

**LABORATORY DATA CONSULTANTS, INC.**

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

June 15, 2010

SUBJECT: Tronox LLC Facility, 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 27, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 23265:**

**SDG #**

**Fraction**

G0D080425, G0D130519, G0D150462	Dioxins/Dibenzofurans
G0D150582, G0D150589, G0D160435	
G0D160472, G0D170485, G0D170489	
G0D170491, G0D170492, G0D200427	
G0D200558, G0D240497, G0D270515	
G0D270522, G0D270529, G0D270573	
G0D270574, G0D280571, G0D280586	
G0D300450, G0D300454	

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

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

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

Sincerely,

Erlinda T. Rauto  
Operations Manager/Senior Chemist



EDD CHECKLIST

LDC #: 23265

SDG #: G0D080425, G0D130519, G0D150462, G0D150582, G0D150589  
G0D160435, G0D160472, G0D170485, G0D170489, G0D170491  
G0D170492, G0D200427, G0D200558, G0D240497, G0D270515  
G0D270522, G0D270529, G0D270573, G0D270574, G0D270574  
G0D280571, G0D280586, G0D300450, G0D300454

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_LDC23265_061510.doc
<b>IV. EDD Delivery</b>				
Was the final EDD sent to the client?	X			

**Tronox LLC Facility, PCS, Henderson, Nevada**  
**Data Validation Reports**  
**LDC #23265**

Dioxins/Dibenzofurans

LD C

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 6, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D080425

### Sample Identification

SSA06-02-1BPC  
SSA06-02-5BPC  
RSAQ3-3BPC\*\*  
SA169-3BPC  
RSAQ3-3BPC\_FD  
SSA06-02-1BPC\_FD  
SSAJ8-01-3BPC\*\*  
SSA06-02-5BPCMS  
SSA06-02-5BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 9 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
4/22/10	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	30.1	RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD SSA06-02-5BPCMSD	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P
4/27/10	1,2,3,7,8,9-HxCDF	20.2	SSAJ8-01-3BPC**	1,2,3,7,8,9-HxCDF	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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SSAJ8-01-3BPC**
0106187MB	4/16/10	2,3,7,8-TCDD 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 1,2,3,4,6,7,8-HpCDF OCDF	0.036 pg/g 0.14 pg/g 0.45 pg/g 0.088 pg/g 0.040 pg/g 0.062 pg/g 0.054 pg/g 0.076 pg/g 0.15 pg/g	SSA06-02-1BPC SSA06-02-5BPC RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD SSA06-02-1BPC_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
SSA06-02-1BPC	2,3,7,8-TCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.082 pg/g 0.42 pg/g 1.4 pg/g	0.082U pg/g 0.42U pg/g 1.4U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
SSA06-02-5BPC	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 1,2,3,4,6,7,8-HpCDF OCDF	0.14 pg/g 0.72 pg/g 0.12 pg/g 0.12 pg/g 0.084 pg/g 0.093 pg/g 0.21 pg/g 0.49 pg/g	0.14U pg/g 0.72U pg/g 0.12U pg/g 0.12U pg/g 0.084U pg/g 0.093U pg/g 0.21U pg/g 0.49U pg/g
RSAQ3-3BPC**	2,3,7,8-TCDD 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	0.038 pg/g 0.23 pg/g 0.61 pg/g 0.24 pg/g 0.12 pg/g 0.13 pg/g 0.20 pg/g	0.038U pg/g 0.23U pg/g 0.61U pg/g 0.24U pg/g 0.12U pg/g 0.13U pg/g 0.20U pg/g
RSAQ3-3BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	0.43 pg/g 1.1 pg/g	0.43U pg/g 1.1U pg/g
SSA06-02-1BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	0.30 pg/g 0.62 pg/g	0.30U pg/g 0.62U pg/g

Samples FB-04072010-RZD (from SDG G0D090441), FB04062010-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-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	SSAJ8-01-3BPC**
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	RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZC	4/8/10	1,2,3,4,7,8-HxCDD	0.77 pg/L	SSA06-02-1BPC
		1,2,3,6,7,8-HxCDD	0.74 pg/L	SSA06-02-5BPC
		1,2,3,7,8,9-HxCDD	0.82 pg/L	SSA06-02-1BPC_FD
		1,2,3,4,6,7,8-HpCDD	4.2 pg/L	
		OCDD	37 pg/L	
		2,3,7,8-TCDF	0.57 pg/L	
		1,2,3,7,8-PeCDF	0.96 pg/L	
		2,3,4,7,8-PeCDF	0.67 pg/L	
		1,2,3,4,7,8-HxCDF	1.1 pg/L	
		1,2,3,6,7,8-HxCDF	0.96 pg/L	
		2,3,4,6,7,8-HxCDF	1.0 pg/L	
		1,2,3,7,8,9-HxCDF	1.0 pg/L	
		1,2,3,4,6,7,8-HpCDF	2.1 pg/L	
		1,2,3,4,7,8,9-HpCDF	1.5 pg/L	
		OCDF	6.7 pg/L	

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) and MS/MSD relative percent differences (RPD) were not within QC limits for several compounds, the MS or MSD 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.

#### 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
SA169-3BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSA06-02-1BPC SSA06-02-5BPC RSAQ3-3BPC** RSAQ3-3BPC_FD	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 G0D080425	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 G0D080425	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 SSA06-02-1BPC and SSA06-02-1BPC\_FD and samples RSAQ3-3BPC\*\* and RSAQ3-3BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSA06-02-1BPC	SSA06-02-1BPC_FD				
2,3,7,8-TCDD	0.082	1.0U	-	0.918 ( $\leq 1.0$ )	-	-
1,2,3,7,8-PeCDD	0.19	0.082	-	0.108 ( $\leq 5.2$ )	-	-
1,2,3,4,7,8-HxCDD	0.13	5.2U	-	5.07 ( $\leq 5.2$ )	-	-
1,2,3,6,7,8-HxCDD	0.17	0.14	-	0.03 ( $\leq 5.2$ )	-	-
1,2,3,7,8,9-HxCDD	0.19	0.11	-	0.08 ( $\leq 5.2$ )	-	-
1,2,3,4,6,7,8-HpCDD	0.42	0.30	-	0.12 ( $\leq 5.2$ )	-	-
OCDD	1.4	0.62	-	0.78 ( $\leq 10$ )	-	-
2,3,7,8-TCDF	0.91	0.76	-	0.15 ( $\leq 1.0$ )	-	-
1,2,3,7,8-PeCDF	0.62	1.0	-	0.38 ( $\leq 5.2$ )	-	-
2,3,4,7,8-PeCDF	0.39	0.47	-	0.08 ( $\leq 5.2$ )	-	-
1,2,3,4,7,8-HxCDF	0.92	1.6	-	0.68 ( $\leq 5.2$ )	-	-
1,2,3,6,7,8-HxCDF	0.49	0.85	-	0.36 ( $\leq 5.2$ )	-	-
2,3,4,6,7,8-HxCDF	0.16	0.27	-	0.11 ( $\leq 5.2$ )	-	-
1,2,3,7,8,9-HxCDF	0.19	0.16	-	0.03 ( $\leq 5.2$ )	-	-
1,2,3,4,6,7,8-HpCDF	1.2	2.0	-	0.8 ( $\leq 5.2$ )	-	-
1,2,3,4,7,8,9-HpCDF	0.83	1.1	-	0.27 ( $\leq 5.2$ )	-	-
OCDF	2.8	4.4	-	1.6 ( $\leq 10$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAQ3-3BPC**	RSAQ3-3BPC_FD				
2,3,7,8-TCDD	0.038	1.0U	-	0.962 ( $\leq 1.0$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	RSAQ3-3BPC**	RSAQ3-3BPC_FD				
1,2,3,4,7,8-HxCDD	0.066	5.2U	-	5.134 ( $\leq 5.2$ )	-	-
1,2,3,6,7,8-HxCDD	0.079	0.10	-	0.021 ( $\leq 5.2$ )	-	-
1,2,3,7,8,9-HxCDD	0.15	0.20	-	0.05 ( $\leq 5.2$ )	-	-
1,2,3,4,6,7,8-HpCDD	0.23	0.43	-	0.2 ( $\leq 5.2$ )	-	-
OCDD	0.61	1.1	-	0.49 ( $\leq 10$ )	-	-
2,3,7,8-TCDF	0.24	0.64	-	0.4 ( $\leq 1.0$ )	-	-
1,2,3,7,8-PeCDF	0.12	5.2U	-	5.08 ( $\leq 5.2$ )	-	-
2,3,4,7,8-PeCDF	0.13	5.2U	-	5.07 ( $\leq 5.2$ )	-	-
1,2,3,4,7,8-HxCDF	0.20	0.79	-	0.59 ( $\leq 5.2$ )	-	-
1,2,3,6,7,8-HxCDF	0.11	0.38	-	0.27 ( $\leq 5.2$ )	-	-
2,3,4,6,7,8-HxCDF	0.091	5.2U	-	5.109 ( $\leq 5.2$ )	-	-
1,2,3,7,8,9-HxCDF	0.12	0.33	-	0.21 ( $\leq 5.2$ )	-	-
1,2,3,4,6,7,8-HpCDF	0.39	1.2	-	0.81 ( $\leq 5.2$ )	-	-
1,2,3,4,7,8,9-HpCDF	0.20	0.63	-	0.43 ( $\leq 5.2$ )	-	-
OCDF	0.92	2.8	-	1.88 ( $\leq 10$ )	-	-

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D080425**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D080425	RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P	Routine calibration (%D) (c)
G0D080425	SSAJ8-01-3BPC**	1,2,3,7,8,9-HxCDF	J+ (all detects)	P	Routine calibration (%D) (c)
G0D080425	SA169-3BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D080425	SSA06-02-1BPC SSA06-02-5BPC RSAQ3-3BPC** RSAQ3-3BPC_FD	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0D080425	SSA06-02-1BPC SSA06-02-5BPC RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD SSA06-02-1BPC_FD SSAJ8-01-3BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D080425	SSA06-02-1BPC SSA06-02-5BPC RSAQ3-3BPC** SA169-3BPC RSAQ3-3BPC_FD SSA06-02-1BPC_FD SSAJ8-01-3BPC**	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  
G0D080425**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D080425	SSA06-02-1BPC	2,3,7,8-TCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.082U pg/g 0.42U pg/g 1.4U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D080425	SSA06-02-5BPC	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 1,2,3,4,6,7,8-HpCDF OCDF	0.14U pg/g 0.72U pg/g 0.12U pg/g 0.12U pg/g 0.084U pg/g 0.093U pg/g 0.21U pg/g 0.49U pg/g	A	bl
G0D080425	RSAQ3-3BPC**	2,3,7,8-TCDD 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	0.038U pg/g 0.23U pg/g 0.61U pg/g 0.24U pg/g 0.12U pg/g 0.13U pg/g 0.20U pg/g	A	bl
G0D080425	RSAQ3-3BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	0.43U pg/g 1.1U pg/g	A	bl
G0D080425	SSAO6-02-1BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	0.30U pg/g 0.62U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D080425**

No Sample Data Qualified in this SDG



LDC #: 23265A21  
 SDG #: G0D080425  
 Laboratory: Test America

**Tronox Northgate Henderson**  
**VALIDATION COMPLETENESS WORKSHEET**  
 Stage 2B/4

Date: 4/8/10  
 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: <u>4/6/10</u>
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ <del>ICV</del>	<del>SW</del>	
V.	Blanks	<del>SW</del>	
VI.	Matrix spike/Matrix spike duplicates	<del>SW</del>	
VII.	Laboratory control samples	A	<u>LOS</u>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	<del>SW</del>	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	<del>SW</del>	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	<del>SW</del>	<u>D = 1 + 6 . 3 + 5</u>
XV.	Field blanks	<del>SW</del>	FB-0407210-RZD (G0D090441)FB04062010-RZB (G0D120488) FB-04072010-RZC (G0D130519)

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

Validated Samples: \*\* level IV

1	SSA06-02-1BPC	c	11	<u>0106187MB</u>	21		31
2	SSA06-02-5BPC	c	12	<u>0110255MB</u>	22		32
3	RSAQ3-3BPC **	B	13		23		33
4	SA169-3BPC		14		24		34
5	RSAQ3-3BPC_FD		15		25		35
6	SSA06-02-1BPC_FD	c	16		26		36
7	SSAJ8-01-3BPC **	D	17		27		37
8	SSA06-02-5BPCMS		18		28		38
9	SSA06-02-5BPCMSD		19		29		39
10			20		30		40

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

LDC #: 03265A21  
 SDG #: see count

VALIDATION FINDINGS CHECKLIST

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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 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

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:













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

**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, 6 Associated Samples:

Compound	Blank ID	5X	Sample Identification
	EB-04072010-RZC		
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, "U".







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 SDG#: See Cover

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

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

Y N NA  
Y N NA

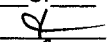
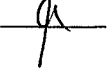
Were field duplicate pairs identified in this SDG?  
 Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	6				
A	0.082	1.0U		0.918	(≤1.0)	
B	0.19	0.082		0.108	(≤5.2)	
C	0.13	5.2U		5.07	(≤5.2)	
D	0.17	0.14		0.03	(≤5.2)	
E	0.19	0.11		0.08	(≤5.2)	
F	0.42	0.30		0.12	(≤5.2)	
G	1.4	0.62		0.78	(≤10)	
H	0.91	0.76		0.15	(≤1.0)	
I	0.62	1.0		0.38	(≤5.2)	
J	0.39	0.47		0.08	(≤5.2)	
K	0.92	1.6		0.68	(≤5.2)	
L	0.49	0.85		0.36	(≤5.2)	
M	0.16	0.27		0.11	(≤5.2)	
N	0.19	0.16		0.03	(≤5.2)	
O	1.2	2.0		0.8	(≤5.2)	
P	0.83	1.1		0.27	(≤5.2)	
Q	2.8	4.4		1.6	(≤10)	

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	5				
A	0.038	1.0U		0.962	(≤1.0)	
C	0.066	5.2U		5.134	(≤5.2)	
D	0.079	0.10		0.021	(≤5.2)	
E	0.15	0.20		0.05	(≤5.2)	
F	0.23	0.43		0.2	(≤5.2)	
G	0.61	1.1		0.49	(≤10)	
H	0.24	0.64		0.4	(≤1.0)	

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SDG#: See Cover

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

Page: 2 of 2  
Reviewer:   
2nd Reviewer: 

**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)		(<math>\leq 50</math>) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	5				
I	0.12	5.2U		5.08	(<math>\leq 5.2</math>)	
J	0.13	5.2U		5.07	(<math>\leq 5.2</math>)	
K	0.20	0.79		0.59	(<math>\leq 5.2</math>)	
L	0.11	0.38		0.27	(<math>\leq 5.2</math>)	
M	0.091	5.2U		5.109	(<math>\leq 5.2</math>)	
N	0.12	0.33		0.21	(<math>\leq 5.2</math>)	
O	0.39	1.2		0.81	(<math>\leq 5.2</math>)	
P	0.20	0.63		0.43	(<math>\leq 5.2</math>)	
Q	0.92	2.8		1.88	(<math>\leq 10</math>)	

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**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:

$RRF = (A_x)(C_s)/(A_s)(C_x)$   
 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,  
 $A_s$  = Area of associated internal standard  
 $C_s$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	RRF (CS3std)	Average RRF (Initial)	RRF (CS3std)	%RSD	%RSD	RRF (CS3 std)	%RSD
1	1042 (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	10.6	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	12.8	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	11.0	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	12.2	12.2	1.05	12.2
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.52	1.437	1.52	14.1	14.0	1.52	14.0
2	1042	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	1.20	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-OCDD)								
3	1042 (405)	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	0.98	4.33
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.04	1.021	1.04	8.03	8.97	1.04	8.97
			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	1.19	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	1.11	3.75
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.51	1.445	1.51	5.85	5.89	1.51	5.89

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	21A104DS	4/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	1.04	9.6	1.04	9.6
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.03	1.2	1.03	1.2
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.26	13.5	1.26	13.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.09	3.0	1.09	3.0
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.50	3.9	1.50	3.9
2	21A104BS	4/22/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	1.03	8.7	1.03	8.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	0.97	5.3	0.97	5.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.13	1.6	1.13	1.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.03	3.5	1.03	3.5
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.42	1.7	1.42	1.7
3	21A104DS	4/28/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.97	2.8	0.97	2.8
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	0.95	6.6	0.95	6.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.09	2.4	1.09	2.4
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.03	4.0	1.03	4.0
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.34	6.9	1.34	6.9

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

VALIDATION FINDINGS WORKSHEET I  
 Routine Calibration Results Verification

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SDG #: Ser con

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	2AP10A15	4/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.92	7.0	0.92	7.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.934	0.93	0.1	0.93	0.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.058	1.15	8.9	1.15	8.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.998	1.06	6.2	1.06	6.2
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.54	7.2	1.54	7.2
2	2AP10A12	4/27/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.04	4.5	1.04	4.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-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      MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 8/9

Compound	Spike Added (pg)		Sample Concentration (pg)	Spiked Sample Concentration (pg)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	21.9	21.9	ND	1.5	20.6	98	98	94	94	4.0	4.3
1,2,3,7,8-PeCDD	11.0	11.0	↓	10.4	10.9	95	95	99	99	4.7	4.7
1,2,3,4,7,8-HxCDD	↓	↓	↓	10.2	13.1	93	93	119	119	2.7	2.5
1,2,3,4,7,8,9-HpCDF	↓	↓	↓	13.2	18.7	120	120	171	170	3.5	3.4
OCDF	21.9	21.9	0.49	26.8	4.00	122	122	182	182	3.9	3.9

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

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

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

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

LCS ID: 0106187

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
2,3,7,8-TCDD	20.0	NA	20.2	NA	101	101								
1,2,3,7,8-PeCDD	100		103		103	103								
1,2,3,4,7,8-HxCDD			96.0		96	96								
1,2,3,4,7,8,9-HpCDF			110		110	110								
OCDF	200		204		102	102								

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>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF			
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		M+4	409.7788	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		M	419.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>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>2</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>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		M+4	425.7737	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		M+2	435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		M+4	437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE		M+4	479.7165	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	NCDPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		LOCK	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>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>2</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF (S)		M+2	457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		M+4	459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	M+2	469.7780		M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	M+4	471.7750		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	M+2	513.6775		M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	M+4	[422.9278]		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDFE	LOCK			LOCK	C <sub>10</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK								
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF							
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> O	HxCDF (S)							
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>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>4</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>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)								
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE								
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK								

(a) The following nuclidic masses were used:

H = 1.007825  
 C = 12.000000  
<sup>13</sup>C = 13.003355  
 F = 18.9984  
 O = 15.994915  
<sup>35</sup>Cl = 34.968853  
<sup>37</sup>Cl = 36.966903

S = internal/recovery standard



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 8, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** GOD130519

**Sample Identification**

FB-04072010-RZC  
EB-04072010-RZC

## Introduction

This data review covers 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.



The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0123308MB	4/19/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 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	0.88 pg/L 1.6 pg/L 2.6 pg/L 0.94 pg/L 0.89 pg/L 0.50 pg/L 1.1 pg/L 0.93 pg/L 0.98 pg/L	All samples in SDG G0D130519

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-RZC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD 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	0.82 pg/L 4.2 pg/L 0.67 pg/L 1.1 pg/L 0.96 pg/L 1.0 pg/L 2.1 pg/L 1.5 pg/L	0.82U pg/L 4.2U pg/L 0.67U pg/L 1.1U pg/L 0.96U pg/L 1.0U pg/L 2.1U pg/L 1.5U pg/L
EB-04072010-RZC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD 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	0.65 pg/L 5.5 pg/L 1.0 pg/L 1.8 pg/L 1.1 pg/L 0.97 pg/L 4.5 pg/L 1.1 pg/L	0.65U pg/L 5.5U pg/L 1.0U pg/L 1.8U pg/L 1.1U pg/L 0.97U pg/L 4.5U pg/L 1.1U pg/L

Sample EB-04072010-RZC was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04072010-RZC	4/8/10	2,3,7,8-TCDD 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	1.0 pg/L 0.69 pg/L 0.65 pg/L 5.5 pg/L 53 pg/L 2.6 pg/L 1.5 pg/L 1.0 pg/L 1.8 pg/L 1.1 pg/L 0.97 pg/L 4.5 pg/L 1.1 pg/L 12 pg/L	No associated samples in this SDG

Sample FB-04072010-RZC 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-RZC	4/8/10	1,2,3,4,7,8-HxCDD	0.77 pg/L	No associated samples in this SDG
		1,2,3,6,7,8-HxCDD	0.74 pg/L	
		1,2,3,7,8,9-HxCDD	0.82 pg/L	
		1,2,3,4,6,7,8-HpCDD	4.2 pg/L	
		OCDD	37 pg/L	
		2,3,7,8-TCDF	0.57 pg/L	
		1,2,3,7,8-PeCDF	0.96 pg/L	
		2,3,4,7,8-PeCDF	0.67 pg/L	
		1,2,3,4,7,8-HxCDF	1.1 pg/L	
		1,2,3,6,7,8-HxCDF	0.96 pg/L	
		2,3,4,6,7,8-HxCDF	1.0 pg/L	
		1,2,3,7,8,9-HxCDF	1.0 pg/L	
		1,2,3,4,6,7,8-HpCDF	2.1 pg/L	
		1,2,3,4,7,8,9-HpCDF	1.5 pg/L	
		OCDF	6.7 pg/L	

#### 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 G0D130519	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 G0D130519	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 G0D130519	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 G0D130519**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D130519	FB-04072010-RZC EB-04072010-RZC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0D130519	FB-04072010-RZC EB-04072010-RZC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D130519	FB-04072010-RZC EB-04072010-RZC	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 G0D130519**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D130519	FB-04072010-RZC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD 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	0.82U pg/L 4.2U pg/L 0.67U pg/L 1.1U pg/L 0.96U pg/L 1.0U pg/L 2.1U pg/L 1.5U pg/L	A	bl
G0D130519	EB-04072010-RZC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD 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	0.65U pg/L 5.5U pg/L 1.0U pg/L 1.8U pg/L 1.1U pg/L 0.97U pg/L 4.5U pg/L 1.1U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D130519**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D130519**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265C21  
 SDG #: G0D130519  
 Laboratory: Test America

Date: 4/8/10  
 Page: 11 of 17  
 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	W	
VI.	Matrix spike/Matrix spike duplicates	N	diat spiked
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	All EMPC - JK(E)
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB = 1. EB = 2

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

Validated Samples:

1	210-RZC FB-040710-RZC	W	11	0103308MB	21		31
2	210-RZC EB-040710-RZC	W	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:





LDC #: 23265C21  
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)

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: EB Associated Samples: None

Compound	Blank ID	Sample Identification
	EB-04072010-RZC	5X
A	1.0	0.005
D	0.69	0.00345
E	0.65	0.00325
F	5.5	0.0275
G	53	0.265
H	2.6	0.013
I	1.5	0.0075
J	1.0	0.005
K	1.8	0.009
L	1.1	0.0055
M	0.97	0.00485
O	4.5	0.0225
P	1.1	0.0055
Q	12	0.06
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**  
**Field Blanks**

**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: None Associated Samples: None

Compound	Blank ID	5X	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, "U".



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 13, 2010  
**LDC Report Date:** June 10, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D150462

**Sample Identification**

SSAI3-02-1BPC  
SSAI3-03-1BPC  
SSAK3-01-1BPC  
SSAJ3-03-1BPC  
SSAI2-01-1BPC\*\*  
SA207-12BPC  
SSAI3-02-1BPCMS  
SSAI3-02-1BPCMSD

\*\*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.

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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SA207-12BPC
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	SSAI3-02-1BPC SSAI3-03-1BPC SSAK3-01-1BPC SSAJ3-03-1BPC SSAI2-01-1BPC**

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
SSAI3-02-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF	0.31 pg/g 0.31 pg/g 0.34 pg/g 0.53 pg/g	0.31U pg/g 0.31U pg/g 0.34U pg/g 0.53U pg/g
SSAI3-03-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.47 pg/g 2.8 pg/g 0.22 pg/g	0.47U pg/g 2.8U pg/g 0.22U pg/g
SSAJ3-03-1BPC	OCDD 2,3,7,8-TCDF 1,2,3,7,8,9-HxCDF	0.87 pg/g 1.1 pg/g 0.30 pg/g	0.87U pg/g 1.1U pg/g 0.30U pg/g

Sample EB-04132010-RIG3-RZD (from SDG G0D150582) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04132010-RIG3-RZD	4/13/10	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.39 pg/L 1.3 pg/L 3.1 pg/L 2.8 pg/L 2.3 pg/L 1.4 pg/L 4.6 pg/L 2.7 pg/L 1.0 pg/L 0.48 pg/L 5.9 pg/L 2.3 pg/L 9.9 pg/L	SSAI3-02-1BPC SSAI3-03-1BPC SSAK3-01-1BPC SSAJ3-03-1BPC SSAI2-01-1BPC**

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

Samples FB-04072010-RZD (from SDG G0D090441) 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-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	SSAI3-02-1BPC SSAI3-03-1BPC SSAK3-01-1BPC SSAJ3-03-1BPC SSAI2-01-1BPC**
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	SA207-12BPC

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAI3-03-1BPC	<sup>13</sup> C-OCDD	22 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAJ3-03-1BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## 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
SSAK3-01-1BPC	1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D150462	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 G0D150462	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 SSAI3-03-1BPC\_FD (from SDG G0D150589) and SSAI3-03-1BPC and samples SSAI2-01-1BPC\_FD (from SDG G0D150589) and SSAI2-01-1BPC were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI3-03-1BPC_FD	SSAI3-03-1BPC				
1,2,3,7,8-PeCDD	0.12	2.5U	-	2.38 (≤2.5)	-	-
1,2,3,4,7,8-HxCDD	0.090	0.061	-	0.029 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	0.24	0.16	-	0.08 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	0.24	0.17	-	0.07 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	2.8	0.47	-	2.33 (≤2.6)	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI3-03-1BPC_FD	SSAI3-03-1BPC				
OCDD	59	2.8	-	56.2 ( $\leq 5.2$ )	J (all detects)	A
2,3,7,8-TCDF	0.80	0.49U	-	0.31 ( $\leq 0.49$ )	-	-
1,2,3,7,8-PeCDF	0.91	0.72	-	0.19 ( $\leq 2.6$ )	-	-
2,3,4,7,8-PeCDF	0.55	0.33	-	0.22 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDF	1.7	1.5	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDF	1.1	1.2	-	0.1 ( $\leq 2.6$ )	-	-
2,3,4,6,7,8-HxCDF	0.29	0.25	-	0.04 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDF	0.30	0.22	-	0.08 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDF	3.9	3.1	-	0.8 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8,9-HpCDF	1.6	1.4	-	0.2 ( $\leq 2.6$ )	-	-
OCDF	16	12	-	4 ( $\leq 5.2$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI2-01-1BPC_FD	SSAI2-01-1BPC				
2,3,7,8-TCDD	0.10	0.13	-	0.03 ( $\leq 0.53$ )	-	-
1,2,3,7,8-PeCDD	0.23	0.18	-	0.05 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDD	0.26	0.24	-	0.02 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDD	3.3	2.9	-	0.4 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDD	1.3	0.85	-	0.45 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDD	22	21	5 ( $\leq 50$ )	-	-	-
OCDD	2.8	3.6	-	0.8 ( $\leq 5.3$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI2-01-1BPC_FD	SSAI2-01-1BPC				
2,3,7,8-TCDF	1.4	2.3	-	0.9 ( $\leq 0.53$ )	-	-
1,2,3,7,8-PeCDF	2.6	3.8	-	1.2 ( $\leq 2.6$ )	-	-
2,3,4,7,8-PeCDF	1.5	1.7	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDF	8.0	7.1	-	0.9 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDF	4.9	5.7	-	0.8 ( $\leq 2.6$ )	-	-
2,3,4,6,7,8-HxCDF	1.1	1.3	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDF	0.85	0.61	-	0.24 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDF	18	19	5 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	7.3	9.0	-	1.7 ( $\leq 2.6$ )	-	-
OCDF	48	60	22 ( $\leq 50$ )	-	-	-

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D150462**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D150462	SSAI3-03-1BPC SSAJ3-03-1BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D150462	SSAK3-01-1BPC	1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D150462	SSAI3-02-1BPC SSAI3-03-1BPC SSAK3-01-1BPC SSAJ3-03-1BPC SSAI2-01-1BPC** SA207-12BPC	All compounds reported below the PQL	J (all detects)	A	Project Quantitation Limit (sp)
G0D150462	SSAI3-02-1BPC SSAI3-03-1BPC SSAK3-01-1BPC SSAJ3-03-1BPC SSAI2-01-1BPC** SA207-12BPC	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D150462	SSAI3-03-1BPC	OCDD	J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D150462**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D150462	SSAI3-02-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF	0.31U pg/g 0.31U pg/g 0.34U pg/g 0.53U pg/g	A	bl
G0D150462	SSAI3-03-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8,9-HxCDF	0.47U pg/g 2.8U pg/g 0.22U pg/g	A	bl
G0D150462	SSAJ3-03-1BPC	OCDD 2,3,7,8-TCDF 1,2,3,7,8,9-HxCDF	0.87U pg/g 1.1U pg/g 0.30U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D150462**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D150462**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 23265D21  
 SDG #: G0D150462  
 Laboratory: Test America

Date: 4/8/10  
 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: 4/13/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/TCV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	109
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	W	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	W	D=2+SSAI3-03-1BPC-FD, 5+SSAI2-01-7BPC(G0D150589)
XV.	Field blanks	W	FB-04072010-R2D(G0D090441), FB-04072010-R2C(G0D150589), EB-04132010-R1G3-R2D(G0D150589)

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

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

1	SSAI3-02-1BPC	11	0109260MB	21	31
2	SSAI3-03-1BPC	12	0110455MB	22	32
3	SSAK3-01-1BPC	13		23	33
4	SSAJ3-03-1BPC	14		24	34
5	SSAI2-01-1BPC **	15		25	35
6	SA207-12BPC	16		26	36
7	SSAI3-02-1BPCMS	17		27	37
8	SSAI3-02-1BPCMSD	18		28	38
9		19		29	39
10		20		30	40

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

VALIDATION FINDINGS CHECKLIST

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 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"/>	

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:











LDC # 23265D21  
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: Associated Samples: 6

Compound	Blank ID	5X	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, "U".





LDC#: 23265D21  
SDG#: See Cover

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

Page: 1 of 2  
Reviewer:   
2nd Reviewer: 

**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)
	SSAI3-03-1BPC_FD	2				
B	0.12	2.5U		2.38	( <=2.5)	
C	0.090	0.061		0.029	( <=2.6)	
D	0.24	0.16		0.08	( <=2.6)	
E	0.24	0.17		0.07	( <=2.6)	
F	2.8	0.47		2.33	( <=2.6)	
G	59	2.8		56.2	( <=5.2)	Islets A
H	0.80	0.49U		0.31	( <=0.49)	
I	0.91	0.72		0.19	( <=2.6)	
J	0.55	0.33		0.22	( <=2.6)	
K	1.7	1.5		0.2	( <=2.6)	
L	1.1	1.2		0.1	( <=2.6)	
M	0.29	0.25		0.04	( <=2.6)	
N	0.30	0.22		0.08	( <=2.6)	
O	3.9	3.1		0.8	( <=2.6)	
P	1.6	1.4		0.2	( <=2.6)	
Q	16	12		4	( <=5.2)	

Compound	Concentration (pg/g)		(<=50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	SSAI2-01-1BPC_FD	5				
A	0.10	0.13		0.03	( <=0.53)	
B	0.23	0.18		0.05	( <=2.6)	
C	0.26	0.24		0.02	( <=2.6)	
D	3.3	2.9		0.4	( <=2.6)	
E	1.3	0.85		0.45	( <=2.6)	
F	22	21	5			
G	2.8	3.6		0.8	( <=5.3)	
H	1.4	2.3		0.9	( <=0.53)	

LDC#: 23265<sup>P</sup>F21  
 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)		( $\leq 50$ )	(pg/g)	(pg/g)	Qualifications (Parent Only)
	SSAI2-01-1BPC_FD	5	RPD	Difference	Limits	
I	2.6	3.8		1.2	( $\leq 2.6$ )	
J	1.5	1.7		0.2	( $\leq 2.6$ )	
K	8.0	7.1		0.9	( $\leq 2.6$ )	
L	4.9	5.7		0.8	( $\leq 2.6$ )	
M	1.1	1.3		0.2	( $\leq 2.6$ )	
N	0.85	0.61		0.24	( $\leq 2.6$ )	
O	18	19	5			
P	7.3	9.0		1.7	( $\leq 2.6$ )	
Q	48	60	22			

**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,  
 $X$  = 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)	Average RRF (Initial)		Average RRF (Recalculated)		RRF (CS3std)		RRF (CS3std)		%RSD		
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated	Reported	Recalculated			
1	12AZ (305)	3/4/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.98315	0.98315	1.0872	1.01872	1.0872	1.01872	4.65926	4.65926	4.65926	4.659	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.05105	1.05105	1.11325	1.11325	1.11325	1.11325	7.43940	7.43940	7.43940	7.440	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.08449	1.08449	1.09101	1.09101	1.09101	1.09101	5.63091	5.63091	5.63091	5.631	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.03068	1.03068	1.08562	1.08562	1.08562	1.08562	4.49467	4.49467	4.49467	4.495	
			OCDF ( <sup>13</sup> C-OCDF)	1.42582	1.42582	1.57900	1.57900	1.57900	1.57900	8.93881	8.93881	8.93881	8.939	
2	12AZ (405)	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	0.98	0.98	4.44	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	1.04	1.04	3.03	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	1.19	1.19	5.33	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	1.11	1.11	3.60	3.60	3.60	3.75	
			OCDF ( <sup>13</sup> C-OCDF)	1.445	1.445	1.51	1.51	1.51	1.51	5.83	5.83	5.83	5.89	
3	12AZ	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	1.10	1.10	1.09	1.09	1.09	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)											

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 #: 226522  
 SDG #: 226522

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 Reviewer: [Signature]  
 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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       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	<u>24810405</u>	<u>4/29/10</u>	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
2	<u>28810405</u>	<u>4/29/10</u>	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.98315	0.91217	7.2	0.91217	7.2
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.05105	1.06595	1.4	1.06595	1.4
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.08249	1.08867	0.1	1.08867	0.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.03068	1.06354	3.2	1.06354	3.2
			OCDF ( <sup>13</sup> C-OCDD)	1.42582	1.43843	0.9	1.43843	0.9
3	<u>611102512</u>	<u>5/1/10</u>	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.08	1.0	1.08	1.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-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 # 236502  
 SDG #: RLC004

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

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

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

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

% Recovery =  $100 * SSC/SA$

Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $100 * |LCS - LCSD| / ((LCS + LCSD) / 2)$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0109260

Compound	Spike Added (ppg)		Spiked Sample Concentration (ppg)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	NA	19.8	NA	99	98								
1,2,3,7,8-PeCDD	100		102		102	102								
1,2,3,4,7,8-HxCDD			108		108	108								
1,2,3,4,7,8,9-HpCDF			104		104	104								
OCDF	200		202		101	101								

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

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 13, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D150582

**Sample Identification**

EB-04132010-RIG3-RZD  
FB-04132010-RIG2-RZE

## Introduction

This data review covers 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0111312MB	4/21/10	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 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	0.64 pg/L 0.83 pg/L 1.0 pg/L 1.3 pg/L 2.1 pg/L 6.6 pg/L 0.78 pg/L 0.79 pg/L 1.2 pg/L 1.2 pg/L 1.6 pg/L 2.2 pg/L 1.6 pg/L 1.7 pg/L 3.5 pg/L	All samples in SDG GOD150582

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04132010-RIG3-RZD	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.39 pg/L 1.3 pg/L 3.1 pg/L 2.3 pg/L 1.4 pg/L 4.6 pg/L 2.7 pg/L 1.0 pg/L 0.48 pg/L 5.9 pg/L 2.3 pg/L 9.9 pg/L	0.39U pg/L 1.3U pg/L 3.1U pg/L 2.3U pg/L 1.4U pg/L 4.6U pg/L 2.7U pg/L 1.0U pg/L 0.48U pg/L 5.9U pg/L 2.3U pg/L 9.9U pg/L
FB-04132010-RIG2-RZE	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.40 pg/L 0.65 pg/L 2.5 pg/L 0.66 pg/L 0.41 pg/L 0.53 pg/L 0.97 pg/L	0.40U pg/L 0.65U pg/L 2.5U pg/L 0.66U pg/L 0.41U pg/L 0.53U pg/L 0.97U pg/L

Sample EB-04132010-RIG3-RZD was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:



Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04132010-RIG3-RZD	4/13/10	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.39 pg/L 1.3 pg/L 3.1 pg/L 2.8 pg/L 2.3 pg/L 1.4 pg/L 4.6 pg/L 2.7 pg/L 1.0 pg/L 0.48 pg/L 5.9 pg/L 2.3 pg/L 9.9 pg/L	No associated samples in this SDG

Sample FB-04132010-RIG2-RZE 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-04132010-RIG2-RZE	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.40 pg/L 0.65 pg/L 2.5 pg/L 0.66 pg/L 0.41 pg/L 0.53 pg/L 0.97 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
EB-04132010-RIG3-RZD	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 G0D150582	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 G0D150582	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 G0D150582**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D150582	EB-04132010-RIG3-RZD	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0D150582	EB-04132010-RIG3-RZD FB-04132010-RIG2-RZE	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D150582	EB-04132010-RIG3-RZD FB-04132010-RIG2-RZE	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 G0D150582**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D150582	EB-04132010-RIG3-RZD	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.39U pg/L 1.3U pg/L 3.1U pg/L 2.3U pg/L 1.4U pg/L 4.6U pg/L 2.7U pg/L 1.0U pg/L 0.48U pg/L 5.9U pg/L 2.3U pg/L 9.9U pg/L	A	bl
G0D150582	FB-04132010-RIG2-RZE	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.40U pg/L 0.65U pg/L 2.5U pg/L 0.66U pg/L 0.41U pg/L 0.53U pg/L 0.97U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D150582**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D150582**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265E21

SDG #: G0D150582

Laboratory: Test America

Date: 6/8/10

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: 4/13/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	N	diel specified
VII.	Laboratory control samples	A	100
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	W	EB = 1. FB = 2.

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	EB-04132010-RIG3-RZD W	11		21		31	
2	FB-04132010-RIG2-RZE V	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 PeCDD
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:







**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?

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

**Sampling date:** 4/13/10

**Field blank type:** (circle one) Field Blank / Rinsate / Other: None

Compound	Blank ID	5X	Associated Samples:	Sample Identification
	FB-04132010-RIG2-RZF	5X		
E	0.40	0.002		
F	0.65	0.00325		
G	2.5	0.0125		
K	0.66	0.0033		
M	0.41	0.00205		
O	0.53	0.00265		
Q	0.97	0.00485		
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 # 22627  
SDG # 202

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 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
		<u>M</u>	<u>zape results</u>	<u>u</u>	<u>NK (k)</u>
		<u>1</u>	<u>No confirmation for 2.3.7.8-TCDF</u>	<u>1</u>	<u>None</u>

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 13, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D150589

**Sample Identification**

SSAI3-03-1BPC\_FD  
SSAI2-01-1BPC\_FD

## Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
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 GOD150589

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
SSAI3-03-1BPC_FD	2,3,7,8-TCDF 1,2,3,7,8,9-HxCDF	0.80 pg/g 0.30 pg/g	0.80U pg/g 0.30U pg/g
SSAI2-01-1BPC_FD	OCDD	2.8 pg/g	2.8U pg/g

Sample EB-04132010-RIG3-RZD (from SDG GOD150582) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04132010-RIG3-RZD	4/13/10	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.39 pg/L 1.3 pg/L 3.1 pg/L 2.8 pg/L 2.3 pg/L 1.4 pg/L 4.6 pg/L 2.7 pg/L 1.0 pg/L 0.48 pg/L 5.9 pg/L 2.3 pg/L 9.9 pg/L	All samples in SDG GOD150589

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

Sample FB-04072010-RZD (from SDG GOD090441) 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 G0D150589

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
SSAI3-03-1BPC_FD	<sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	38 (40-135) 28 (40-135) 25 (40-135) 10 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P



## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D150589	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 G0D150589	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

Samples SSAI3-03-1BPC\_FD and SSAI3-03-1BPC (from SDG G0D150462) and samples SSAI2-01-1BPC\_FD and SSAI2-01-1BPC (from SDG G0D150462) were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI3-03-1BPC_FD	SSAI3-03-1BPC				
1,2,3,7,8-PeCDD	0.12	2.5U	-	2.38 ( $\leq 2.5$ )	-	-
1,2,3,4,7,8-HxCDD	0.09	0.061	-	0.029 ( $\leq 2.6$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI3-03-1BPC_FD	SSAI3-03-1BPC				
1,2,3,6,7,8-HxCDD	0.24	0.16	-	0.08 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDD	0.24	0.17	-	0.07 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDD	2.8	0.47	-	2.33 ( $\leq 2.6$ )	-	-
OCDD	59	2.8	-	56.2 ( $\leq 5.2$ )	J (all detects)	A
2,3,7,8-TCDF	0.80	0.49U	-	0.31 ( $\leq 0.49$ )	-	-
1,2,3,7,8-PeCDF	0.91	0.72	-	0.19 ( $\leq 2.6$ )	-	-
2,3,4,7,8-PeCDF	0.55	0.33	-	0.22 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDF	1.7	1.5	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDF	1.1	1.2	-	0.1 ( $\leq 2.6$ )	-	-
2,3,4,6,7,8-HxCDF	0.29	0.25	-	0.04 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDF	0.30	0.22	-	0.08 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDF	3.9	3.1	-	0.8 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8,9-HpCDF	1.6	1.4	-	0.2 ( $\leq 2.6$ )	-	-
OCDF	16	12	-	4 ( $\leq 5.2$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI2-01-1BPC_FD	SSAI2-01-1BPC				
2,3,7,8-TCDD	0.10	0.13	-	0.03 ( $\leq 0.53$ )	-	-
1,2,3,7,8-PeCDD	0.23	0.18	-	0.05 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDD	0.26	0.24	-	0.02 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDD	3.3	2.9	-	0.4 ( $\leq 2.6$ )	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAI2-01-1BPC_FD	SSAI2-01-1BPC				
1,2,3,7,8,9-HxCDD	1.3	0.85	-	0.45 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDD	22	21	5 ( $\leq 50$ )	-	-	-
OCDD	2.8	3.6	-	0.8 ( $\leq 5.3$ )	-	-
2,3,7,8-TCDF	1.4	2.3	-	0.9 ( $\leq 0.53$ )	-	-
1,2,3,7,8-PeCDF	2.6	3.8	-	1.2 ( $\leq 2.6$ )	-	-
2,3,4,7,8-PeCDF	1.5	1.7	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDF	8.0	7.1	-	0.9 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDF	4.9	5.7	-	0.8 ( $\leq 2.6$ )	-	-
2,3,4,6,7,8-HxCDF	1.1	1.3	-	0.2 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDF	0.85	0.61	-	0.24 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDF	18	19	5 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	7.3	9.0	-	1.7 ( $\leq 2.6$ )	-	-
OCDF	48	60	22 ( $\leq 50$ )	-	-	-

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D150589**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D150589	SSAI3-03-1BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D150589	SSAI3-03-1BPC_FD SSAI2-01-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D150589	SSAI3-03-1BPC_FD SSAI2-01-1BPC_FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D150589	SSAI3-03-1BPC_FD	OCDD	J (all detects)	A	Field duplicates (Difference) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D150589**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D150589	SSAI3-03-1BPC_FD	2,3,7,8-TCDF 1,2,3,7,8,9-HxCDF	0.80U pg/g 0.30U pg/g	A	bl
G0D150589	SSAI2-01-1BPC_FD	OCDD	2.8U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D150589**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D150589**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265F21  
 SDG #: G0D150589  
 Laboratory: Test America

Date: 6/3/10  
 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: 4/13/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No sp/ ass'd - No Anal
VII.	Laboratory control samples	A	LOS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	TW/N	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	TW	D = 1 + SSA13-03-1BPC, 2 + SSA12-01-1BPC (CFD15046
XV.	Field blanks	TW	FB-04072010-R2D (CFD090441), 2B-04132010-R3R2 CFD150582

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

Validated Samples:

1	SSAI3-03-1BPC_FD	5	11	0109=60NB	21		31
2	SSAI2-01-1BPC_FD	✓	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 PeCDD
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

**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

Compound	Blank ID	5X	1	2	Sample Identification
	0109260MR				
F	0.14	0.7	-	-	
G	0.59	2.95	-	2.8U	
H	0.28	1.4	0.80/U	-	
I	0.14	0.7	-	-	
K	0.24	1.2	-	-	
L	0.14	0.7	-	-	
N	0.086	0.43	0.30/U	-	
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".

**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?

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

Sampling date: 4/13/10

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

Associated Samples: All

Compound	Blank ID	Sample Identification																
	FB-04132010-RIG3-RZD											5X						
D	0.39											0.00195						
F	1.3											0.0065						
G	3.1											0.0155						
H	2.8											0.014						
I	2.3											0.0115						
J	1.4											0.007						
K	4.6											0.023						
L	2.7											0.0135						
M	1.0											0.005						
N	0.48											0.0024						
O	5.9											0.0295						
P	2.3											0.0115						
Q	9.9											0.0495						
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#: 23265F21  
 SDG#: See Cover

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

Page: 1 of 2  
 Reviewer: Q  
 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)
	1	SSAI3-03-1BPC				
B	0.12	2.5U		2.38	( ≤2.5)	
C	0.090	0.061		0.029	( ≤2.6)	
D	0.24	0.16		0.08	( ≤2.6)	
E	0.24	0.17		0.07	( ≤2.6)	
F	2.8	0.47		2.33	( ≤2.6)	
G	59	2.8		56.2	( ≤5.2)	<u>lots A</u> (+d)
H	0.80	0.49U		0.31	( ≤0.49)	
I	0.91	0.72		0.19	( ≤2.6)	
J	0.55	0.33		0.22	( ≤2.6)	
K	1.7	1.5		0.2	( ≤2.6)	
L	1.1	1.2		0.1	( ≤2.6)	
M	0.29	0.25		0.04	( ≤2.6)	
N	0.30	0.22		0.08	( ≤2.6)	
O	3.9	3.1		0.8	( ≤2.6)	
P	1.6	1.4		0.2	( ≤2.6)	
Q	16	12		4	( ≤5.2)	

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	SSAI2-01-1BPC				
A	0.10	0.13		0.03	( ≤0.53)	
B	0.23	0.18		0.05	( ≤2.6)	
C	0.26	0.24		0.02	( ≤2.6)	
D	3.3	2.9		0.4	( ≤2.6)	
E	1.3	0.85		0.45	( ≤2.6)	
F	22	21	5			
G	2.8	3.6		0.8	( ≤5.3)	
H	1.4	2.3		0.9	( ≤0.53)	

LDC#: 23265F21  
SDG#: See Cover

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

Page: 2 of 2  
Reviewer:                       
2nd Reviewer:                     

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

Y N NA Were field duplicate pairs identified in this SDG?

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

Compound	Concentration (pg/g)		(≤50)	(pg/g)	(pg/g)	Qualifications (Parent Only)
	2	SSAI2-01-1BPC	RPD	Difference	Limits	
I	2.6	3.8		1.2	(≤2.6)	
J	1.5	1.7		0.2	(≤2.6)	
K	8.0	7.1		0.9	(≤2.6)	
L	4.9	5.7		0.8	(≤2.6)	
M	1.1	1.3		0.2	(≤2.6)	
N	0.85	0.61		0.24	(≤2.6)	
O	18	19	5			
P	7.3	9.0		1.7	(≤2.6)	
Q	48	60	22			

V:\FIELD DUPLICATES\23265F21.wpd

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 14 through April 15, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** GOD160435

**Sample Identification**

SSAL4-01-1BPC  
SSAL4-02-1BPC  
SSAK5-01-1BPC  
SSAO6-01-1BPC  
SSAL2-01-1BPC  
SSAL3-01-1BPC  
SSAL3-02-1BPC  
SSAK4-01-1BPC

## 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.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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



## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0113286MB	4/23/10	OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.75 pg/g 0.22 pg/g 0.25 pg/g 0.35 pg/g 0.50 pg/g 0.16 pg/g 0.75 pg/g	SSAL2-01-1BPC
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SSAL4-01-1BPC SSAL4-02-1BPC SSAK5-01-1BPC SSAO6-01-1BPC SSAL3-01-1BPC SSAK4-01-1BPC
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	SSAL3-02-1BPC

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
SSAL2-01-1BPC	OCDD	3.4 pg/g	3.4U pg/g
SSAL3-01-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF	0.90 pg/g 1.0 pg/g	0.90U pg/g 1.0U pg/g

Samples EB-04142010-RIG1-RZC and EB-04142010-RIG2-RZC (both from SDG GOD160472) were identified as equipment 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-04142010-RIG1-RZC	4/14/10	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.2 pg/L 9.2 pg/L 0.50 pg/L 0.32 pg/L 0.69 pg/L 0.39 pg/L 0.28 pg/L 0.20 pg/L 0.79 pg/L 0.30 pg/L 1.8 pg/L	SSAO6-01-1BPC

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04142010-RIG2-RZC	4/14/10	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	0.29 pg/L 0.39 pg/L 0.77 pg/L 2.5 pg/L 3.3 pg/L 1.6 pg/L 0.73 pg/L 4.2 pg/L 2.2 pg/L 0.71 pg/L 0.43 pg/L 6.6 pg/L 1.9 pg/L 13 pg/L	SSAO6-01-1BPC

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

Samples FB-04072010-RZC (from SDG G0D130519) and FB-04072010-RZD (from SDG G0D090441) 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-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	SSAO6-01-1BPC
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	SSAL4-01-1BPC SSAL4-02-1BPC SSAK5-01-1BPC SSAL2-01-1BPC SSAL3-01-1BPC SSAL3-02-1BPC SSAK4-01-1BPC

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
SSAL4-01-1BPC	<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
SSAL4-02-1BPC	<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
SSAO6-01-1BPC	<sup>13</sup> C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-01-1BPC	<sup>13</sup> C-OCDD	22 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL3-01-1BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## 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
SSAL4-02-1BPC	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)	P
SSAK5-01-1BPC	OCDD 2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SSAK4-01-1BPC	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	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D160435	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 G0D160435	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 G0D160435**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D160435	SSAL4-01-1BPC SSAL4-02-1BPC SSAO6-01-1BPC SSAL2-01-1BPC SSAL3-01-1BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D160435	SSAL4-02-1BPC	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)	P	Project Quantitation Limit (e)
G0D160435	SSAK5-01-1BPC	OCDD 2,3,7,8-TCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D160435	SSAK4-01-1BPC	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	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D160435	SSAL4-01-1BPC SSAL4-02-1BPC SSAK5-01-1BPC SSAO6-01-1BPC SSAL2-01-1BPC SSAL3-01-1BPC SSAL3-02-1BPC SSAK4-01-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D160435	SSAL4-01-1BPC SSAL4-02-1BPC SSAK5-01-1BPC SSAO6-01-1BPC SSAL2-01-1BPC SSAL3-01-1BPC SSAL3-02-1BPC SSAK4-01-1BPC	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  
G0D160435**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D160435	SSAL2-01-1BPC	OCDD	3.4U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D160435	SSAL3-01-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF	0.90U pg/g 1.0U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D160435**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D160435**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265G21  
 SDG #: G0D160435  
 Laboratory: Test America

Date: 4/2/10  
 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: 4/15/10, 4/14/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No spl ass'd - No Anal
VII.	Laboratory control samples	A	LCC
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	EB-04142010-R1F1-R2C, EB-04142010-R1F2-R2C (F0D166), FB-04072010-R2D (F0D090441), FB-04072010 (F0D13059)

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

Validated Samples:

MM 50:15

1	SSAL4-01-1BPC	11	0110455MB	21		31	
2	SSAL4-02-1BPC	12	0112236MB	22		32	
3	SSAK5-01-1BPC	13		23		33	
4	SSAO6-01-1BPC	14		24		34	
5	SSAL2-01-1BPC	15		25		35	
6	SSAL3-01-1BPC	16		26		36	
7	SSAL3-02-1BPC	17		27		37	
8	SSAK4-01-1BPC	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:

LDC #: 32657  
 SDG #: 201

**VALIDATION FINDINGS WORKSHEET**

**Blanks**

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

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

Conc. units: ppg Associated Samples: 5

Compound	Blank ID	Sample Identification
[Shaded]	<u>0.13</u>	<u>MB</u>
<u>F</u>	<u>0.75</u>	<u>5</u>
<u>H</u>	<u>0.22</u>	<u>34</u>
<u>I</u>	<u>0.25</u>	<u>(1.7)</u>
<u>K</u>	<u>0.35</u>	<u>(2.2)</u>
<u>O</u>	<u>0.50</u>	<u>(4.6)</u>
<u>T</u>	<u>0.16</u>	<u>(13)</u>
<u>R</u>	<u>0.75</u>	<u>(5.0)</u>
		<u>(3.9)</u>

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

Compound	Blank ID	Sample Identification
[Shaded]		

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

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

**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) (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:** 4/20/10 **Blank analysis date:** 4/28/10

**Conc. units:** ppb **Associated Samples:** 1-4, 6, 8 (bl)

Compound	Blank ID	Sample Identification
	011P455H2	
H	0.31	6
I	0.31	0.90N 1.0N
O	0.30	4.2

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

**Conc. units:** ppb **Associated Samples:** 7 (1, 2, 5-X)

Compound	Blank ID	Sample Identification
	011P236H2	
F	0.85	
O	0.64	
B	1.3	

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

LDC #: 23265G21  
 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: 4/14/10  
 Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: 4 (>5X)

Compound	Blank ID	Sample Identification				
	FB-04142010-RIG1-RZC			5X		
F	1.2			0.006		
G	9.2			0.046		
H	0.50			0.0025		
I	0.32			0.0016		
K	0.69			0.00345		
L	0.39			0.00195		
M	0.28			0.0014		
N	0.20			0.001		
O	0.79			0.00395		
P	0.30			0.0015		
Q	1.8			0.009		
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 #: 23265G21  
 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: 4/14/10

Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: 4 (>5X)

Compound	Blank ID	Sample Identification
	EB-04142010-RIG2-RZC	5X
D	0.29	0.00145
E	0.39	0.00195
F	0.77	0.00385
G	2.5	0.0125
H	3.3	0.0165
I	1.6	0.008
J	0.73	0.00365
K	4.2	0.021
L	2.2	0.011
M	0.71	0.00355
N	0.43	0.00215
O	6.6	0.033
P	1.9	0.0095
Q	13	0.065
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**  
**Field Blanks**

**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: Associated Samples: 4 (>5X)

Compound	Blank ID	5X	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, "U".

LDC #: 23265G21  
 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 8230)

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: \_\_\_\_\_

Associated Samples: 1-3, 5-8

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".





LDC #: 232654  
 SDG #: See below

**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 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 (R)
			<u>spds &gt; edit range</u>		
		<u>2</u>	<u>I.K.L.O.P.R</u>	<u>2</u>	<u>White/P (e)</u>
		<u>3</u>	<u>S.H.</u>	<u>3</u>	<u>↓</u>
		<u>8</u>	<u>I.N.K.L.</u>	<u>8</u>	<u>White/A (e)</u>
		<u>All</u>	<u>zinc results</u>	<u>ml</u>	<u>VF (K)</u>

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 14, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D160472

**Sample Identification**

EB-04142010-RIG1-RZC  
EB-04142010-RIG2-RZC

## Introduction

This data review covers 2 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0111312MB	4/21/10	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 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	0.64 pg/L 0.83 pg/L 1.0 pg/L 1.3 pg/L 2.1 pg/L 6.6 pg/L 0.78 pg/L 0.79 pg/L 1.2 pg/L 1.2 pg/L 1.6 pg/L 2.2 pg/L 1.6 pg/L 1.7 pg/L 3.5 pg/L	All samples in SDG G0D160472

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04142010-RIG1-RZC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.2 pg/L 9.2 pg/L 0.32 pg/L 0.69 pg/L 0.39 pg/L 0.28 pg/L 0.20 pg/L 0.79 pg/L 0.30 pg/L 1.8 pg/L	1.2U pg/L 9.2U pg/L 0.32U pg/L 0.69U pg/L 0.39U pg/L 0.28U pg/L 0.20U pg/L 0.79U pg/L 0.30U pg/L 1.8U pg/L
EB-04142010-RIG2-RZC	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.29 pg/L 0.39 pg/L 0.77 pg/L 2.5 pg/L 1.6 pg/L 0.73 pg/L 4.2 pg/L 2.2 pg/L 0.71 pg/L 0.43 pg/L 6.6 pg/L 1.9 pg/L 13 pg/L	0.29U pg/L 0.39U pg/L 0.77U pg/L 2.5U pg/L 1.6U pg/L 0.73U pg/L 4.2U pg/L 2.2U pg/L 0.71U pg/L 0.43U pg/L 6.6U pg/L 1.9U pg/L 13U pg/L

Samples EB-04142010-RIG1-RZC and EB-04142010-RIG2-RZC were identified as equipment 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-04142010-RIG1-RZC	4/14/10	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.2 pg/L 9.2 pg/L 0.50 pg/L 0.32 pg/L 0.69 pg/L 0.39 pg/L 0.28 pg/L 0.20 pg/L 0.79 pg/L 0.30 pg/L 1.8 pg/L	No associated samples in this SDG
EB-04142010-RIG2-RZC	4/14/10	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	0.29 pg/L 0.39 pg/L 0.77 pg/L 2.5 pg/L 3.3 pg/L 1.6 pg/L 0.73 pg/L 4.2 pg/L 2.2 pg/L 0.71 pg/L 0.43 pg/L 6.6 pg/L 1.9 pg/L 13 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 G0D160472	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 G0D160472	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 G0D160472	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 G0D160472**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D160472	EB-04142010-RIG1-RZC EB-04142010-RIG2-RZC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0D160472	EB-04142010-RIG1-RZC EB-04142010-RIG2-RZC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D160472	EB-04142010-RIG1-RZC EB-04142010-RIG2-RZC	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 G0D160472**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D160472	EB-04142010-RIG1-RZC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.2U pg/L 9.2U pg/L 0.32U pg/L 0.69U pg/L 0.39U pg/L 0.28U pg/L 0.20U pg/L 0.79U pg/L 0.30U pg/L 1.8U pg/L	A	bl
G0D160472	EB-04142010-RIG2-RZC	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.29U pg/L 0.39U pg/L 0.77U pg/L 2.5U pg/L 1.6U pg/L 0.73U pg/L 4.2U pg/L 2.2U pg/L 0.71U pg/L 0.43U pg/L 6.6U pg/L 1.9U pg/L 13U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D160472**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265H21  
 SDG #: G0D160472  
 Laboratory: Test America

Date: 6/8/10  
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 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: 4/14/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/IX	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	N	did not certify
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	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	TW	EB = 1.2

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

Validated Samples:

1	EB-04142010-RIG1-RZC	W	11	0111312MB	21		31
2	EB-04142010-RIG2-RZC	V	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 PeCDD
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

**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:** 4/21/10 **Blank analysis date:** 5/1/10

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

Compound	Blank ID	Sample Identification				
		5X	1	2		
	0111312MB					
B	0.64	3.2				
C	0.83	4.15				
D	1.0	5		0.29/U		
E	1.3	6.5		0.39/U		
F	2.1	10.5	1.2/U	0.77/U		
G	6.6	33	9.2/U	2.5/U		
I	0.78	3.9	0.32/U	1.6/U		
J	0.79	3.95		0.73/U		
K	1.2	6	0.69/U	4.2/U		
L	1.2	6	0.39/U	2.2/U		
M	1.6	8	0.28/U	0.71/U		
N	2.2	11	0.20/U	0.43/U		
O	1.6	8	0.79/U	6.6/U		
P	1.7	8.5	0.30/U	1.9/U		
Q	3.5	17.5	1.8/U	13/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".

LDC #: 23265H21  
 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: 4/14/10

Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: None

Compound	Blank ID	Sample Identification			
	FB-04142010-RIG1-RZC	5X			
F	1.2	0.006			
G	9.2	0.046			
H	0.50	0.0025			
I	0.32	0.0016			
K	0.69	0.00345			
L	0.39	0.00195			
M	0.28	0.0014			
N	0.20	0.001			
O	0.79	0.00395			
P	0.30	0.0015			
Q	1.8	0.009			
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 Field Blanks

**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: 4/14/10

Field blank type: (circle one) Field Blank / Rinsate / Other:    Associated Samples:    None

Compound	Blank ID	Sample Identification	Sample Identification
	ER-04142010-BIG2-RZC	5X	
D	0.29	0.00145	
E	0.39	0.00195	
F	0.77	0.00385	
G	2.5	0.0125	
H	3.3	0.0165	
I	1.6	0.008	
J	0.73	0.00365	
K	4.2	0.021	
L	2.2	0.011	
M	0.71	0.00355	
N	0.43	0.00215	
O	6.6	0.033	
P	1.9	0.0095	
Q	13	0.065	
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 # 23-26121  
 SDG # 26121

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		All	No confirmation for 2,3,7,8-TCDF		N/A
		All	ZDFC results (R-flag)		N/A

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 15, 2010  
**LDC Report Date:** June 10, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D170485

**Sample Identification**

SSA06-03-1BPC  
SA129-3BPC  
SA129-4BPC  
SA175-5BPC  
SA175-8BPC\*\*

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 5 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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SSAO6-03-1BPC SA129-3BPC SA129-4BPC
0113206MB	4/23/10	OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.75 pg/g 0.22 pg/g 0.25 pg/g 0.35 pg/g 0.50 pg/g 0.16 pg/g 0.75 pg/g	SA175-5BPC SA175-8BPC**

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

Samples FB-04132010-RIG2-RZE (from SDG G0D150582) 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-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	SSAO6-03-1BPC
FB-04132010-RIG2-RZE	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.40 pg/L 0.65 pg/L 2.5 pg/L 0.66 pg/L 0.41 pg/L 0.53 pg/L 0.97 pg/L	SA129-3BPC SA129-4BPC SA175-5BPC SA175-8BPC**

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
SSAO6-03-1BPC	<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	28 (40-135) 32 (40-135) 16 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SA129-3BPC	<sup>13</sup> C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA129-4BPC	<sup>13</sup> C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA175-5BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-1,2,3,7,8-PeCDF	36 (40-135) 163 (40-135)	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF	J (all detects) UJ (all non-detects)	P
SA175-8BPC**	<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	25 (40-135) 31 (40-135) 22 (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
SA129-3BPC	1,2,3,4,7,8-HxCDF 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
SA129-4BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 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) J (all detects)	P
SA175-5BPC	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)	A
SA175-8BPC**	1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D170485	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 G0D170485	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**

No field duplicates were identified in this SDG.



**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D170485**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D170485	SSAO6-03-1BPC SA175-8BPC**	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D170485	SA129-3BPC SA129-4BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D170485	SA175-5BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D170485	SA129-3BPC	1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D170485	SA129-4BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D170485	SA175-5BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	A	Project Quantitation Limit (e)
G0D170485	SA175-8BPC**	1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Project Quantitation Limit (e)
G0D170485	SSAO6-03-1BPC SA129-3BPC SA129-4BPC SA175-5BPC SA175-8BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D170485	SSAO6-03-1BPC SA129-3BPC SA129-4BPC SA175-5BPC SA175-8BPC**	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  
G0D170485**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D170485**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23265121

SDG #: G0D170485

Laboratory: Test America

Stage 2B/4

Date: 4/9/10

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: 4/15/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No sp/ass'd - No Cond
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	W	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB-04132010-RIG2-R2E (G0D150582) FB-04072010 (G0D130519)

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

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

1	SSAO6-03-1BPC	11	0110455MB	21	31
2	SA129-3BPC	12	0113286MB	22	32
3	SA129-4BPC	13		23	33
4	SA175-5BPC	14		24	34
5	SA175-8BPC **	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 CHECKLIST

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"/>	

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 $\geq 2.5$ ?			/
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 PCDPE channel?			/
Was an acceptable lock mass recorded and monitored?			/
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?			/
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?			/
XII. System performance			
System performance was found to be acceptable.			/
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.			/
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.			/
Target compounds were detected in the field duplicates.			/
XV. Field blanks			
Field blanks were identified in this SDG.			/
Target compounds were detected in the field blanks.			/

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:



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

**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: Associated Samples: 1 (>5X)

Compound	Blank ID	5X	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, "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".  
Are all internal standard recoveries within the 40-135% criteria?  
Was the S/N ratio all internal standard peaks > 10?

Y N N/A  
Y N N/A

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	F	28 (40-135)	✓ N/A (F-F, O-R)
			H	32	
			I	16	
		2	F	35	(F, O)
		3	J	30	
		4	A	36	(H-N)
			C	163	
		5	F	25	
			H	31	
			I	32	
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 #: 232652  
 SDG #: 200000

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

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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
			<i>spike &gt; calib range</i>		
2			K.O. R	2	<del>None</del> (e)
3			H.K.O. R	3	↓
4			O.R	4	<del>None</del> / A (e)
5			I.N.K.L.O.P. R	5	↓
All			ZMPC results	nd	None (f)

Comments: See sample calculation verification worksheet for recalculations

**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:

$RRF = (A_x)(C_s)/(A_s)(C_x)$   
 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,  
 $A_s$  = Area of associated internal standard  
 $C_s$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	Average RRF (Initial)	RRF (CS3std)	RRF (CS3std)	%RSD	%RSD	RRF (CS3 std)	%RSD
1	1012 (105)	1/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.860	0.87	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	0.934	0.95	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	1.058	1.09	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	0.998	1.05	1.05	10.2	10.2	1.05	12.2
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.437	1.52	1.52	14.1	14.1	1.52	14.0
2	1012	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	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-OCDD)								
3	1012 (405)	4/21/10	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	0.98	4.33
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.021	1.04	1.04	8.03	8.03	1.04	8.97
			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	1.19	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	1.11	3.75
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.445	1.51	1.51	5.85	5.85	1.51	5.89

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

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 \* SDG #: SAI 0010

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### 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	294701DS	4/29/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.90	4.2	0.90	4.2
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.934	0.97	3.6	0.97	3.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.058	1.19	12.1	1.19	12.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.998	1.01	1.6	1.01	1.6
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.29	10.0	1.29	10.0
2	07M102DS	5/8/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	1.00	5.6	1.00	5.6
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.00	2.4	1.00	2.4
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.16	3.7	1.16	3.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.08	1.0	1.08	1.0
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.45	0.4	1.45	0.4
3	10M1052R	5/10/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	0.91	16.0	0.91	16.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-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 =  $100 \cdot \text{SSC}/\text{SA}$  Where: SSC = Spiked sample concentration  
 SA = Spike added

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0113286

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	23.2	NA	116	116	116	116						
1,2,3,7,8-PeCDD	100	/	116	/	116	116	116	116						
1,2,3,4,7,8-HxCDD	/	/	107	/	107	107	107	107						
1,2,3,4,7,8,9-HpCDF	/	/	126	/	126	126	126	126						
OCDF	200	↓	240	↓	120	120	120	120						

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>(N)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(N)</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 <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) HxCDFPE 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>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>3</sub> <sup>37</sup> ClO <sup>13</sup> 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>3</sub> <sup>37</sup> ClO 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 <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> <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 <sub>2</sub> 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> ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sup>13</sup> 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>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> <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> <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> 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) HxCDFPE 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 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>3</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>3</sub> <sup>37</sup> ClO 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>3</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> 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> 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>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>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</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>3</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>3</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) OCDFPE PFK						

(e) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 23265/21  
 SDG #: see cover

**VALIDATION FINDINGS WORKSHEET**  
 Sample Calculation Verification

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

**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?

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. S, A:  
 Conc. =  $\frac{42123900 (2000)}{15166900 (1.021) (10.55) (0.89)}$   
 = 57.9 ppb/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 14, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** GOD170489

**Sample Identification**

SSAP3-01-1BPC  
SA182-5BPC

## Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SSAP3-01-1BPC

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
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	SA182-5BPC

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

Samples EB04142010-RIG1-RZC and EB-04142010-RIG2-RZC (both from SDG GOD160472) were identified as equipment 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-04142010-RIG1-RZC	4/14/10	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 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.2 pg/L 9.2 pg/L 0.50 pg/L 0.32 pg/L 0.69 pg/L 0.39 pg/L 0.28 pg/L 0.20 pg/L 0.79 pg/L 0.30 pg/L 1.8 pg/L	All samples in SDG GOD170489
EB-04142010-RIG2-RZC	4/14/10	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	0.29 pg/L 0.39 pg/L 0.77 pg/L 2.5 pg/L 3.3 pg/L 1.6 pg/L 0.73 pg/L 4.2 pg/L 2.2 pg/L 0.71 pg/L 0.43 pg/L 6.6 pg/L 1.9 pg/L 13 pg/L	All samples in SDG GOD170489

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

Sample FB-04072010-RZC (from SDG GOD130519) 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-RZC	4/8/10	1,2,3,4,7,8-HxCDD	0.77 pg/L	All samples in SDG GOD170489
		1,2,3,6,7,8-HxCDD	0.74 pg/L	
		1,2,3,7,8,9-HxCDD	0.82 pg/L	
		1,2,3,4,6,7,8-HpCDD	4.2 pg/L	
		OCDD	37 pg/L	
		2,3,7,8-TCDF	0.57 pg/L	
		1,2,3,7,8-PeCDF	0.96 pg/L	
		2,3,4,7,8-PeCDF	0.67 pg/L	
		1,2,3,4,7,8-HxCDF	1.1 pg/L	
		1,2,3,6,7,8-HxCDF	0.96 pg/L	
		2,3,4,6,7,8-HxCDF	1.0 pg/L	
		1,2,3,7,8,9-HxCDF	1.0 pg/L	
		1,2,3,4,6,7,8-HpCDF	2.1 pg/L	
		1,2,3,4,7,8,9-HpCDF	1.5 pg/L	
		OCDF	6.7 pg/L	

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.

#### 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
SSAP3-01-1BPC	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
SA182-5BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D170489	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 G0D170489	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 G0D170489**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D170489	SSAP3-01-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D170489	SA182-5BPC	OCDF	J (all detects)	P	Project Quantitation Limit (e)
G0D170489	SSAP3-01-1BPC SA182-5BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D170489	SSAP3-01-1BPC SA182-5BPC	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  
G0D170489**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D170489**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D170489**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265J21  
 SDG #: G0D170489  
 Laboratory: Test America

Date: 6/8/10  
 Page: 1 of 2  
 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: 4/14/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No sp/ass'd - No Anal
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB-04072010-R2C (FOD130519) EB-04142010-R1F1-R2C, EB-04142010-R1F2-R2C (FOD1604)

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

Validated Samples:

1	SSAP3-01-1BPC	S	11	0110455MB	21		31
2	SA182-5BPC	V	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:







**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?

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

Sampling date: 4/8/10

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

Associated Samples: All (>5X)

Compound	Blank ID	5X	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, "U".

LDC #: 2321  
 SDG #: See below

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

Page: 1 of 1  
 Reviewer: g  
 2nd Reviewer: g

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
		1	<del>0.8</del> calib range	1	plots (e)
		2	↓	2	↓
		ad	ZMPE results (CA Flag)	ad	JK (E)

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 15, 2010  
**LDC Report Date:** June 10, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D170491

**Sample Identification**

SSAL5-04-1BPC  
SSAL4-03-1BPC  
SSAM4-01-1BPC\*\*

\*\*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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	All samples in SDG G0D170491

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
SSAL4-03-1BPC	1,2,3,7,8-PeCDF	0.72 pg/g	0.72U pg/g
SSAM4-01-1BPC** (5X)	2,3,7,8-TCDF 1,2,3,7,8-PeCDF	2.9 pg/g 5.2 pg/g	2.9U pg/g 5.2U 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 G0D170491

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
SSAL5-04-1BPC	<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	31 (40-135) 35 (40-135) 23 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAL4-03-1BPC	<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

### 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 compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D170491	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 G0D170491	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**

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D170491**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D170491	SSAL5-04-1BPC	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)
G0D170491	SSAL4-03-1BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D170491	SSAL5-04-1BPC SSAL4-03-1BPC SSAM4-01-1BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D170491	SSAL5-04-1BPC SSAL4-03-1BPC SSAM4-01-1BPC**	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  
G0D170491**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D170491	SSAL4-03-1BPC	1,2,3,7,8-PeCDF	0.72U pg/g	A	bl
G0D170491	SSAM4-01-1BPC** (5X)	2,3,7,8-TCDF 1,2,3,7,8-PeCDF	2.9U pg/g 5.2U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D170491**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/A

LDC #: 23265K21  
 SDG #: G0D170491  
 Laboratory: Test America

Date: 4/13/10  
 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: 4/15/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/IOX	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No sp/ass'd - No Anal
VII.	Laboratory control samples	A	CCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	TW	FB-04072010-RZD (G0D090441)

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

Validated Samples: \*\* 1 env/V

1	SSAL5-04-1BPC	5	11	0110455M3	21	31
2	SSAL4-03-1BPC		12		22	32
3	SSAM4-01-1BPC **	V	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.	✓			
Cooler temperature criteria was met.	✓			
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $< 30\%$ for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $\geq 10$ ?	✓			
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	✓			
Was a method blank performed for each matrix and concentration?	✓			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	✓			
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	✓			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		✓		
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	✓			
Was an LCS analyzed per extraction batch?	✓			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	✓			

VALIDATION FINDINGS CHECKLIST

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:









**VALIDATION FINDINGS WORKSHEET**  
**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:

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	(%RSD)	Average RRF (Initial)	(%RSD)	RRF (C53std)	(%RSD)	RRF (C53 std)	(%RSD)
1	1072 (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	0.87	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	0.95	13.9	0.95	13.8
			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	1.09	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	1.05	12.2	1.05	12.2
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.52	1.437	1.52	14.1	1.52	14.0	
2	1072	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	1.10	12.9	1.10	12.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDD)								
3	1072 (405)	4/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.98	0.945	0.98	0.98	4.44	0.98	4.33
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	1.04	1.021	1.04	1.04	8.03	1.04	8.97
			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	1.19	5.33	1.19	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	1.11	3.60	1.11	3.75
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.51	1.445	1.51	5.85	1.51	5.89	

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 =  $100 * (ave. RRF - RRF) / ave. RRF$       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	26AP10A115	4/26/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.92	7.0	0.92	7.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.932	0.93	0.1	0.93	0.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.058	1.15	8.9	1.15	8.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.998	1.06	6.2	1.06	6.2
			OCDF ( <sup>13</sup> C-OCDD)	1.437	1.54	7.2	1.54	7.2
2	27AP10A115	4/27/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.08		1.08	
			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	28AP10A115	5/6/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945	0.96	1.7	0.96	1.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	0.96	6.1	0.96	6.1
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.11	0.3	1.11	0.3
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.072	1.00	6.3	1.00	6.3
			OCDF ( <sup>13</sup> C-OCDD)	1.445	1.40	3.0	1.40	3.0

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

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

LCS ID: 0110455

Compound	Spike Added		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	NA	19.1	NA	96	96								
1,2,3,7,8-PeCDD	100	✓	103	✓	103	103								
1,2,3,4,7,8-HxCDD	✓	✓	95.5	✓	95	95								
1,2,3,4,7,8,9-HpCDF	200	✓	81.4	✓	81	81								
OCDF			198	✓	99	99								

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

(a) The following nucleic masses were used:

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

S = internal/recovery standard



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 16, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil/Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D170492

**Sample Identification**

EB-04152010-1-RZD  
SSAL8-01-1BPC  
SSAK8-02-1BPC

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
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	All soil samples in SDG G0D170492



Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0112250MB	4/22/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.1 pg/L 8.4 pg/L 0.92 pg/L 0.52 pg/L 0.50 pg/L 1.6 pg/L 1.0 pg/L 1.7 pg/L	All water samples in SDG G0D170492

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04152010-1-RZD	1,2,3,4,6,7,8-HpCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	7.3 pg/L 1.3 pg/L 0.33 pg/L 2.2 pg/L 0.62 pg/L 6.8 pg/L	7.3U pg/L 1.3U pg/L 0.33U pg/L 2.2U pg/L 0.62U pg/L 6.8U pg/L

Samples EB-04152010-2-RZD (from SDG G0D200558) and EB-04152010-1-RZD were identified as equipment 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-04152010-2-RZD	4/16/10	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.6 pg/L 2.9 pg/L 1.4 pg/L 2.6 pg/L 2.3 pg/L 9.8 pg/L 11 pg/L 45 pg/L 48 pg/L 29 pg/L 70 pg/L 56 pg/L 13 pg/L 7.6 pg/L 180 pg/L 58 pg/L 450 pg/L	All soil samples in SDG G0D170492

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04152010-1-RZD	4/16/10	2,3,7,8-TCDD 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 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	0.34 pg/L 0.41 pg/L 0.41 pg/L 7.3 pg/L 60 pg/L 0.19 pg/L 0.39 pg/L 1.3 pg/L 0.33 pg/L 0.63 pg/L 2.2 pg/L 0.62 pg/L 6.8 pg/L	All soil samples in SDG G0D170492

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

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 soil samples in SDG G0D170492

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
SSAL8-01-1BPC	<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	33 (40-135) 37 (40-135) 22 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

### XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAK8-02-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D170492	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 G0D170492	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 G0D170492**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D170492	SSAL8-01-1BPC	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)
G0D170492	SSAK8-02-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D170492	EB-04152010-1-RZD SSAL8-01-1BPC SSAK8-02-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D170492	EB-04152010-1-RZD SSAL8-01-1BPC SSAK8-02-1BPC	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  
G0D170492**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D170492	EB-04152010-1-RZD	1,2,3,4,6,7,8-HpCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	7.3U pg/L 1.3U pg/L 0.33U pg/L 2.2U pg/L 0.62U pg/L 6.8U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D170492**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D170492**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23265L21  
 SDG #: G0D170492  
 Laboratory: Test America

Stage 2B

Date: 4/16/10  
 Page: 11 of 11  
 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: 4/16/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration <del>NV</del>	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No sp/ass'd - no anal
VII.	Laboratory control samples	A	10 S
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	TW	EB=1 ZB04152010-2-R2D, FB04072010-1-R2D

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

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

(FOD 200558)  
 D = Duplicate  
 TB = Trip blank  
 EB = Equipment blank

(FOD 092441)

Validated Samples:

1	EB-04152010- <del>R2D</del> <del>XR2D</del> W	11	0112250MB	21		31	
2	SSAL8-01-1BPC S	12	0112236MB	22		32	
3	SSAK8-02-1BPC S	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**

**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: 4/16/10

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

Compound	Blank ID	5X	Sample Identification
	EB-04152010-2-RZD	5X	
A	1.6	0.008	
B	2.9	0.0145	
C	1.4	0.007	
D	2.6	0.013	
E	2.3	0.0115	
F	9.8	0.049	
G	11	0.055	
H	45	0.225	
I	48	0.24	
J	29	0.145	
K	70	0.35	
L	56	0.28	
M	13	0.065	
N	7.6	0.038	
O	180	0.9	
P	58	0.29	
Q	450	2.25	
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".









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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 16, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** GOD200427

**Sample Identification**

SSAK5-02-1BPC  
SSAK6-01-1BPC  
SSAK7-03-1BPC  
SSAL7-01-1BPC  
SSAI3-01-1BPC

## Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.



The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
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	All samples in SDG G0D200427

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
SSAL7-01-1BPC	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	1.3 pg/g 0.91 pg/g 2.5 pg/g	1.3U pg/g 0.91U pg/g 2.5U pg/g

Samples EB-04152010-2-RZD (from SDG G0D200558) and EB-04152010-1-RZD (from SDG G0D170492) were identified as equipment 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-04152010-2-RZD	4/16/10	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.6 pg/L 2.9 pg/L 1.4 pg/L 2.6 pg/L 2.3 pg/L 9.8 pg/L 11 pg/L 45 pg/L 48 pg/L 29 pg/L 70 pg/L 56 pg/L 13 pg/L 7.6 pg/L 180 pg/L 58 pg/L 450 pg/L	All samples in SDG G0D200427
EB-04152010-1-RZD	4/16/10	2,3,7,8-TCDD 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 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	0.34 pg/L 0.41 pg/L 0.41 pg/L 7.3 pg/L 60 pg/L 0.19 pg/L 0.39 pg/L 1.3 pg/L 0.33 pg/L 0.63 pg/L 2.2 pg/L 0.62 pg/L 6.8 pg/L	All samples in SDG G0D200427

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

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 G0D200427

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
SSAK5-02-1BPC	<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	33 (40-135) 37 (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
SSAK6-01-1BPC	<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	25 (40-135) 26 (40-135) 16 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK7-03-1BPC	<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	31 (40-135) 35 (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
SSAL7-01-1BPC	<sup>13</sup> C-OCDD	33 (40-135)	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAI3-01-1BPC	<sup>13</sup> C-OCDD	17 (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
SSAK6-01-1BPC	2,3,7,8-TCDF 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)	P
SSAI3-01-1BPC	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
SSAL7-01-1BPC	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 G0D200427	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 G0D200427	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 G0D200427**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D200427	SSAK5-02-1BPC SSAK6-01-1BPC SSAK7-03-1BPC	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)
G0D200427	SSAL7-01-1BPC SSAI3-01-1BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D200427	SSAK6-01-1BPC	2,3,7,8-TCDF 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)	P	Project Quantitation Limit (e)
G0D200427	SSAI3-01-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D200427	SSAL7-01-1BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0D200427	SSAK5-02-1BPC SSAK6-01-1BPC SSAK7-03-1BPC SSAL7-01-1BPC SSAI3-01-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D200427	SSAK5-02-1BPC SSAK6-01-1BPC SSAK7-03-1BPC SSAL7-01-1BPC SSAI3-01-1BPC	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  
G0D200427**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D200427	SSAL7-01-1BPC	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	1.3U pg/g 0.91U pg/g 2.5U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D200427**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D200427**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265M21  
 SDG #: G0D200427  
 Laboratory: Test America

Date: 4/8/10  
 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: 4/16/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No sp/ass'd - No Qual
VII.	Laboratory control samples	A	LCs
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
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	(FOD200558) FB-04072010-R2D (FOD090441), ZB-04152010-2-R2D ZB-04152010-1-R2D (FOD170492)

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

Validated Samples:

1	SSAK5-02-1BPC	5	11	0112236 MB	21		31
2	SSAK6-01-1BPC		12		22		32
3	SSAK7-03-1BPC		13		23		33
4	SSAL7-01-1BPC		14		24		34
5	SSAI3-01-1BPC		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:



LDC #: 23265M21  
 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: 4/16/10

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

Compound	Blank ID	Sample Identification				
	FR-04152010-2-RZD					5X
A	1.6					0.008
B	2.9					0.0145
C	1.4					0.007
D	2.6					0.013
E	2.3					0.0115
F	9.8					0.049
G	11					0.055
H	45					0.225
I	48					0.24
J	29					0.145
K	70					0.35
L	56					0.28
M	13					0.065
N	7.6					0.038
O	180					0.9
P	58					0.29
Q	450					2.25
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 #: 23265 M2  
 SDG #: DR Egan

**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 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
			<u>spds &gt; calibration</u>		
		<u>2</u>	<u>H.L.O.P.A</u>		<u>dots (e)</u>
		<u>5</u>	<u>H.F.K.L.O.P.A</u>		
		<u>4</u>	<u>No e.s.t.s - Troof confirmation</u>		<u>None</u>
		<u>MA</u>	<u>ZNPO results (R-tag)</u>	<u>ml</u>	<u>JK (K)</u>

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 16, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D200558

**Sample Identification**

EB-04152010-2-RZD

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0112250MB	4/22/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.1 pg/L 8.4 pg/L 0.92 pg/L 0.52 pg/L 0.50 pg/L 1.6 pg/L 1.0 pg/L 1.7 pg/L	All samples in SDG G0D200558

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04152010-2-RZD	1,2,3,4,6,7,8-HpCDD OCDD	9.8 pg/L 11 pg/L	9.8U pg/L 11U pg/L

Sample EB-04152010-2-RZD was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04152010-2-RZD	4/16/10	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.6 pg/L 2.9 pg/L 1.4 pg/L 2.6 pg/L 2.3 pg/L 9.8 pg/L 11 pg/L 45 pg/L 48 pg/L 29 pg/L 70 pg/L 56 pg/L 13 pg/L 7.6 pg/L 180 pg/L 58 pg/L 450 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 with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
EB-04152010-2-RZD	<sup>13</sup> C-1,2,3,7,8-PeCDD	39 (40-135)	1,2,3,7,8-PeCDD	J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D200558	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 G0D200558	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 G0D200558**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D200558	EB-04152010-2-RZD	1,2,3,7,8-PeCDD	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D200558	EB-04152010-2-RZD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D200558	EB-04152010-2-RZD	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  
G0D200558**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D200558	EB-04152010-2-RZD	1,2,3,4,6,7,8-HpCDD OCDD	9.8U pg/L 11U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D200558**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265N21  
 SDG #: G0D200558  
 Laboratory: Test America

Date: 6/7/10  
 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: 4/16/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	N	
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	S N	All EMPC results - JK(K)
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	TW	EB = 1

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

Validated Samples:

1	EB-04152010-2-RZD	N	11	0112250 MB	21		31	
2			12		22		32	
3			13		23		33	
4			14		24		34	
5			15		25		35	
6			16		26		36	
7			17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:



**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?

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

**Sampling date:** 4/16/10

**Field blank type:** (circle one) Field Blank / Rinsate / Other: EB Associated Samples: None

Compound	Blank ID	5X	Sample Identification
	EB-04152010-2-R7D	5X	
A	1.6	0.008	
B	2.9	0.0145	
C	1.4	0.007	
D	2.6	0.013	
E	2.3	0.0115	
F	9.8	0.049	
G	11	0.055	
H	45	0.225	
I	48	0.24	
J	29	0.145	
K	70	0.35	
L	56	0.28	
M	13	0.065	
N	7.6	0.038	
O	180	0.9	
P	58	0.29	
Q	450	2.25	
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".



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 22, 2010  
**LDC Report Date:** June 10, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D240497

**Sample Identification**

SSAL3-04-1BPC  
SSAL3-03-1BPC  
SSAM2-01-3BPC\*\*  
SSAL3-04-1BPCMS  
SSAL3-04-1BPCMSD  
SSAM2-01-3BPCMS  
SSAM2-01-3BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 7 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
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	SSAM2-01-3BPC**
0118247MB	4/28/10	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	0.038 pg/g 0.026 pg/g 0.10 pg/g 0.28 pg/g 0.17 pg/g 0.24 pg/g 0.18 pg/g 0.31 pg/g 0.13 pg/g 0.15 pg/g 0.064 pg/g 0.19 pg/g 0.15 pg/g 0.23 pg/g	SSAL3-04-1BPC SSAL3-03-1BPC

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

Samples FB-04072010-RZD (from SDG G0D090441) and FB-04132010-RIG2-RZE (from SDG G0D150582) 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-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	SSAL3-04-1BPC SSAL3-03-1BPC
FB-04132010-RIG2-RZE	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.40 pg/L 0.65 pg/L 2.5 pg/L 0.66 pg/L 0.41 pg/L 0.53 pg/L 0.97 pg/L	SSAM2-01-3BPC**

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the MS, 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
SSAL3-04-1BPC	<sup>13</sup> C-OCDD	36 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAM2-01-3BPC**	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	36 (40-135) 33 (40-135) 39 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,6,7,8-HpCDD	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
SSAL3-04-1BPC SSAL3-03-1BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

Sample	Compound	Finding	Criteria	Flag	A or P
SSAM2-01-3BPC**	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 G0D240497	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 G0D240497	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

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D240497**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D240497	SSAL3-04-1BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D240497	SSAM2-01-3BPC**	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,6,7,8-HpCDD	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D240497	SSAL3-04-1BPC SSAL3-03-1BPC	OCDF	J (all detects)	P	Project Quantitation Limit (e)
G0D240497	SSAM2-01-3BPC**	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (e)
G0D240497	SSAL3-04-1BPC SSAL3-03-1BPC SSAM2-01-3BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D240497	SSAL3-04-1BPC SSAL3-03-1BPC SSAM2-01-3BPC**	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  
G0D240497**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D240497**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23265021  
 SDG #: G0D240497  
 Laboratory: Test America

Stage 2B/4

Date: 4/9/10  
 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: 4/22/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	
VII.	Laboratory control samples	A	LOS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB-0407=2010-R2D (G0D090441), FB-0413=2010-R1G2-R226G0D15

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

Validated Samples:

M 50.15

1	SSAL3-04-1BPC	11	0118247MB	21	31
2	SSAL3-03-1BPC	12	0123335MB	22	32
3	SSAM2-01-3BPC **	13		23	33
4	SSAL3-04-1BPCMS	14		24	34
5	SSAL3-04-1BPCMSD	15		25	35
6	SSAM2-01-3BPCMS	16		26	36
7	SSAM2-01-3BPCMSD	17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

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

VALIDATION FINDINGS CHECKLIST

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



# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC #: 23265021

SDG #: Seccon

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 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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".

Y 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: 5/10 Blank analysis date: 5/10

Conc. units: pg Associated Samples: 3 (>5x)

Compound	Blank ID	Sample Identification
	<u>01233514B</u>	
<u>F</u>	<u>0.085</u>	
<u>F</u>	<u>0.60</u>	
<u>H</u>	<u>0.086</u>	
<u>D</u>	<u>0.079</u>	
<u>A</u>	<u>0.14</u>	

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".

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: 4/28/10 Blank analysis date: 5/15/10 Associated samples: 1-2 (bl) (75x)

Compound	Blank ID	5X	Sample Identification
	011824ZMR		
D	0.038	0.19	
E	0.026	0.13	
F	0.10	0.5	
G	0.28	1.4	
H	0.17	0.85	
I	0.24	1.2	
J	0.18	0.9	
K	0.31	1.55	
L	0.13	0.65	
M	0.15	0.75	
N	0.064	0.32	
O	0.19	0.95	
P	0.15	0.75	
Q	0.23	1.15	

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 #: 23265021  
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)

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: 1-2 (>5X)

Compound	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".

LDC #: 23265021  
 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:** 4/13/10  
**Field blank type:** (circle one) Field Blank / Rinsate / Other:

Associated Samples: 3 (>5X)

Compound	Blank ID	5X	Sample Identification
	EB-04132010-RIG2-RZE	5X	
E	0.40	0.002	
F	0.65	0.00325	
G	2.5	0.0125	
K	0.66	0.0033	
M	0.41	0.00205	
O	0.53	0.00265	
Q	0.97	0.00485	
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 REPORT NUMBER: [Number]

VALIDATION FINDINGS WORKSHEET  
 Matrix Spike/Matrix Spike Duplicates

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 a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.
- N N/A Was a MS/MSD analyzed every 20 samples of each matrix?
- Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		4/5	H	217 (79-137)	0 (79-137)	( ) ( )	1	No Qual
			O	44 (81-137)	59 (81-137)	( ) ( )		
			R	84 (75-141)	117 (75-141)	( ) ( )		
				( ) ( )	( ) ( )	( ) ( )		Some > 4x SA
				( ) ( )	( ) ( )	( ) ( )		and LCS in
	6/7		B	( ) ( )	149 (79-134)	35 (≤29)	3	No Qual
			D	( ) ( )	152 (73-147)	40 (≤36)		
			E	71 (80-143)	( ) ( )	34 (≤31)		
			F	73 (86-134)	231 (86-134)	73 (≤28)		
			G	( ) ( )	201 (80-137)	58 (≤32)		
			H	0 (79-137)	0 (79-137)	( ) ( )		
			I	87 (81-134)	( ) ( )	51 (≤27)		
			J	0.0 (76-132)	26 (76-132)	( ) ( )		
			K	257 (72-140)	( ) ( )	66 (≤32)		
			L	( ) ( )	162 (63-152)	45 (≤38)		
			N	43 (72-152)	55 (82-152)	( ) ( )		
			O	35 (81-137)	658 (81-137)	128 (≤33)		
			P	75 (79-139)	362 (79-139)	96 (≤35)		
			Q	0 (75-141)	1170 (75-141)	123 (≤45)		
			A	( ) ( )	0 (77-130)	200 (≤30)		(MS) LCS in
				( ) ( )	( ) ( )	( ) ( )		LCS in
				( ) ( )	( ) ( )	( ) ( )		Some conc > 4x SA



# VALIDATION FINDINGS WORKSHEET

## Compound Quantitation and Reported CRQLs

LDC #: 236502  
 SDG #: See early

Page: 1 of 1  
 Reviewer: g  
 2nd Reviewer: g

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
		1, 2	R - calib range	1-2	<del>plots</del> (R)
		3	H	3	↓
		all	ZNDC results	all	<del>plots</del> (R)

Comments: See sample calculation verification worksheet for recalculations



**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:

$RRF = (A_x)(C_s)/(A_s)(C_x)$   
 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,  
 $A_s$  = Area of associated internal standard  
 $C_s$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated		
				Average RRF (Initial)	Average RRF (Initial)	RRF (CS std)	RRF (CS std)	%RSD	%RSD	RRF (CS std)	%RSD	
1	12AZ (305)	3/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.92135	0.94963	0.94963	6.1085	6.1085	6.109	6.109	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.03870	1.04720	1.04720	7.89709	7.89709	4.897	4.897	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.09904	1.11730	1.11730	4.93827	4.93827	4.938	4.938	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	0.99684	1.05541	1.05541	5.31525	5.31525	5.315	5.315	
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.26224	1.32988	1.32988	9.39998	9.39998	9.400	9.400	
2	12AZ	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	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)									
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)									
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)									
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)									
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)									
			OCDF ( <sup>13</sup> C-OCDD)									

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

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

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA 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 =  $(A_x)(C_u) / (A_u)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_u$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_u$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated		Recalculated %D
					RRF (CC)	%D	RRF (CC)	%D	
1	1A-MX10375 -25	5/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	1.00105	8.7	1.00105	8.7	8.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.13915	9.7	1.13915	9.7	9.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.17849	7.2	1.17849	7.2	7.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99682	1.10758	11.1	1.10758	11.1	11.1
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.57414	20.0	1.57414	20.0	20.0
2	1A-MX10572	5/16/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	0.97	10.5	0.97	10.5	10.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)						
			OCDF ( <sup>13</sup> C-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      MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 6/7

Compound	Spike Added (ppg)		Sample Concentration (ppg)	Spiked Sample Concentration (ppg)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	21.7	19.6	6.7	30.3	0	109	109	0	0	200	200
1,2,3,7,8-PeCDD	108	97.8	13	126	157	104	105	149	147	35	22
1,2,3,4,7,8-HxCDD	↓	↓	5.4	81.8	75.1	71	71	72	72	4.9	8.5
1,2,3,4,7,8,9-HpCDF	217	196	9.6	177	438	75	75	262	350	96	85
OCDF			1000	754	5170	0	0	1170	1107	0	124

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.

lab used 50 R to calculate RPD due to different spike amount.

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

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

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

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

LCS ID: 0/23335

Compound	Spike Added (PS/g)		Spiked Sample Concentration (PS/g)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	200	NA	24.3	NA	121	121								
1,2,3,7,8-PeCDD	100		114		114	114								
1,2,3,4,7,8-HxCDD			115		115	115								
1,2,3,4,7,8,9-HpCDF			122		122	122								
OCDF	200		256		128	128								

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> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>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		423.7767	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <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> ClO <sub>2</sub>	TCDD (S)		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>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<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>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDPE							
[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>5</sub> O	HxCDF (S)						
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>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>9</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) The following nucleic masses were used:

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

S = internal/recovery standard



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 23, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D270515

**Sample Identification**

SSAI3-06-1BPC  
SSAJ2-01-1BPC

## Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.



The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	All samples in SDG G0D270515

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

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 G0D270515

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
SSAI3-06-1BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	24 (40-135) 20 (40-135) 22 (40-135) 19 (40-135) 20 (40-135) 18 (40-135) 20 (40-135) 20 (40-135) 18 (40-135)	All TCL compounds	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
SSAJ2-01-1BPC	2,3,7,8-TCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D270515	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 G0D270515	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 G0D270515**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D270515	SSAI3-06-1BPC	All TCL compounds	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D270515	SSAJ2-01-1BPC	2,3,7,8-TCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D270515	SSAI3-06-1BPC SSAJ2-01-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D270515	SSAI3-06-1BPC SSAJ2-01-1BPC	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  
G0D270515**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D270515**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 23265P21

VALIDATION COMPLETENESS WORKSHEET

SDG #: G0D270515

Stage 2B

Laboratory: Test America

Date: 6/7/10

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: 4/23/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	NW	
VI.	Matrix spike/Matrix spike duplicates	NW	No spl ass'd - No Anal
VII.	Laboratory control samples	A	CS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	NW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	NW	FB-04072010-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	SSAI3-06-1BPC	5	11	0123375MAB	21		31
2	SSAJ2-01-1BPC	✓	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:



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".

Y  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: 5/10/10 Blank analysis date: 5/15/10

Conc. units: ~~ppb~~ Associated Samples: NA (>5x)

Compound	Blank ID	Sample Identification
	013335413	
F	0.085	
G	0.60	
H	0.086	
I	0.079	
R	0.14	

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 #: 23265P21  
 SDG #: See Cover

### VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: 1 of 2  
 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: \_\_\_\_\_

Associated Samples: \_\_\_\_\_

All

( > S X )

Compound	Blank ID	5X	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

## 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)
		I	A	24	✓/UN/P (all)
			B	20	
			C	22	
			D	19	
			E	20	
			F	18	
			G	20	
			H	20	
			I	18	

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 # 236521  
SDG #: 210001

### VALIDATION FINDINGS WORKSHEET

Compound Quantitation and Reported CRQLs

Page: 1 of 1  
Reviewer: \_\_\_\_\_  
2nd Reviewer: R

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		<u>2</u>	<u>H.R. &gt; calib range</u>		<u>Notes/P (e)</u>
		<u>all</u>	<u>all RRF results</u>		<u>✓ (15)</u>

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 23, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D270522

**Sample Identification**

EB-04232010-RZE

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
5/16/10	1,2,3,4,7,8-HxCDD	20.1	0120219MB	1,2,3,4,7,8-HxCDD	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
0120219MB	4/30/10	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 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	0.65 pg/L 0.82 pg/L 1.9 pg/L 6.8 pg/L 0.67 pg/L 1.1 pg/L 2.8 pg/L 0.71 pg/L 0.89 pg/L 0.99 pg/L 0.34 pg/L 2.7 pg/L 3.8 pg/L	All samples in SDG G0D270522

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04232010-RZE	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.7 pg/L 21 pg/L 5.1 pg/L 2.0 pg/L 2.0 pg/L 7.3 pg/L 3.0 pg/L 16 pg/L	2.7U pg/L 21U pg/L 5.1U pg/L 2.0U pg/L 2.0U pg/L 7.3U pg/L 3.0U pg/L 16U pg/L

Sample EB-04232010-RZE was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04232010-RZE	4/23/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.7 pg/L 21 pg/L 3.8 pg/L 1.7 pg/L 5.1 pg/L 2.0 pg/L 2.0 pg/L 7.3 pg/L 3.0 pg/L 16 pg/L	No associated samples in this SDG

## VI. Matrix Spike/Matrix Spike Duplicates

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

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

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

**VIII. Regional Quality Assurance and Quality Control**

Not applicable.

**IX. Internal Standards**

All internal standard recoveries were within QC limits.

**X. Target Compound Identifications**

Raw data were not reviewed for this SDG.

**XI. Project Quantitation Limit**

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D270522	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 G0D270522	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 G0D270522**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D270522	EB-04232010-RZE	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D270522	EB-04232010-RZE	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 G0D270522**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D270522	EB-04232010-RZE	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.7U pg/L 21U pg/L 5.1U pg/L 2.0U pg/L 2.0U pg/L 7.3U pg/L 3.0U pg/L 16U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D270522**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265Q21  
 SDG #: G0D270522  
 Laboratory: Test America

Date: 4/7/10  
 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: 4/23/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/W	W	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	N	
VII.	Laboratory control samples	A	LCG
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	S, N	All 2MPE results (2/10/09) - JK(R)
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	EB=1

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

Validated Samples:

1	EB-04232010-RZE	W	11	0/20219MB	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:

**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".  
 Was a routine calibration performed at the beginning and end of each 12 hour period? N  
 Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled? N  
 Did all routine calibration standards meet the Ion Abundance Ratio criteria? N

#	Date	Standard ID	Compound	Finding %D (Limit: ≤30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications (C)
	5/16/10	14MY104D5-4	C	20.1		Bas	<u>J + Kelly P C C</u>

SDG #: See Cover

Blanks

Reviewer:   

2nd Reviewer:   

**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: 4/30/10

Blank analysis date: 5/16/10

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

Compound	Blank ID	5X	1	Sample Identification
	0.120219MR			
D	0.65	3.25		
E	0.82	4.1		
F	1.9	9.5	2.7/U	
G	6.8	34	21/U	
H	0.67	3.35		
I	1.1	5.5		
K	2.8	14	5.1/U	
L	0.71	3.55	2.0/U	
M	0.89	4.45	2.0/U	
N	0.99	4.95		
O	3.4	17	7.3/U	
P	2.7	13.5	3.0/U	
Q	3.8	19	16/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)

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

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

Sampling date: 4/23/10

Field blank type: (circle one) Field Blank / Rinsate / Other: EB Associated Samples: \_\_\_\_\_ None

Compound	Blank ID	Blank ID	Sample Identification
	EB-04232010-RZE	5X	
F	2.7	0.0135	
G	21	0.105	
H	3.8	0.019	
J	1.7	0.0085	
K	5.1	0.0255	
L	2.0	0.01	
M	2.0	0.01	
O	7.3	0.0365	
P	3.0	0.015	
Q	16	0.08	
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".



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 23, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D270529

**Sample Identification**

SSAI3-05-3BPC

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	All samples in SDG G0D270529

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

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	0.89 pg/L	All samples in SDG G0D270529
		1,2,3,7,8,9-HxCDD	1.5 pg/L	
		1,2,3,4,6,7,8-HpCDD	2.2 pg/L	
		OCDD	8.3 pg/L	
		1,2,3,4,7,8-HxCDF	1.4 pg/L	
		1,2,3,6,7,8-HxCDF	1.6 pg/L	
		2,3,4,6,7,8-HxCDF	1.5 pg/L	
		1,2,3,7,8,9-HxCDF	1.6 pg/L	
		1,2,3,4,6,7,8-HpCDF	1.3 pg/L	
		1,2,3,4,7,8,9-HpCDF	1.4 pg/L	
		OCDF	4.1 pg/L	

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.

#### 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
SSAI3-05-3BPC	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 G0D270529	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 G0D270529	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 G0D270529**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason (Code)</b>
G0D270529	SSAI3-05-3BPC	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (e)
G0D270529	SSAI3-05-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D270529	SSAI3-05-3BPC	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 G0D270529**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D270529**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265R21  
 SDG #: G0D270529  
 Laboratory: Test America

Date: 4/10  
 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: 4/23/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/TQ	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	SN	No sp/ass'd - No Anal
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	TW	FB-040(2010-R2D(G0D 90441))

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

Validated Samples:

1	SSAI3-05-3BPC	5	11	0123335MB	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 PeCDD
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:







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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 26, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D270573

**Sample Identification**

SSAI3-04-1BPC

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	All samples in SDG G0D270573



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

Sample EB-04262010-1-RZD (from SDG G0D280571) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04262010-1-RZD	4/26/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.1 pg/L 6.8 pg/L 7.6 pg/L 4.5 pg/L 1.3 pg/L 12 pg/L 5.3 pg/L 25 pg/L	All samples in SDG G0D270573

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

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 G0D270573

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.

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D270573	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 G0D270573	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 G0D270573**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason (Code)</b>
G0D270573	SSAI3-04-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D270573	SSAI3-04-1BPC	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 G0D270573**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D270573**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265S21  
 SDG #: G0D270573  
 Laboratory: Test America

Date: 4/7/10  
 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: 4/26/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ISV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	No sp/ass'd - No dual
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	All ZMPC results (2/10/09) - JK (K)
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	EB-042629104-1R2D(40D280571), FB-04072010R2D (40D090441)

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	SSAI3-04-1BPC	S	11	01233351XB	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 PeCDD
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

## VALIDATION FINDINGS WORKSHEET Blanks

**METHOD:** HRGC/HRMS PCB Congeners (EPA Method 1668)

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: 5/13/10  
Conc. units: PPA

Blank analysis date: 5/15/10

Associated samples: M (>5x)

Compound	Blank ID	Sample Identification
	0123335MB	
F	0.085	
G	0.60	
H	0.086	
O	0.079	
R	0.14	

Blank extraction date: \_\_\_\_\_  
Conc. units: \_\_\_\_\_

Blank analysis date: \_\_\_\_\_

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".







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

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

**Collection Date:** April 26, 2010

**LDC Report Date:** June 9, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 4

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D270574

**Sample Identification**

SSAJ2-02-3BPC

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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).

## 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.

## 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
5/16/10	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	38.9	SSAJ2-02-3BPC	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
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	All samples in SDG G0D270574

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

Sample EB-04262010-1-RZD (from SDG G0D280571) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04262010-1-RZD	4/26/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.1 pg/L 6.8 pg/L 7.6 pg/L 4.5 pg/L 1.3 pg/L 12 pg/L 5.3 pg/L 25 pg/L	All samples in SDG G0D270574

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

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 G0D270574

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.

#### X. Target Compound Identifications

All target compound identifications were within validation 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
SSAJ2-02-3BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D270574	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 G0D270574	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

## XII. System Performance

The system performance was acceptable.

## 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 G0D270574**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D270574	SSAJ2-02-3BPC	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P	Routine calibration (%D) (c)
G0D270574	SSAJ2-02-3BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D270574	SSAJ2-02-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D270574	SSAJ2-02-3BPC	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 G0D270574**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D270574**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D270574**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23265T21  
 SDG #: G0D270574  
 Laboratory: Test America

Stage 2B4

Date: 6/9/10  
 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: 4/26/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ <del>IX</del>	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	No sp/ass'd - No anal
VII.	Laboratory control samples	A	LES
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SWA	
X.	Target compound identifications	A	
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-04072010-R2D(40D090411), 2B-04262010-1-R2D(40D090411)

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

Validated Samples:

1	SSAJ2-02-3BPC	S	11	0123375 MB	21		31
2			12		22		32
3			13		23		33
4			14		24		34
5			15		25		35
6			16		26		36
7			17		27		37
8			18		28		38
9			19		29		39
10			20		30		40

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

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. GC/MS Instrument performance check</b>				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Initial calibration</b>				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input 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"/>	

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 $\geq 2.5$ ?			/
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 PCDPE channel?			/
Was an acceptable lock mass recorded and monitored?			/
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?			/
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?			/
XII. System performance			
System performance was found to be acceptable.			/
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.			/
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.			/
Target compounds were detected in the field duplicates.			/
XV. Field blanks			
Field blanks were identified in this SDG.			/
Target compounds were detected in the field blanks.			/

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:













**VALIDATION FINDINGS WORKSHEET**  
**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:

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	(C.S) std)	Average RRF (Initial)	(C.S) std)	RRF (C.S) std)	%RSD	RRF (C.S) std)	%RSD
1	1EATZ (305)	3/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.94963	0.92135	0.94963	6.1085	6.109	6.1085	6.109
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.04720	1.03870	1.04720	4.89709	4.897	4.89709	4.897
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.11730	1.09904	1.11730	4.93827	4.938	4.93827	4.938
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.05541	0.99684	1.05541	5.31525	5.315	5.31525	5.315
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.32988	1.26224	1.32988	9.39998	9.400	9.39998	9.400
2	1EATZ	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	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)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDD)								

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

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

LDC #: 232652  
SDG #: See cover

Page: 6 of 7  
Reviewer: [Signature]  
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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where:    ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 A<sub>x</sub> = Area of compound,      A<sub>s</sub> = Area of associated internal standard  
 C<sub>x</sub> = Concentration of compound,      C<sub>s</sub> = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	1A11/0375	5/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	1.00105	8.7	1.00105	8.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.13915	9.7	1.13915	9.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.17829	7.2	1.17829	7.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.10758	11.1	1.10758	11.1
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.51414	20.0	1.51414	20.0
2	1A11/0375	5/17/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	0.997	10.5	0.997	10.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)			13.2		13.1
			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	1A11/0375	5/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	1.02878	11.7	1.02878	11.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.18429	14.0	1.18429	14.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.19487	8.7	1.19487	8.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.10722	10.7	1.10722	10.7
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.49485	18.4	1.49485	18.4

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.



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

(a) The following nuclidic masses were used:

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

S = internal/recovery standard



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 26, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Water  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D280571

**Sample Identification**

EB-04262010-1-RZD

## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.



The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.
- U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.
- 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
5/6/10	1,2,3,6,7,8-HxCDF	20.1	0123391MB	1,2,3,6,7,8-HxCDF	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
0123391MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.3 pg/L 4.1 pg/L 2.7 pg/L 1.0 pg/L 2.1 pg/L 3.1 pg/L 4.4 pg/L 3.4 pg/L	All samples in SDG G0D280571

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04262010-1-RZD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	2.1 pg/L 6.8 pg/L 7.6 pg/L 4.5 pg/L 12 pg/L 5.3 pg/L	2.1U pg/L 6.8U pg/L 7.6U pg/L 4.5U pg/L 12U pg/L 5.3U pg/L

Sample EB-04262010-1-RZD was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04262010-1-RZD	4/26/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2.1 pg/L 6.8 pg/L 7.6 pg/L 4.5 pg/L 1.3 pg/L 12 pg/L 5.3 pg/L 25 pg/L	No associated samples in this SDG

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D280571	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 G0D280571	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 G0D280571**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D280571	EB-04262010-1-RZD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D280571	EB-04262010-1-RZD	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 G0D280571**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D280571	EB-04262010-1-RZD	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	2.1U pg/L 6.8U pg/L 7.6U pg/L 4.5U pg/L 12U pg/L 5.3U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D280571**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265U21  
 SDG #: G0D280571  
 Laboratory: Test America

Date: 6/5/10  
 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: 4/26/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	W	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	All ZMP C (Q flag) - JK (K)
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	ZB = 1

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

Validated Samples:

1	EB-04262010-1-RZD	W	11	0/2339/MP	21		31
2			12				32
3			13				33
4			14				34
5			15				35
6			16				36
7			17				37
8			18				38
9			19				39
10			20				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:









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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 27, 2010  
**LDC Report Date:** June 8, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D280586

**Sample Identification**

SSAK3-05-1BPC  
SSAK8-05-1BPC

## Introduction

This data review covers 2 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0123335MB	5/3/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.085 pg/g 0.60 pg/g 0.086 pg/g 0.079 pg/g 0.14 pg/g	All samples in SDG GOD280586

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

Samples 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 G0D280586

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

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

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
SSAK8-05-1BPC	<sup>13</sup> C-OCDD	37 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## 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
SSAK3-05-1BPC	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D280586	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 G0D280586	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 G0D280586**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D280586	SSAK8-05-1 BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D280586	SSAK3-05-1 BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	A	Project Quantitation Limit (e)
G0D280586	SSAK3-05-1 BPC SSAK8-05-1 BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D280586	SSAK3-05-1 BPC SSAK8-05-1 BPC	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  
G0D280586**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D280586**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D280586**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265V21  
 SDG #: G0D280586  
 Laboratory: Test America

Date: 4/27/10  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

Validation Area			Comments
I.	Technical holding times	A	Sampling dates: 4/27/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/IC	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No spl assid - No Anal
VII.	Laboratory control samples	A	LES
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	N	
XV.	Field blanks	W	FB-04272010-R2D (401090441)

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

Validated Samples:

1	SSAK3-05-1BPC	S	11	0123335MB	21		31
2	SSAK8-05-1BPC	V	12				32
3			13				33
4			14				34
5			15				35
6			16				36
7			17				37
8			18				38
9			19				39
10			20				40

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?
- Y 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: 5/3/12 Blank analysis date: 5/15/12

Conc. units: pg/l Associated Samples: M (75X)

Compound	Blank ID	Sample Identification
[Shaded]	012333SMIB	
F	0.085	
G	0.60	
H	0.086	
O	0.079	
R	0.14	

Blank extraction date: \_\_\_\_\_ Blank analysis date: \_\_\_\_\_

Conc. units: \_\_\_\_\_ Associated Samples: \_\_\_\_\_

Compound	Blank ID	Sample Identification
[Shaded]		

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)

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 Associated Samples: All

Compound	Blank ID	5X	Sample Identification
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 > 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		2	F	37	( 70-135 ) <i>Y/A (S.R.)</i>

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

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

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

Notes:

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

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

Y N N/A N/A Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound?  
 Y N N/A 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
		1	0.8 > calc based	/	detg/P(e)
		all	ZNFC RESULTS (R)	all	VF (R)

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 28, 2010  
**LDC Report Date:** June 9, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 4  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** GOD300450  
**Sample Identification**  
SSAN6-07-1BPC



## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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).

## 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.

## 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
5/17/10	1,2,3,4,7,8-HxCDD	20.2	SSAN6-07-1BPC	1,2,3,4,7,8-HxCDD	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
0124370MB	5/4/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.49 pg/g 0.26 pg/g 0.13 pg/g 0.28 pg/g 0.15 pg/g 0.35 pg/g 0.18 pg/g 0.80 pg/g	All samples in SDG G0D300450

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

Sample FB-04072010-RZC (from SDG G0D130519) 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-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	All samples in SDG G0D300450

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
SSAN6-07-1BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	22 (40-135) 27 (40-135) 30 (40-135) 31 (40-135) 32 (40-135) 29 (40-135) 33 (40-135) 33 (40-135) 28 (40-135)	All TCL compounds	J (all detects) UJ (all non-detects)	P
0124370MB	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	36 (40-135) 37 (40-135) 38 (40-135) 33 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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.

## 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
SSAN6-07-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D300450	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 G0D300450	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

## **XII. System Performance**

The system performance was acceptable.

## **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 G0D300450**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D300450	SSAN6-07-1BPC	1,2,3,4,7,8-HxCDD	J+ (all detects)	P	Routine calibration (%D) (c)
G0D300450	SSAN6-07-1BPC	All TCL compounds	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D300450	SSAN6-07-1BPC	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)
G0D300450	SSAN6-07-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D300450	SSAN6-07-1BPC	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 G0D300450**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D300450**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage ~~2B~~ 4

LDC #: 23265W21  
 SDG #: G0D300450  
 Laboratory: Test America

Date: 4/9/10  
 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: 4/28/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ <del>DC</del>	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	No sp/ass'd
VII.	Laboratory control samples	A	LOG
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB-04072010-RZC(G0D130519)

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

Validated Samples:

1	SSAN6-07-1BPC	S	11	012437 MB	21		31	
2			12		22		32	
3			13		23		33	
4			14		24		34	
5			15		25		35	
6			16		26		36	
7			17		27		37	
8			18		28		38	
9			19		29		39	
10			20		30		40	

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



LDC #: 23265WB1  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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

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

VALIDATION FINDINGS CHECKLIST

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:



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".  
 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: 5/4/10 Blank analysis date: 5/15/10

Conc. units: pg/g Associated samples: All (>5X)

Compound	Blank ID	Sample Identification																			
		5X																			
	0124370MR																				
F	0.12	5X																			
G	0.49		0.6																		
H	0.26		2.45																		
I	0.13		1.3																		
K	0.28		0.65																		
L	0.15		1.4																		
O	0.35		0.75																		
P	0.18		1.75																		
Q	0.80		0.9																		
			4																		

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

**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?

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

Sampling date: 4/8/10

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

Compound	Blank ID	Sample Identification				
		5X				
	FB-04072010-R7C					
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, "U".







**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:

$$RRF = (A_x)(C_s)/(A_s)(C_x)$$

$$\text{average RRF} = \text{sum of the RRFs}/\text{number of standards}$$

$$\%RSD = 100 * (S/X)$$

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	(C.3) std)	Average RRF (Initial)	(C.3) std)	RRF (C.3) std)	%RSD	RRF (C.3) std)	%RSD
1	1e2z (305)	3/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.94963	0.92135	0.94963	0.94963	6.1085	0.94963	6.109
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.04720	1.03870	1.04720	1.04720	4.89709	1.04720	4.897
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.11730	1.09904	1.11730	1.11730	4.93827	1.11730	4.938
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.05541	0.99684	1.05541	1.05541	5.31535	1.05541	5.315
			OCDF ( <sup>13</sup> C-OCDF)	1.26224	1.32988	1.26224	1.32988	1.32988	9.39998	1.32988	9.400
2	1e2z	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	1.10	1.29	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	1e2z	5/11/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.017	1.06	1.017	1.06	1.06	5.39	1.06	5.70
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.991	1.01	0.991	1.01	1.01	5.17	1.01	4.90
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.155	1.21	1.155	1.21	1.21	7.50	1.21	7.75
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.044	1.12	1.044	1.12	1.12	8.97	1.12	8.81
			OCDF ( <sup>13</sup> C-OCDF)	1.574	1.66	1.574	1.66	1.66	8.84	1.66	8.72

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

LDC #: 23265W2  
 SDG #: 23265W2

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 2nd Reviewer: R

### 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	1745/1025	5/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	1.02878	11.7	1.02878	11.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.18429	14.0	1.18429	14.0
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.19487	8.7	1.19487	8.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.10322	10.7	1.10322	10.7
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.49485	18.4	1.49485	18.4
2	1745/1025	5/17/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.017	1.04	2.4	1.04	2.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.991	1.07	7.7	1.07	7.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.155	1.21	4.5	1.21	4.5
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.044	1.12	7.2	1.12	7.2
			OCDF ( <sup>13</sup> C-OCDD)	1.544	1.61	4.1	1.61	4.1
3	1745/1025	5/18/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.15	5.3	1.15	5.3
			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)					

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 #: 23265W7  
 SDG #: 21 con

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

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

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

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

RPD =  $100 \cdot \text{LCS} - \text{LCS}2 / (\text{LCS} + \text{LCS}2)$

LCS = Laboratory control sample percent recovery

LCS2 = Laboratory control sample duplicate percent recovery

LCS ID: 0124370

Compound	Spike Added		Spiked Sample Concentration		LCS		LCS2		LCS2		RPD	
	LCS	LCS2	LCS	LCS2	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	20.0	23NA	23.1	NA	116	116						
1,2,3,7,8-PeCDD	100	/	117	/	117	117						
1,2,3,4,7,8-HxCDD	/	/	105	/	105	105						
1,2,3,4,7,8,9-HpCDF	/	/	89.7	/	90	90						
OCDF	200	/	248	/	124	124						

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>9</sub> <sup>35</sup> Cl <sub>3</sub> O	TCDF	4	407.7618	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFPE		479.7165	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	NCDPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
369.8919		M+4	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFPE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDF (S)						
		385.8610	M+2	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF (S)						
	389.8156	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFPE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(e) The following nuclidic masses were used:

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

S = internal/recovery standard



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 28, 2010  
**LDC Report Date:** June 8, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** GOD300454

**Sample Identification**

RSAQ3-1BPC  
RSAQ3-2BPC  
SA169-4BPC  
RSAQ3-2BPCMS  
RSAQ3-2BPCMSD

## Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

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



## **I. Technical Holding Times**

All technical holding time requirements were met.

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

## **II. HRGC/HRMS Instrument Performance Check**

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all homologues. The chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomer was less than or equal to 25% .

## **III. Initial Calibration**

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

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

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

## **IV. Routine Calibration (Continuing)**

Routine calibration was performed at the required frequencies.

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

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

## **V. Blanks**

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0124370MB	5/4/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.49 pg/g 0.26 pg/g 0.13 pg/g 0.28 pg/g 0.15 pg/g 0.35 pg/g 0.18 pg/g 0.80 pg/g	All samples in SDG G0D300454

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

Samples EB-04282010-RZB (from SDG G0D300598) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04282010-RZB	4/28/10	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	3.8 pg/L 5.2 pg/L 5.2 pg/L 7.5 pg/L 3.7 pg/L 16 pg/L 12 pg/L 2.7 pg/L 2.0 pg/L 43 pg/L 9.9 pg/L 76 pg/L	All samples in SDG G0D300454

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

Samples FB-04062010-RZB (from SDG G0D120488) 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-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	All samples in SDG G0D300454

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the 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
RSAQ3-1BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	32 (40-135) 35 (40-135) 36 (40-135) 31 (40-135) 38 (40-135) 36 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
RSAQ3-2BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,6,7,8-HxCDD	37 (40-135) 35 (40-135) 38 (40-135) 39 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 2,3,7,8-TCDF	J (all detects) UJ (all non-detects)	P
SA169-4BPC	<sup>13</sup> C-2,3,7,8-TCDF <sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,7,8-HxCDF <sup>13</sup> C-1,2,3,6,7,8-HxCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	25 (40-135) 31 (40-135) 35 (40-135) 30 (40-135) 32 (40-135) 31 (40-135) 26 (40-135) 22 (40-135) 12 (40-135)	All TCL compounds	J (all detects) UJ (all non-detects)	P
0124370MB	<sup>13</sup> C-2,3,7,8-TCDD <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	36 (40-135) 37 (40-135) 38 (40-135) 33 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SA169-4BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG GOD300454	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 G0D300454	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 G0D300454**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D300454	RSAQ3-1BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 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	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D300454	RSAQ3-2BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 2,3,7,8-TCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D300454	SA169-4BPC	All TCL compounds	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D300454	SA169-4BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D300454	RSAQ3-1BPC RSAQ3-2BPC SA169-4BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D300454	RSAQ3-1BPC RSAQ3-2BPC SA169-4BPC	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  
G0D300454**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D300454**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada**  
**Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D300454**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23265X21  
 SDG #: G0D300454  
 Laboratory: Test America

Date: 6/9/10  
 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: 4/28/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	W	
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	FB0406210-R2B (EOD 120488), ZB0428210-R2 C EOD 30059

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	RSAQ3-1BPC	5	11	0124370MB	21		31
2	RSAQ3-2BPC		12		22		32
3	SA169-4BPC		13		23		33
4	RSAQ3-2BPCMS		14		24		34
5	RSAQ3-2BPCMSD		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: 5/4/10 Blank analysis date: 5/15/10

Conc. units: pg/g Associated samples: All (>5X)

Compound	Blank ID	Sample Identification				
[Shaded]	0124370MB	5X				
F	0.12	0.6				
G	0.49	2.45				
H	0.26	1.3				
I	0.13	0.65				
K	0.28	1.4				
L	0.15	0.75				
O	0.35	1.75				
P	0.18	0.9				
Q	0.80	4				

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

LDC #: 23265X21  
 SDG #: See Cover

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

Page: 1 of 9  
 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: 4/28/10

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

Compound	Blank ID	5X	Sample Identification	
	ER04282010-RZR			
F	3.8	0.019		
G	5.2	0.026		
H	5.2	0.026		
I	7.5	0.0375		
J	3.7	0.0185		
K	16	0.08		
L	12	0.06		
M	2.7	0.0135		
N	2.0	0.01		
O	43	0.215		
P	9.9	0.0495		
Q	76	0.38		
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".



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

N N/A

Y (N/A)

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

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

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		A/S	F	145 (86-134)	( )	31 (≤28)	2	No Qual
			G	220 (80-137)	( )	63 (≤32)		
			H	( )	9.4 (79-137)	33 (≤30)		
			I	136 (81-134)	( )	( )		
			K	218 (70-140)	( )	49 (≤32)		
			L	155 (63-152)	( )	( )		
			N	154 (70-152)	( )	( )		
			O	329 (81-137)	23 (81-137)	75 (≤33)		
			P	189 (79-139)	( )	( )		
			R	166 (75-141)	161 (75-141)	( )		
				( )	( )	( )		(MSD only in)
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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LDC #: 232652  
 SDG #: SEC 2004

**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".

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 (if)
		1	A	32	✓ M/P (A-B-F-H-N)
			B	35	
			C	36	
			D	37	
			E	38	
			H	36	
		2	A	37	✓ M/P (A-E-H)
			B	35	
			D	38	
			F	39	
		3	A	35	✓ M/P (All)
			B	37	
			C	35	
			D	30	
			E	32	
			F	31	
			G	26	
			H	22	
			I	12	

	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**  
**Internal Standards**

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (CI)
		<u>012437MB</u>	<del>A</del> B	<u>36</u> ( <u>40-135</u> )	<u>Y/N/A (A,B,F,O)</u>
			<del>B</del> D	<u>37</u> ( )	
			<del>E</del>	<u>38</u> ( )	
			<del>F</del>	<u>33</u> ( )	
		<u>4 (MS)</u>	A	<u>35</u> ( )	<u>No Qual</u>
			B	<u>34</u> ( )	
			D	<u>35</u> ( )	
			F	<u>38</u> ( )	
		<u>5 (MSD)</u>	A	<u>37</u> ( )	
			B	<u>34</u> ( )	
			C	<u>38</u> ( )	
			D	<u>34</u> ( )	
			E	<u>30</u> ( )	
			F	<u>33</u> ( )	
			<del>G</del>	<u>27</u> ( )	
			<del>H</del>	<u>25</u> ( )	
				<u>12</u> ( )	<u>Y</u>

	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-HpCDD
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 #: 23265/1  
SDG #: 23265/1

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

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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      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
		3	H. K. L. O. P. & > carb. range	3	↳ (TS/P (E))
		All	EMPE results (R)	ml	↳ K (K)

Comments: See sample calculation verification worksheet for recalculations