samples in SDG G0D120488

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
FB04062010-RZB	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.68 pg/L 2.5 pg/L 6.2 pg/L 2.7 pg/L 1.4 pg/L 0.82 pg/L 0.94 pg/L 1.8 pg/L 1.2 pg/L 4.4 pg/L	0.68U pg/L 2.5U pg/L 6.2U pg/L 2.7U pg/L 1.4U pg/L 0.82U pg/L 0.94U pg/L 1.8U pg/L 1.2U pg/L 4.4U pg/L

Sample FB04062010-RZB was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB04062010-RZB	4/6/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.68 pg/L 2.5 pg/L 6.2 pg/L 2.7 pg/L 1.4 pg/L 0.82 pg/L 0.94 pg/L 1.8 pg/L 1.2 pg/L 4.4 pg/L	No associated samples in this SDG

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
All samples in SDG G0D120488	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 G0D120488	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 G0D120488	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, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D120488**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D120488	FB04062010-RZB	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P	Routine calibration (%D) (c)
G0D120488	FB04062010-RZB	2,3,7,8-TCDF	None	P	Compound quantitation and CRQLs (o)
G0D120488	FB04062010-RZB	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D120488	FB04062010-RZB	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D120488**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D120488	FB04062010-RZB	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.68U pg/L 2.5U pg/L 6.2U pg/L 2.7U pg/L 1.4U pg/L 0.82U pg/L 0.94U pg/L 1.8U pg/L 1.2U pg/L 4.4U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D120488**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23125D21

SDG #: G0D120488

Laboratory: Test America

Date: 5/18/10

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: 4/6/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB = 1

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

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

D = Duplicate  
 TB = Trip blank  
 EB = Equipment blank

Validated Samples:

1	FB04062010-RZB	N	11	0125215M13	21		31	
2			12		22		32	
3			13		23		33	
4			14		24		34	
5			15		25		35	
6			16		26		36	
7			17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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

# 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 (EPA Method 8290)

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

- Y  N  N/A Were all samples associated with a method blank?
- Y  N  N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y  N  N/A Was the method blank contaminated? If yes, please see qualification below.

Blank extraction date: 4/15/10 Blank analysis date: 4/22/10

Conc. units: pg/L Associated samples: All (bl)

Compound	Blank ID	Sample Identification																			
	0105215MB	5X									1										
B	0.72	3.6																			
C	0.74	3.7																			
D	0.89	4.45																			
E	1.3	6.5									0.68/U										
F	2.2	11									2.5/U										
G	6.2	31									6.2/U										
H	2.1	10.5									2.7/U										
J	0.91	4.55																			
K	1.4	7									1.4/U										
L	0.85	4.25									0.82/U										
M	1.2	6																			
N	1.3	6.5									0.94/U										
O	1.4	7									1.8/U										
P	1.7	8.5									1.2/U										
Q	3.5	17.5									4.4/U										

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

**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

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

Y/N N/A Were field blanks identified in this SDG? None  
 Blank units: pg/L Associated sample units: pg/g Associated Samples: AT  
 Sampling date: 4/6/10  
 Field blank type: (circle one) Field Blank/ Rinsate / Other:

Compound	Blank ID	5X	Sample Identification
	FER04062010-RZB	5X	
E	0.68	0.0034	
F	2.5	0.0125	
G	6.2	0.031	
H	2.7	0.0135	
K	1.4	0.007	
L	0.82	0.0041	
N	0.94	0.0047	
O	1.8	0.009	
P	1.2	0.006	
Q	4.4	0.022	
CRQL			

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

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		D11	EMPC results (R flag)	M	NK (K)
		1	No 2.3.7.8-TCDF confirmation		None

Comments: See sample calculation verification worksheet for recalculations

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 12, 2010  
**LDC Report Date:** May 25, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D140435

**Sample Identification**

SSAN6-02-1BPC  
SSAN6-02-2BPC  
SA156-3BPC\*\*

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 3 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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
0109260MB	4/19/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.59 pg/g 0.28 pg/g 0.14 pg/g 0.24 pg/g 0.14 pg/g 0.086 pg/g 0.30 pg/g 0.13 pg/g 0.63 pg/g	All samples in SDG G0D140435

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
SA156-3BPC**	2,3,7,8-TCDF	0.51 pg/g	0.51U pg/g

Samples EB-04122010-RIG2-RZB and EB-04122010-RIG1-RZB (both from SDG G0D140534) were identified as field blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG2-RZB	4/12/10	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	0.31 pg/L 0.35 pg/L 0.60 pg/L 1.8 pg/L 4.0 pg/L 4.8 pg/L 4.3 pg/L 2.2 pg/L 7.3 pg/L 4.6 pg/L 1.3 pg/L 0.85 pg/L 13 pg/L 5.6 pg/L 26 pg/L	SA156-3BPC**

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG1-RZB	4/12/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.57 pg/L 0.59 pg/L 0.82 pg/L 2.8 pg/L 10 pg/L 2.0 pg/L 1.2 pg/L 1.3 pg/L 1.7 pg/L 1.2 pg/L 1.1 pg/L 2.0 pg/L 0.95 pg/L 3.6 pg/L	SA156-3BPC**

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

Samples FB-04062010-RZB (from SDG G0D120488) and FB-04072010-RZC (from SDG G0D130519) were identified as field blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04062010-RZB	4/6/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.68 pg/L 2.5 pg/L 6.2 pg/L 2.7 pg/L 1.4 pg/L 0.82 pg/L 0.94 pg/L 1.8 pg/L 1.2 pg/L 4.4 pg/L	SA156-3BPC**
FB-04072010-RZC	4/8/10	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	0.77 pg/L 0.74 pg/L 0.82 pg/L 4.2 pg/L 37 pg/L 0.57 pg/L 0.96 pg/L 0.67 pg/L 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 2.1 pg/L 1.5 pg/L 6.7 pg/L	SSAN6-02-1BPC SSAN6-02-2BPC

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits. Since there were no associated samples, no data were qualified.

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-02-1BPC	<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
SSAN6-02-2BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	37 (40-135) 35 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SA156-3BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	30 (40-135) 30 (40-135) 17 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a 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 CRQLs were within validation criteria with the following exceptions:

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN6-02-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAN6-02-2BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) 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 G0D140435	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 G0D140435	All compounds reported 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 a 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 SA156-3BPC\*\* and SA156-3BPC\_FD (from SDG G0D140543) 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
	SA156-3BPC**	SA156-3BPC_FD				
2,3,7,8-TCDD	0.15	0.54U	-	0.39 ( $\leq 0.54$ )	-	-
1,2,3,7,8-PeCDD	0.56	2.7U	-	2.17 ( $\leq 2.7$ )	-	-
1,2,3,4,7,8-HxCDD	0.84	2.7U	-	1.86 ( $\leq 2.7$ )	-	-
1,2,3,6,7,8-HxCDD	0.68	2.7U	-	2.02 ( $\leq 2.7$ )	-	-
1,2,3,7,8,9-HxCDD	0.81	2.7U	-	1.89 ( $\leq 2.7$ )	-	-
1,2,3,4,6,7,8-HpCDD	1.4	2.7U	-	1.3 ( $\leq 2.7$ )	-	-
OCDD	5.6	5.4U	-	0.2 ( $\leq 5.4$ )	-	-
2,3,7,8-TCDF	0.51	0.16	-	0.35 ( $\leq 0.54$ )	-	-
1,2,3,7,8-PeCDF	1.7	2.7U	-	1 ( $\leq 2.7$ )	-	-
1,2,3,4,7,8-HxCDF	4.1	2.7U	-	1.4 ( $\leq 2.7$ )	-	-
1,2,3,6,7,8-HxCDF	3.3	2.7U	-	0.6 ( $\leq 2.7$ )	-	-
2,3,4,6,7,8-HxCDF	1.4	2.7U	-	1.3 ( $\leq 2.7$ )	-	-
1,2,3,7,8,9-HxCDF	1.7	2.7U	-	1 ( $\leq 2.7$ )	-	-
1,2,3,4,6,7,8-HpCDF	10	1.2	-	8.8 ( $\leq 2.7$ )	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	5.1	2.7U	-	2.4 ( $\leq 2.7$ )	-	-
OCDF	25	5.4	-	19.6 ( $\leq 5.4$ )	J (all detects)	A

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D140435**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D140435	SSAN6-02-1BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D140435	SSAN6-02-2BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D140435	SA156-3BPC**	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D140435	SSAN6-02-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D140435	SSAN6-02-2BPC	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D140435	SSAN6-02-1BPC SSAN6-02-2BPC SA156-3BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D140435	SSAN6-02-1BPC SSAN6-02-2BPC SA156-3BPC**	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D140435	SA156-3BPC**	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D140435**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Modified Final Concentration</b>	<b>A or P</b>	<b>Code</b>
G0D140435	SA156-3BPC**	2,3,7,8-TCDF	0.51U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D140435**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D140435**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/A

LDC #: 23125E21  
 SDG #: G0D140435  
 Laboratory: Test America

Date: 5/18/10  
 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: 4/12/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/10X	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No sp/ass'd - No Genal
VII.	Laboratory control samples	A	LCG
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SN	D = 3 + SA156-3BPC - FD (FOD140543, (FOD120488)
XV.	Field blanks	W	FB-04062010-RZB, FB-040710-RZC (FOD130519, ZB-04122010-R42-RZB, ZB-04122010-R41-RZB (FOD140534)

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

Validated Samples: \*\* | ene | 11V

1	SSAN6-02-1BPC	11	01092601B	21		31	
2	SSAN6-02-2BPC	12		22		32	
3	SA156-3BPC **	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: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_



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 $\geq 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

VALIDATION FINDINGS CHECKLIST

VIII. Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?		/	
Were the performance evaluation (PE) samples within the acceptance limits?		/	
IX. Internal standards			
Were internal standard recoveries within the 40-135% criteria?		/	
Was the minimum S/N ratio of all internal standard peaks > 10?	/		
X. Target compound identification			
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	/		
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	/		
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	/		
Did compound spectra contain all characteristic ions listed in the table attached?	/		
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	/		
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?	/		
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?	/		
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/		
XII. System performance			
System performance was found to be acceptable.	/		
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.	/		
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.	/		
Target compounds were detected in the field duplicates.		/	
XV. Field blanks			
Field blanks were identified in this SDG.	/		
Target compounds were detected in the field blanks.	/		

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

# VALIDATION FINDINGS WORKSHEET

## Blanks

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

- Y  N  N/A Were all samples associated with a method blank?
- Y  N  N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
- Y  N  N/A Was the method blank contaminated? If yes, please see qualification below.

Blank extraction date: 4/19/10 Blank analysis date: 4/25/10  
 Conc. units: pg/g Associated samples: All (66)

Compound	Blank ID	5X	3	Sample Identification
	0109260MB			
F	0.14	0.7	-	<i>all samples &gt; 5X</i>
G	0.59	2.95	-	
H	0.28	1.4	0.51/U	
I	0.14	0.7	-	
K	0.24	1.2	-	
L	0.14	0.7	-	
N	0.086	0.43	-	
O	0.30	1.5	-	
P	0.13	0.65	-	
Q	0.63	3.15	-	

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

LDC #: 2312522  
 SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

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

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

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

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

Sampling date: 4/8/10

Field blank type: (circle one) Field Blank  Rinsate / Other: 1-2

Associated Samples: 1-2

Compound	Blank ID	Sample Identification									
	FB-04072010-RZC	5X									
C	0.77	0.00385									
D	0.74	0.0037									
E	0.82	0.0041									
F	4.2	0.021									
G	37	0.185									
H	0.57	0.00285									
I	0.96	0.0048									
J	0.67	0.00335									
K	1.1	0.0055									
L	0.96	0.0048									
M	1.0	0.005									
N	1.0	0.005									
O	2.1	0.0105									
P	1.5	0.0075									
Q	6.7	0.0335									
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, "J".

## VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

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

Sampling date: 4/6/10

Field blank type: (circle one) Field Blank / Rinsate / Other: Field Blank Associated Samples: 3 (25X)

Compound	Blank ID	5X	Sample Identification	
	FB04062010-RZR	5X		
E	0.68	0.0034		
F	2.5	0.0125		
G	6.2	0.031		
H	2.7	0.0135		
K	1.4	0.007		
L	0.82	0.0041		
N	0.94	0.0047		
O	1.8	0.009		
P	1.2	0.006		
Q	4.4	0.022		
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 #: 23125E21  
 SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

## Field Blanks

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

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

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

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

Sampling date: 4/12/10

Field blank type: (circle one) Field Blank / Rinsate / Other: Field Blank Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification
	FB_04122010-RIG2-RZR	5X
C	0.31	0.00155
D	0.35	0.00175
E	0.60	0.003
F	1.8	0.009
G	4.0	0.02
H	4.8	0.024
I	4.3	0.0215
J	2.2	0.011
K	7.3	0.0365
L	4.6	0.023
M	1.3	0.0065
N	0.85	0.00425
O	13	0.065
P	5.6	0.028
Q	26	0.13
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,

**VALIDATION FINDINGS WORKSHEET**  
**Field Blanks**

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

N/A Were field blanks identified in this SDG?

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

Sampling date: 4/12/10

Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: 3 (>5X)

Compound	Blank ID	5X	Sample Identification				
	EB-04122010-RIG1-R7B	5X					
C	0.57	0.00285					
D	0.59	0.00295					
E	0.82	0.0041					
F	2.8	0.014					
G	10	0.05					
H	2.0	0.01					
I	1.2	0.006					
J	1.3	0.0065					
K	1.7	0.0085					
L	1.2	0.006					
M	1.1	0.0055					
O	2.0	0.01					
P	0.95	0.00475					
Q	3.6	0.018					
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 #: 03/05/21  
 SDG #: Becaney

VALIDATION FINDINGS WORKSHEET  
Compound Quantitation and Reported CRQLs

Page: of 9  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

Y N N/A  
Y N N/A

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		1	<i>spbs &gt; calib range</i>	1	<i>Notes/P(e)</i>
		2	<del>H.I.K.L.O.P.R</del> <del>M.F.H.I.V.K.L</del> M.N.O.P.R	2	✓
		N/A	ZMPC results (CR flags)		NK(R)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23125E21  
 SDG#: See Cover

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

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

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

Y N NA  
Y N NA

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

Compound	Concentration (pg/g)		(<=50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	SA156-3BPC_FD				
A	0.15	0.54U		0.39	( <=0.54)	
B	0.56	2.7U		2.14	( <=2.7)	
C	0.84	2.7U		1.86	( <=2.7)	
D	0.68	2.7U		2.02	( <=2.7)	
E	0.81	2.7U		1.89	( <=2.7)	
F	1.4	2.7U		1.3	( <=2.7)	
G	5.6	5.4U		0.2	( <=5.4)	
H	0.51	0.16		0.35	( <=0.54)	
I	1.7	2.7U		1	( <=2.7)	
K	4.1	2.7U		1.4	( <=2.7)	
L	3.3	2.7U		0.6	( <=2.7)	
M	1.4	2.7U		1.3	( <=2.7)	
N	1.7	2.7U		1	( <=2.7)	
O	10	1.2		8.8	( <=2.7)	ndets/A (fd)
P	5.1	2.7U		2.4	( <=2.7)	
Q	25	5.4		19.6	( <=5.4)	ndets/A (fd)

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 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,  $X$  = Mean of the RRFs  
 $A_{is}$  = Area of associated internal standard  
 $C_{is}$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	Average RRF (Initial)	RRF (CS3 std)	RRF (CS3 std)	%RSD	%RSD		
1	1CAZ (SD5)	4/12/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.945	0.98	0.98	4.44	4.44	4.33	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.021	1.04	1.04	3.03	3.03	2.97	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.114	1.19	1.19	5.33	5.33	5.25	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.072	1.11	1.11	3.60	3.60	3.75	
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.445	1.51	1.51	5.85	5.85	5.89	
2	1CAZ	3/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.000	1.000	1.00	1.00	1.36	1.36	1.58	
			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)								
3	1CAZ (SD2)	4/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.088	1.10	1.10	1.29	1.29	1.20	
			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 SW 846 Method 8290)

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	27AP10031	4/27/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.97	3.1	0.97	3.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	0.94	7.5	0.94	7.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.10	1.2	1.10	1.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.04	2.9	1.04	2.9
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.42	2.0	1.42	2.0
2	27AP10035	4/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.90	4.3	0.90	4.3
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	0.96	6.3	0.96	6.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.08	3.1	1.08	3.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.03	3.9	1.03	3.9
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.36	5.9	1.36	5.9
3	28AP10032	4/28/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.09	0.3	1.09	0.3
			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-OCDD)					

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

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

**METHOD:** GC/MS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

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

RPD =  $100 * \frac{LCS - LCSD}{LCS + LCSD}$  LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0109260

Compound	Spike Added ( <u>172/9</u> )		Spiked Sample Concentration ( <u>172/9</u> )		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	<u>20.0</u>	<u>NA</u>	<u>19.8</u>	<u>NA</u>	<u>99</u>	<u>99</u>								
1,2,3,7,8-PeCDD	<u>100</u>	<u>/</u>	<u>102</u>	<u>/</u>	<u>102</u>	<u>102</u>								
1,2,3,4,7,8-HxCDD	<u>1</u>	<u>/</u>	<u>108</u>	<u>/</u>	<u>108</u>	<u>108</u>								
1,2,3,4,7,8,9-HpCDF	<u>200</u>	<u>/</u>	<u>104</u>	<u>/</u>	<u>104</u>	<u>104</u>								
OCDF			<u>202</u>	<u>/</u>	<u>101</u>	<u>101</u>								

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

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

(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

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

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

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

- A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured
- A<sub>s</sub> = Area of the characteristic ion (EICP) for the specific internal standard
- I<sub>s</sub> = Amount of internal standard added in nanograms (ng)
- V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).
- RRF = Relative Response Factor (average) from the initial calibration
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. 3, Q:

$$\text{Conc.} = \frac{(1578946)(4000)}{(1842910)(1.445)(10.03)(0.93)}$$

$$= 24.3 \text{ pg/g}$$

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification



EDD CHECKLIST

LDC #: 23125

SDG #: G0D090441, G0D100462, G0D100464, G0D120488, G0D140435

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_LDC23125_052510.doc
<b>IV. EDD Delivery</b>				
Was the final EDD sent to the client?	X			