



**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 22, 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 June 3, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 23307:**

**SDG #**

**Fraction**

G0D200500, G0D220587, G0D230609  
G0D280587, G0D300598, G0E010426  
G0E010431, G0E030471, G0E030477  
G0E030478, G0E040578, G0E040582  
G0E050582, G0E050610, G0E060621  
G0E100428, G0E140423

Dioxins/Dibenzofurans

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

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

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

Sincerely,

Erlinda T. Rauto  
Operations Manager/Senior Chemist

LDC #23307 (Tronox LLC-Northgate, Henderson NV / Tronox PCS)

Stage 2B/4

LDC	SDG#	DATE REC'D	(3) DATE DUE	Dioxins (8290)		W		S		W		S		W		S		W		S		W		S	
				W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
Matrix: Water/Soil																									
A	G0D200500	06/03/10	06/24/10	0	3																				
A	G0D200500	06/03/10	06/24/10	0	1																				
B	G0D220587	06/03/10	06/24/10	0	3																				
C	G0D230609	06/03/10	06/24/10	0	3																				
C	G0D230609	06/03/10	06/24/10	0	1																				
D	G0D280587	06/03/10	06/24/10	0	2																				
D	G0D280587	06/03/10	06/24/10	0	1																				
E	G0D300598	06/03/10	06/24/10	1	0																				
F	G0E010426	06/03/10	06/24/10	0	2																				
G	G0E010431	06/03/10	06/24/10	0	2																				
G	G0E010431	06/03/10	06/24/10	0	2																				
H	G0E030471	06/03/10	06/24/10	0	1																				
H	G0E030471	06/03/10	06/24/10	0	1																				
I	G0E030477	06/03/10	06/24/10	0	3																				
J	G0E030478	06/03/10	06/24/10	0	2																				
K	G0E040578	06/03/10	06/24/10	0	4																				
L	G0E040582	06/03/10	06/24/10	0	3																				
M	G0E050582	06/03/10	06/24/10	0	1																				
M	G0E050582	06/03/10	06/24/10	0	1																				
N	G0E050610	06/03/10	06/24/10	1	0																				
O	G0E060621	06/03/10	06/24/10	0	5																				
P	G0E100428	06/03/10	06/24/10	0	2																				
Q	G0E140423	06/03/10	06/24/10	0	1																				

EDD CHECKLIST

LDC #: 23307

SDG #: G0D200500, G0D220587, G0D230609, G0D280587, G0D300598  
G0E010426, G0E010431, G0E030471, G0E030477, G0E030478  
G0E040578, G0E040582, G0E050582, G0E050610, G0E060621  
G0E100428, G0E140423

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_LDC23307_062210.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 #23307**

Dioxins/Dibenzofurans

**LDC**

**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 17, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D200500

**Sample Identification**

RSAK5-9BPC  
SSAJ6-01-7BPC  
SSAJ6-01-10BPC\*\*  
SA127-6BPC  
SA127-6BPCMS  
SA127-6BPCMSD

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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
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	SA127-6BPC
0116296MB	4/26/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.20 pg/g 0.17 pg/g 1.2 pg/g 0.33 pg/g 0.45 pg/g 1.5 pg/g 1.6 pg/g	RSAK5-9BPC
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	SSAJ6-01-7BPC SSAJ6-01-10BPC**

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

Samples EB-04152010-1-RZD (from SDG G0D170492) and EB-04152010-2-RZD (from SDG G0D200558) 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
EB04152010-1-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 G0D200500

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB04152010-2-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 G0D200500

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 G0D200500

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

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAJ6-01-7BPC	<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	38 (40-135) 30 (40-135) 27 (40-135) 29 (40-135) 32 (40-135) 22 (40-135) 21 (40-135) 18 (40-135)	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAJ6-01-10BPC**	<sup>13</sup> C-1,2,3,7,8-PeCDF <sup>13</sup> C-1,2,3,7,8-PeCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	37 (40-135) 33 (40-135) 39 (40-135) 34 (40-135) 24 (40-135)	1,2,3,7,8-PeCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,6,7,8-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
SSAJ6-01-7BPC	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
SSAJ6-01-10BPC**	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
SA127-6BPC	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 G0D200500	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 G0D200500	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 G0D200500**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D200500	SSAJ6-01-7BPC	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D200500	SSAJ6-01-10BPC**	1,2,3,7,8-PeCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D200500	SSAJ6-01-7BPC	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)
G0D200500	SSAJ6-01-10BPC**	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D200500	SA127-6BPC	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)
G0D200500	RSAK5-9BPC SSAJ6-01-7BPC SSAJ6-01-10BPC** SA127-6BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D200500	RSAK5-9BPC SSAJ6-01-7BPC SSAJ6-01-10BPC** SA127-6BPC	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  
G0D200500**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0D200500**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D200500**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson  
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23307A21  
SDG #: G0D200500  
Laboratory: Test America

Stage 2B/A

Date: 6/16/10  
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>4/16/10</u>
II.	HRGC/HRMS Instrument performance check	A
III.	Initial calibration	A
IV.	Routine calibration/ <del>SW</del>	A
V.	Blanks	SW
VI.	Matrix spike/Matrix spike duplicates	SW
VII.	Laboratory control samples	A <u>LCS</u>
VIII.	Regional quality assurance and quality control	N
IX.	Internal standards	SW
X.	Target compound identifications	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 <u>FB-04012010-R2D(F0D09041), ZB-04152010-R2D</u> <u>ZB-04152010-2-R2D(F0D200500) (F0D170492)</u>

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

Validated Samples:

1	1	RSAK5-9BPC	11	<u>0116296MB</u>	21		31
2	2	SSAJ6-01-7BPC	12	<u>0118247MB</u>	22		32
3	3	SSAJ6-01-10BPC **	13	<u>0113286MB</u>	23		33
4	4	SA127-6BPC	14		24		34
5	5	SA127-6BPCMS	15		25		35
6	6	SA127-6BPCMSD	16		26		36
7	7		17		27		37
8	8		18		28		38
9	9		19		29		39
10	10		20		30		40

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

LDC #: 23307A/1  
 SDG #: 21 CON

VALIDATION FINDINGS CHECKLIST

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

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

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



LDC #: 23307A21  
 SDG #: See above

VALIDATION FINDINGS CHECKLIST

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC #: 23307A2

SDG #: Recon

**VALIDATION FINDINGS WORKSHEET**

**Blanks**

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

**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: 4/23/10 Blank analysis date: 5/30/10

Conc. units: PS/g Associated Samples: 7 (>5)

Compound	Blank ID	Sample Identification
	0113286M13	
<u>F</u>	0.75	
<u>H</u>	0.22	
<u>I</u>	0.25	
<u>K</u>	0.35	
<u>O</u>	0.50	
<u>P</u>	0.16	
<u>R</u>	0.75	

Blank extraction date: 4/24/10 Blank analysis date: 5/14/10

Conc. units: PS/g Associated Samples: 1 (>5x)

Compound	Blank ID	Sample Identification
	0113286M13	
<u>C</u>	0.20	
<u>E</u>	0.17	
<u>F</u>	1.2	
<u>H</u>	0.33	
<u>I</u>	0.45	
<u>O</u>	1.5	
<u>R</u>	1.6	

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





LDC #: 23307A21  
 SDG #: See Cover

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

Page: 1 of 9  
 Reviewer: \_\_\_\_\_  
 2nd Reviewer: R

**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:** 4/16/10

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

Compound	Blank ID	Sample Identification
	EB-04152010-2-BZD	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".







**VALIDATION FINDINGS WORKSHEET**  
Internal Standards

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

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

(N) 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 (if)																																													
		<u>2</u>	<u>B</u>	<u>38</u>	<u>N/A (A-F, I-R)</u>																																													
			<u>C</u>	<u>30</u>																																														
			<u>D</u>	<u>27</u>																																														
			<u>E</u>	<u>29</u>																																														
			<u>F</u>	<u>32</u>																																														
			<u>G</u>	<u>22</u>																																														
			<u>H</u>	<u>21</u>																																														
			<u>I</u>	<u>18</u>																																														
		<u>3</u>	<u>C</u>	<u>37</u>	<u>(B, F, I, O, R)</u>																																													
			<u>D</u>	<u>33</u>																																														
			<u>G</u>	<u>39</u>																																														
			<u>H</u>	<u>34</u>																																														
			<u>I</u>	<u>24</u>																																														
		<u>5 (MS)</u>	<u>G</u>	<u>32</u>	<u>Not Qual</u>																																													
			<u>H</u>	<u>38</u>																																														
			<u>I</u>	<u>31</u>																																														
		<u>6 (MSD)</u>	<u>E</u>	<u>34</u>																																														
		<u>6 (MSD)</u>	<u>G</u>	<u>34</u>																																														
<table border="1"> <thead> <tr> <th colspan="2">Internal Standards</th> <th>Check Standard Used</th> <th>Internal Standards</th> <th>Check Standard Used</th> </tr> </thead> <tbody> <tr> <td>A.</td> <td><sup>13</sup>C-2,3,7,8-TCDF</td> <td></td> <td>I.</td> <td><sup>13</sup>C-OCDD</td> </tr> <tr> <td>B.</td> <td><sup>13</sup>C-2,3,7,8-TCDD</td> <td></td> <td>K.</td> <td><sup>13</sup>C-1,2,3,4-TCDD</td> </tr> <tr> <td>C.</td> <td><sup>13</sup>C-1,2,3,7,8-PeCDF</td> <td></td> <td>L.</td> <td><sup>13</sup>C-1,2,3,7,8,9-HxCDD</td> </tr> <tr> <td>D.</td> <td><sup>13</sup>C-1,2,3,7,8-PeCDD</td> <td></td> <td>M.</td> <td></td> </tr> <tr> <td>E.</td> <td><sup>13</sup>C-1,2,3,4,7,8-HxCDF</td> <td></td> <td>N.</td> <td></td> </tr> <tr> <td>F.</td> <td><sup>13</sup>C-1,2,3,6,7,8-HxCDD</td> <td></td> <td>O.</td> <td></td> </tr> <tr> <td>G.</td> <td><sup>13</sup>C-1,2,3,4,6,7,8-HpCDF</td> <td></td> <td>P.</td> <td></td> </tr> <tr> <td>H.</td> <td><sup>13</sup>C-1,2,3,4,6,7,8-HpCDD</td> <td></td> <td></td> <td></td> </tr> </tbody> </table>						Internal Standards		Check Standard Used	Internal Standards	Check Standard Used	A.	<sup>13</sup> C-2,3,7,8-TCDF		I.	<sup>13</sup> C-OCDD	B.	<sup>13</sup> C-2,3,7,8-TCDD		K.	<sup>13</sup> C-1,2,3,4-TCDD	C.	<sup>13</sup> C-1,2,3,7,8-PeCDF		L.	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	D.	<sup>13</sup> C-1,2,3,7,8-PeCDD		M.		E.	<sup>13</sup> C-1,2,3,4,7,8-HxCDF		N.		F.	<sup>13</sup> C-1,2,3,6,7,8-HxCDD		O.		G.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		P.		H.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			
Internal Standards		Check Standard Used	Internal Standards	Check Standard Used																																														
A.	<sup>13</sup> C-2,3,7,8-TCDF		I.	<sup>13</sup> C-OCDD																																														
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H.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD																																																	



**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  
 $\%RSD = 100 \cdot (S/X)$

$A_i$  = Area of compound,  
 $A_s$  = Area of associated internal standard  
 $C_i$  = 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)	Average RRF (Initial)	RRF (C3 std)	RRF (C3 std)	%RSD	%RSD	RRF (C3 std)	%RSD	
1	1EATZ (305)	2/1/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.92135	0.94463	0.94463	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	4.89709	4.89709	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	4.93867	4.93867	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-OCDF)	1.26224	1.26224	1.32988	1.32988	9.39998	9.39998	9.400	9.400	
2	1EATZ (905)	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-OCDF)									
3	1EATZ (905)	2/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.936	0.936	0.99	0.99	5.24	5.24	5.16	5.16	
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.033	1.033	1.05	1.05	7.77	7.77	7.82	7.82	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.112	1.16	1.16	4.39	4.39	4.38	4.38	
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.957	0.957	1.02	1.02	9.00	9.00	9.08	9.08	
			OCDF ( <sup>13</sup> C-OCDF)	1.347	1.347	1.47	1.47	9.57	9.57	9.61	9.61	

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**  
**Matrix Spike/Matrix Spike Duplicates Results Verification**

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

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

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

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

MS/MSD samples: 5/6

Compound	Spike Added (Pg/S)		Sample Concentration (Pg/g)		Spiked Sample Concentration (Pg/g)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD		Recalculated RPD	
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc	Reported	Recalc	Reported	MSDR	Reported	MSDR
2,3,7,8-TCDD	200	187	18		36.4	38.2	102	92	118	108	4.7		4.8	
1,2,3,7,8-PeCDD	100	<del>187</del>	7	56	159	161	109	103	118	<del>128</del>	0.81		1.2	
1,2,3,4,7,8-HxCDD	↓	↓	38		166	150	132	112	123	120	1.0		1.0	
1,2,3,4,7,8,9-HpCDF	260	187	2300		1820	3160	0.0	0.0	59%	500	0.0		2.53	
OCDF	200	187	12000		8630	1110	0.0	0.0	0.0	0.0	0.0		0.5	

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



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(e)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(e)</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 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>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</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> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>9</sub> F <sub>13</sub>	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) TCDD (S) HxCDFE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 M+4 LOCK	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</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> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</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> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>9</sub> F <sub>17</sub>	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) 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>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>9</sub> F <sub>13</sub>	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HxCDFE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</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> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub> C <sub>9</sub> F <sub>17</sub>	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDFE PFK				

(e) The following nuclidic masses were used:

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

S = internal/recovery standard





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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 21, 2010  
**LDC Report Date:** June 14, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D220587

**Sample Identification**

SSAL7-02-1BPC  
SSAK8-03-1BPC  
SSAL5-03-1BPC  
SSAK8-03-1BPCMS  
SSAK8-03-1BPCMSD

## 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.
- 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	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	38.9	SSAL5-03-1BPC	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
0116296MB	4/26/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.20 pg/g 0.17 pg/g 1.2 pg/g 0.33 pg/g 0.45 pg/g 1.5 pg/g 1.6 pg/g	SSAL7-02-1BPC SSAL5-03-1BPC
0138437MB	5/18/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.046 pg/g 0.25 pg/g 0.044 pg/g 0.066 pg/g	SSAK8-03-1BPC

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
SSAL7-02-1BPC	2,3,7,8-TCDF	0.22 pg/g	0.22U 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 G0D220587

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) were not within QC limits for some compounds, the 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
SSAK8-03-1BPC	<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

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
SSAL5-03-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D220587	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 G0D220587	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 G0D220587**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D220587	SSAL5-03-1BPC	1,2,3,4,6,7,8-HpCDD	J+ (all detects)	P	Routine calibration (%D) (c)
G0D220587	SSAK8-03-1BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D220587	SSAL5-03-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D220587	SSAL7-02-1BPC SSAK8-03-1BPC SSAL5-03-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D220587	SSAL7-02-1BPC SSAK8-03-1BPC SSAL5-03-1BPC	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  
G0D220587**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D220587	SSAL7-02-1BPC	2,3,7,8-TCDF	0.22U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D220587**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23307B21  
 SDG #: G0D220587  
 Laboratory: Test America

Date: 4/19/10  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 4/21/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	No spike added - No Anal [Signature]
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB-040T2010-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	SSAL7-02-1BPC	11	0116296MB	21	31
2	SSAK8-03-1BPC	12	0138137MB	22	32
3	SSAL5-03-1BPC	13		23	33
4	SSAK8-03-1BPCMS	14		24	34
5	SSAK8-03-1BPCMSD	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".

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

Blank extraction date: 4/20/10 Blank analysis date: 5/14/10

Conc. units: PPB Associated Samples: 1, 3 (68)

Compound	Blank ID	Sample Identification
	<u>0116296M B</u>	<u>3 &gt; 5x</u>
<u>C</u>	<u>0.20</u>	
<u>E</u>	<u>0.17</u>	
<u>F</u>	<u>1.2</u>	
<u>H</u>	<u>0.33</u>	<u>0.27/1</u>
<u>J</u>	<u>0.45</u>	
<u>O</u>	<u>1.5</u>	<u>(30)</u>
<u>R</u>	<u>1.6</u>	<u>(120)</u>

Blank extraction date: 5/18/10 Blank analysis date: 5/20/10

Conc. units: PPB Associated Samples: 2 (-5x)

Compound	Blank ID	Sample Identification
	<u>0138437M B</u>	
<u>F</u>	<u>0.046</u>	
<u>G</u>	<u>0.25</u>	
<u>O</u>	<u>0.044</u>	
<u>R</u>	<u>0.066</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".











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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 21, 2010  
**LDC Report Date:** June 17, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D230609

**Sample Identification**

SSAK7-01-10BPC\*\*  
SSAK7-01-9BPC  
RSAJ5-8BPC  
RSAJ5-7BPC

\*\*Indicates sample underwent Stage 4 review

## Introduction

This data review covers 4 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
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	SSAK7-01-10BPC** SSAK7-01-9BPC
0130380MB	5/10/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.41 pg/g 1.6 pg/g 0.65 pg/g 1.3 pg/g 0.67 pg/g 0.28 pg/g 2.0 pg/g 0.82 pg/g 4.0 pg/g	RSAJ5-8BPC RSAJ5-7BPC

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 G0D230609

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
SSAK7-01-10BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	31 (40-135) 26 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P
SSAK7-01-9BPC	<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	36 (40-135) 32 (40-135) 36 (40-135) 21 (40-135) 18 (40-135) 13 (40-135)	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 1,2,3,4,7,8-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
RSAJ5-8BPC	<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	27 (40-135) 30 (40-135) 21 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
RSAJ5-7BPC	<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	35 (40-135) 37 (40-135) 15 (40-135) 16 (40-135) 11 (40-135)	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

### 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
SSAK7-01-10BPC**	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAK7-01-9BPC RSAJ5-8BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
RSAJ5-7BPC	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 G0D230609	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 G0D230609	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 G0D230609**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D230609	SSAK7-01-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D230609	SSAK7-01-9BPC	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 1,2,3,4,7,8-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)
G0D230609	RSAJ5-8BPC	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)
G0D230609	RSAJ5-7BPC	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D230609	SSAK7-01-10BPC**	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0D230609	SSAK7-01-9BPC RSAJ5-8BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D230609	RSAJ5-7BPC	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)
G0D230609	SSAK7-01-10BPC** SSAK7-01-9BPC RSAJ5-8BPC RSAJ5-7BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D230609	SSAK7-01-10BPC** SSAK7-01-9BPC RSAJ5-8BPC RSAJ5-7BPC	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 G0D230609**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D230609**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B/4

LDC #: 23307C21  
 SDG #: G0D230609  
 Laboratory: Test America

Date: 6/17/10  
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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: 4/21/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/IDV	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No ass'd spl - No Anal.
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	TW	
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	TW	FB-04072010-R2D(400090441)

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	SSAK7-01-10BPC ** S	11	0118247MB	21		31	
2	SSAK7-01-9BPC	12	0130380MB	22		32	
3	RSAJ5-8BPC	13		23		33	
4	RSAJ5-7BPC	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 $\geq 10$ ?	<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"/>	

LDC #: 2330T C21  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:









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

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

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	H	81 (40-135)	✓ (F-F-R)
			I	86 ( )	
		2	D	36 ( )	
			E	33 ( )	(B-F-K-R)
			F	36 ( )	
			G	21 ( )	
			H	18 ( )	
			I	13 ( )	
			J	( )	
		3	G	38 27 ( )	(F-F, D-R)
			H	30 ( )	
			I	21 ( )	
			J	( )	
		4	E	35 ( )	(C-F, K-R)
			F	37 ( )	
			G	15 ( )	
			H	16 ( )	
			I	11 ( )	
			J	( )	
		Internal Standards	Check Standard Used	Internal Standards	Check Standard Used
A.		<sup>13</sup> C-2,3,7,8-TCDF		I. <sup>13</sup> C-OCDD	
B.		<sup>13</sup> C-2,3,7,8-TCDD		K. <sup>13</sup> C-1,2,3,4-TCDD	
C.		<sup>13</sup> C-1,2,3,7,8-PeCDF		L. <sup>13</sup> C-1,2,3,7,8,9-HxCDD	
D.		<sup>13</sup> C-1,2,3,7,8-PeCDD		M.	
E.		<sup>13</sup> C-1,2,3,4,7,8-HxCDF		N.	
F.		<sup>13</sup> C-1,2,3,6,7,8-HxCDD		O.	
G.		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		P.	
H.		<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD			



**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,  
 $X$  = 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)	RRF (C.3 std)	Average RRF (Initial)	RRF (C.3 std)	%RSD	%RSD	RRF (C.3 std)	%RSD	
1	10AZ (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	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	4.89709	4.897	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	4.93847	4.938	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	5.31525	5.315	1.05541	5.315	
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.32988	1.26224	1.32988	9.39998	9.400	1.32988	9.400	
2	10AZ	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	1.29	1.10	1.29	
			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 #: 23307021  
 SDG #: 210001

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: 2

**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 = continuing calibration RRF  
 $\text{RRF} = (A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	17M/10305	5/15/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.99473	8.0	0.99473	8.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.18974	14.5	1.18974	14.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.17180	6.6	1.17180	6.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.09231	9.6	1.09231	9.6
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.46157	15.8	1.46157	15.8
2	17M/10305	5/17/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.96173	4.4	0.96173	4.4
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.15010	10.7	1.15010	10.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.17867	7.2	1.17867	7.2
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.04098	4.4	1.04098	4.4
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.47589	16.9	1.47589	16.9
3	19M/0502	5/19/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.13	4.0	1.13	4.0
			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.



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>12</sub> <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> ClO <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>6</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>6</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	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>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>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	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> ClO <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	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)	[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>6</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	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>7</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 27, 2010  
**LDC Report Date:** June 17, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0D280587

**Sample Identification**

SSAK8-04-4BPC  
SSAK8-04-5BPC\*\*  
SSAK8-04-5BPC\_FD

\*\*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
0126326MB	5/6/10	2,3,7,8-TCDD 1,2,3,4,7,8-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	0.030 pg/g 0.021 pg/g 0.030 pg/g 0.043 pg/g 0.28 pg/g 0.061 pg/g 0.032 pg/g 0.046 pg/g 0.063 pg/g 0.081 pg/g 0.035 pg/g 0.046 pg/g 0.11 pg/g 0.079 pg/g 0.21 pg/g	All samples in SDG G0D280587

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 G0D280587

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
SSAK8-04-4BPC	<sup>13</sup> C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which 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
SSAK8-04-4BPC	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 G0D280587	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 G0D280587	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 SSAK8-04-5BPC\*\* and SSAK8-04-5BPC\_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
	SSAK8-04-5BPC**	SSAK8-04-5BPC_FD				
2,3,7,8-TCDD	1.3	1.5	-	0.2 ( $\leq 0.52$ )	-	-
1,2,3,7,8-PeCDD	3.5	4.3	-	0.8 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDD	2.0	2.3	-	0.3 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDD	4.1	4.9	-	0.8 ( $\leq 2.6$ )	-	-
1,2,3,7,8,9-HxCDD	3.3	4.3	-	1 ( $\leq 2.6$ )	-	-
1,2,3,4,6,7,8-HpCDD	11	13	-	2 ( $\leq 2.6$ )	-	-
OCDD	8.9	14	-	5.1 ( $\leq 5.2$ )	-	-
2,3,7,8-TCDF	33	41	22 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDF	53	64	19 ( $\leq 50$ )	-	-	-
2,3,4,7,8-PeCDF	30	35	15 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8-HxCDF	80	100	22 ( $\leq 50$ )	-	-	-
1,2,3,6,7,8-HxCDF	60	71	17 ( $\leq 50$ )	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK8-04-5BPC**	SSAK8-04-5BPC_FD				
2,3,4,6,7,8-HxCDF	14	18	25 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDF	11	15	31 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDF	140	200	35 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	86	120	33 ( $\leq 50$ )	-	-	-
OCDF	340	570	51 ( $\leq 50$ )	-	J (all detects)	A



**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D280587**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D280587	SSAK8-04-4BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0D280587	SSAK8-04-4BPC	OCDF	J (all detects)	P	Project Quantitation Limit (e)
G0D280587	SSAK8-04-4BPC SSAK8-04-5BPC** SSAK8-04-5BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D280587	SSAK8-04-4BPC SSAK8-04-5BPC** SSAK8-04-5BPC_FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D280587	SSAK8-04-5BPC** SSAK8-04-5BPC_FD	OCDF	J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG  
G0D280587**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D280587**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23307D21  
 SDG #: GOD280587  
 Laboratory: Test America

Stage 2B/4

Date: 6/15/10  
 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/100	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No ass'd sp! - No eval
VII.	Laboratory control samples	A	109
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	W	D = 2+3
XV.	Field blanks	W	FB-07072010-RZD (GOD090441)

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:

S

1	SSAK8-04-4BPC	W	11	0126326MB	21	31
2	SSAK8-04-5BPC	xx	12		22	32
3	SSAK8-04-5BPC_FD	↓	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 #: 23307021  
 SDG #: Sei Coner

VALIDATION FINDINGS CHECKLIST

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

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

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

LDC #: 23307021  
 SDG #: See down

VALIDATION FINDINGS CHECKLIST

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

VIII. Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>		
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>		
IX. Internal standards			
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>		
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	<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 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#: 23307D21  
 SDG#: See Cover

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

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

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

Y N NA  
Y N NA

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

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	3				
A	1.3	1.5		0.2	( ≤0.52)	
B	3.5	4.3		0.8	( ≤2.6)	
C	2.0	2.3		0.3	( ≤2.6)	
D	4.1	4.9		0.8	( ≤2.6)	
E	3.3	4.3		1	( ≤2.6)	
F	11	13		2	( ≤2.6)	
G	8.9	14		5.1	( ≤5.2)	
H	33	41	22			
I	53	64	19			
J	30	35	15			
K	80	100	22			
L	60	71	17			
M	14	18	25			
N	11	15	31			
O	140	200	35			
P	86	120	33			
Q	340	570	51			<u>Identified (fd)</u>

V:\FIELD DUPLICATES\23307D21.wpd

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

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

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

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated		
				Average RRF (Initial)	(RRF)	Average RRF (Initial)	(RRF)	(C.S. std)	(C.S. std)	%RSD	%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	6.1085	6.109	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	4.89709	4.897	4.897	
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.11730	1.09904	1.11730	4.93847	4.93847	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	1.05541	0.99684	1.05541	5.31525	5.31525	5.315	5.315	
			OCDF ( <sup>13</sup> C-OCDF)	1.26224	1.32988	1.26224	1.32988	9.39998	9.39998	9.400	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	1.29	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-OCDF)									
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)									
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)									
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)									
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)									
			OCDF ( <sup>13</sup> C-OCDF)									

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

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

LDC #: 23307021  
 SDG #: 2110001

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 Reviewer: \_\_\_\_\_  
 2nd Reviewer: \_\_\_\_\_

**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 = continuing calibration RRF  
 $\text{RRF} = (A_x)(C_s) / (A_s)(C_x)$   
 $A_x$  = 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	<del>17M/10132</del>	5/18/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.96862	5.1	0.96862	5.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.07593	3.6	1.07593	3.6
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.24646	12.9	1.24646	12.9
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.10973	11.3	1.10973	11.3
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.50116	18.9	1.50116	18.9
2	<del>18M/10050</del>	5/18/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.17	7.8	1.17	7.8
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			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.



Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte																				
1	303.9016 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 M+2 M+2 M+2 LOCK	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>4</sub> O C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>4</sub> O C <sub>12</sub> H <sub>5</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>3</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>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>1</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>0</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>0</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>9</sub> F <sub>13</sub>	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE 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+2 M+2 M+4 M+4 M+4 LOCK	C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>7</sub> O C <sub>12</sub> H <sub>5</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>1</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>0</sub> <sup>35</sup> Cl <sub>2</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) 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 M+2 M+2 M+2 LOCK	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>5</sub> <sup>35</sup> Cl <sub>2</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>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>1</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>0</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>9</sub> F <sub>13</sub>	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HxCDDPE 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 M+4 M+4 LOCK	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>8</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>6</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>5</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>1</sub> <sup>35</sup> Cl <sub>2</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 M+4 M+4 M+4 LOCK	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub> C <sub>9</sub> F <sub>17</sub>	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDDPE 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 28, 2010

**LDC Report Date:** June 14, 2010

**Matrix:** Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0D300598

**Sample Identification**

EB04282010-RZB



## Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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.

Sample EB04282010-RZB 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
EB04282010-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	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 G0D300598	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 G0D300598	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 G0D300598**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason (Code)</b>
G0D300598	EB04282010-RZB	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D300598	EB04282010-RZB	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D300598**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D300598**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23307E21  
 SDG #: G0D300598  
 Laboratory: Test America

Stage 2B

Date: 6/10/10  
 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	A
VI.	Matrix spike/Matrix spike duplicates	N client spiked.
VII.	Laboratory control samples	A 10s
VIII.	Regional quality assurance and quality control	N
IX.	Internal standards	A
X.	Target compound identifications	N
XI.	Compound quantitation and CRQLs	N All 2MPC results - OK (K)
XII.	System performance	N
XIII.	Overall assessment of data	A
XIV.	Field duplicates	N
XV.	Field blanks	N 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	EB04282010-RZB W	11	0124412MB	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:





**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 14, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0E010426

**Sample Identification**

SSAQ4-04-1BPC  
SSAO4-03-3BPC

## 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
0126326MB	5/6/10	2,3,7,8-TCDD 1,2,3,4,7,8-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	0.030 pg/g 0.021 pg/g 0.030 pg/g 0.043 pg/g 0.28 pg/g 0.061 pg/g 0.032 pg/g 0.046 pg/g 0.063 pg/g 0.081 pg/g 0.035 pg/g 0.046 pg/g 0.11 pg/g 0.079 pg/g 0.21 pg/g	All samples in SDG G0E010426

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

Samples FB-04072010-RZC (from SDG G0D130519) and FB-04062010-RZB (from SDG G0D120488) 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	SSAQ4-03-3BPC
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	SSAQ4-04-1BPC

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.

## 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
SSAQ4-04-1BPC	1,2,3,4,6,7,8-HpCDF OCDF 2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
SSAO4-03-3BPC	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 G0E010426	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 G0E010426	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 G0E010426**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason (Code)</b>
G0E010426	SSAQ4-04-1BPC	1,2,3,4,6,7,8-HpCDF OCDF 2,3,7,8-TCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E010426	SSAO4-03-3BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E010426	SSAQ4-04-1BPC SSAO4-03-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E010426	SSAQ4-04-1BPC SSAO4-03-3BPC	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  
G0E010426**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E010426**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23307F21  
 SDG #: G0E010426  
 Laboratory: Test America

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

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

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

Validation Area		Comments
I.	Technical holding times	★ Sampling dates: 4/28/10
II.	HRGC/HRMS Instrument performance check	★
III.	Initial calibration	★
IV.	Routine calibration/ICV	★
V.	Blanks	W
VI.	Matrix spike/Matrix spike duplicates	W No ass'd sp - No Anal.
VII.	Laboratory control samples	★ LCC
VIII.	Regional quality assurance and quality control	N
IX.	Internal standards	★
X.	Target compound identifications	N
XI.	Compound quantitation and CRQLs	W N
XII.	System performance	N
XIII.	Overall assessment of data	★
XIV.	Field duplicates	N
XV.	Field blanks	W FB 040720-R2C(FOD 130519), FB 062010-R2B(FOD 10488) <del>SSAO 2B0482010-R2B(FOD 300598) - Q</del>

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	SSAQ4-04-1BPC	B	11	0126326MB	21	31
2	SSAO4-03-3BPC	C	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: 2 (>5X)

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".





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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 29, 2010  
**LDC Report Date:** June 18, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E010431

**Sample Identification**

SSAO4-05-1BPC\*\*  
SSAQ3-01-1BPC\*\*  
SSAQ3-01-2BPC  
SSAO4-05-1BPC\_FD  
SSAO4-05-1BPCMS  
SSAO4-05-1BPCMSD  
SSAQ3-01-1BPCMS  
SSAQ3-01-1BPCMSD

\*\*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.
- 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
0134189MB	5/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.41 pg/g 0.12 pg/g 0.11 pg/g 0.15 pg/g 0.14 pg/g 0.35 pg/g	SSAQ3-01-1BPC**
0126326MB	5/6/10	2,3,7,8-TCDD 1,2,3,4,7,8-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	0.030 pg/g 0.021 pg/g 0.030 pg/g 0.043 pg/g 0.28 pg/g 0.061 pg/g 0.032 pg/g 0.046 pg/g 0.063 pg/g 0.081 pg/g 0.035 pg/g 0.046 pg/g 0.11 pg/g 0.079 pg/g 0.21 pg/g	SSAO4-05-1BPC** SSAQ3-01-2BPC SSAO4-05-1BPC_FD

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

Samples FB-04072010-RZC (from SDG G0D130519) and FB-04062010-RZB (from SDG G0D120488) 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	SSAO4-05-1BPC** SSAO4-05-1BPC_FD

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04062010-RZB	4/6/10	1,2,3,7,8,9-HxCDD	0.68 pg/L	SSAQ3-01-1BPC**
		1,2,3,4,6,7,8-HpCDD	2.5 pg/L	SSAQ3-01-2BPC
		OCDD	6.2 pg/L	
		2,3,7,8-TCDF	2.7 pg/L	
		1,2,3,4,7,8-HxCDF	1.4 pg/L	
		1,2,3,6,7,8-HxCDF	0.82 pg/L	
		1,2,3,7,8,9-HxCDF	0.94 pg/L	
		1,2,3,4,6,7,8-HpCDF	1.8 pg/L	
		1,2,3,4,7,8,9-HpCDF	1.2 pg/L	
		OCDF	4.4 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 MS/MSD percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

## 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
SSAO4-05-1BPC** SSAO4-05-1BPC_FD	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAQ3-01-1BPC**	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAQ3-01-2BPC	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 G0E010431	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 G0E010431	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 SSAO4-05-1BPC\*\* and SSAO4-05-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAO4-05-1BPC**	SSAO4-05-1BPC_FD				
2,3,7,8-TCDD	4.1	4.9	18 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDD	11	13	-	2 ( $\leq 2.6$ )	-	-
1,2,3,4,7,8-HxCDD	6.4	7.0	-	0.6 ( $\leq 2.6$ )	-	-
1,2,3,6,7,8-HxCDD	13	14	7 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDD	13	15	14 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDD	56	50	11 ( $\leq 50$ )	-	-	-
OCDD	82	61	29 ( $\leq 50$ )	-	-	-
2,3,7,8-TCDF	100	130	26 ( $\leq 50$ )	-	-	-
1,2,3,7,8-PeCDF	160	180	12 ( $\leq 50$ )	-	-	-
2,3,4,7,8-PeCDF	82	95	15 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8-HxCDF	290	310	7 ( $\leq 50$ )	-	-	-
1,2,3,6,7,8-HxCDF	220	210	5 ( $\leq 50$ )	-	-	-
2,3,4,6,7,8-HxCDF	52	49	6 ( $\leq 50$ )	-	-	-
1,2,3,7,8,9-HxCDF	36	39	8 ( $\leq 50$ )	-	-	-
1,2,3,4,6,7,8-HpCDF	810	650	22 ( $\leq 50$ )	-	-	-
1,2,3,4,7,8,9-HpCDF	310	330	6 ( $\leq 50$ )	-	-	-
OCDF	3100	2600	18 ( $\leq 50$ )	-	-	-

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0E010431**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E010431	SSAO4-05-1BPC** SSAO4-05-1BPC_FD	OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E010431	SSAQ3-01-1BPC**	1,2,3,4,6,7,8-HpCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E010431	SSAQ3-01-2BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E010431	SSAO4-05-1BPC** SSAQ3-01-1BPC** SSAQ3-01-2BPC SSAO4-05-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E010431	SSAO4-05-1BPC** SSAQ3-01-1BPC** SSAQ3-01-2BPC SSAO4-05-1BPC_FD	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 G0E010431**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E010431**

No Sample Data Qualified in this SDG



Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23307G21  
 SDG #: G0E010431  
 Laboratory: Test America

Stage 2B/A

Date: 4/15/10  
 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: 4/29/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	ICV
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW/A	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 1 + 4
XV.	Field blanks	SW	FB 0406=2010-RZB(FOD)120488, FB-0407=2010-RZC(FOD)130519

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:

MSOIS

1	C	SSAO4-05-1BPC **	11	0126326MB	21		31
2	D	SSAQ3-01-1BPC **	12	0134189MB	22		32
3	B	SSAQ3-01-2BPC	13		23		33
4	C	SSAO4-05-1BPC_FD	14		24		34
5		SSAO4-05-1BPCMS	15		25		35
6		SSAO4-05-1BPCMSD	16		26		36
7		SSAQ3-01-1BPCMS	17		27		37
8		SSAQ3-01-1BPCMSD	18		28		38
9			19		29		39
10			20		30		40

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

LDC #: 233075-2  
 SDG #: Secon

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 $< 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 23307A21  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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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:















LDC #: 23274  
 SDG #: 23274

**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
		1, 4	CPDS > calc range	1, 4	delete P(e)
		2	F.H.I.J.K.L.O.P.Q	2	↓
		3	H.O.Q	3	↓
		M	ZMPC results	M	↓ K(e)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23307G21  
 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)		(<math>\leq 50</math>) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	4				
A	4.1	4.9	18			
B	11	13		2	(<math>\leq 2.6</math>)	
C	6.4	7.0		0.6	(<math>\leq 2.6</math>)	
D	13	14	7			
E	13	15	14			
F	56	50	11			
G	82	61	29			
H	100	130	26			
I	160	180	12			
J	82	95	15			
K	290	310	7			
L	220	210	5			
M	52	49	6			
N	36	39	8			
O	810	650	22			
P	310	330	6			
Q	3100	2600	18			

V:\FIELD DUPLICATES\23307G21.wpd

**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,  $A_{is}$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_{is}$  = Concentration of internal standard  
 $S$  = Standard deviation of the RRFs,  $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	RRF (Initial)	Average RRF (Initial)	RRF (CS3 std)	Average RRF (Initial)	RRF (CS3 std)	%RSD	%RSD
1	1042 (105)	5/8/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.004	1.06	1.004	1.06	8.10	8.24		
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.049	1.06	1.049	1.06	5.12	5.00		
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.20	1.163	1.20	8.25	8.13		
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.073	1.11	1.073	1.11	7.66	7.86		
2			OCDF ( <sup>13</sup> C-OCDD)	1.523	1.58	1.523	1.58	8.42	8.35		
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**  
**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,  
 $A_s$  = 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 (C.S.3 std)	RRF (C.S.3 std)	%RSD	%RSD	RRF	RRF
1	1CAL (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	4.89709	4.89709	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	4.93847	4.93847	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	1CAL (905)	3/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.936	0.936	0.99	0.99	5.24	5.24	5.16	5.16
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.033	1.033	1.05	1.05	7.77	7.77	7.82	7.82
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.112	1.16	1.16	4.39	4.39	4.38	4.38
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.957	0.957	1.02	1.02	9.00	9.00	9.08	9.08
			OCDF ( <sup>13</sup> C-OCDD)	1.347	1.347	1.47	1.47	9.57	9.57	9.61	9.61
3	1CAL	4/24/10	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)								

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 =  $100 \times (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       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	17M/10A305	5/18/10	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.92135	0.96862	5.1	0.96862	5.1
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.03870	1.07593	3.6	1.07593	3.6
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.09904	1.24046	12.9	1.24046	12.9
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8-HpCDD)	0.99684	1.10973	11.3	1.10973	11.3
			OCDF (13C-OCDF)	1.26224	1.50116	18.9	1.50116	18.9
2	17M/10A305	5/18/10	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.92135	0.99756	8.3	0.99756	8.3
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.03870	1.12695	8.5	1.12695	8.5
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.09904	1.18764	8.1	1.18764	8.1
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8-HpCDD)	0.99684	1.05134	5.5	1.05134	5.5
			OCDF (13C-OCDF)	1.26224	1.50957	19.8	1.50957	19.8
3	23M/10502	5/27/10	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.088	1.25	14.9	1.25	14.9
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8-HpCDD)					
			OCDF (13C-OCDF)					

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

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	19M10SD2	4/19/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.13	4.0	1.13	4.1
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					
2	24M10B5	5/29/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.936	0.94	0.5	0.94	0.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.033	0.84	18.2	0.84	18.2
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.19	6.8	1.19	6.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.957	0.98	2.6	0.98	2.6
			OCDF ( <sup>13</sup> C-OCDD)	1.347	1.55	14.9	1.55	14.9
3	20M10ADS	5/27/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.004	1.03	2.7	1.03	2.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.049	1.07	1.7	1.07	1.7
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.18	1.8	1.18	1.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.073	1.13	5.5	1.13	5.5
			OCDF ( <sup>13</sup> C-OCDD)	1.527	1.64	7.9	1.64	7.9

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**  
**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: 5/6

Compound	Spike Added ( <u>43/9</u> )		Sample Concentration ( <u>43/9</u> )	Spiked Sample Concentration ( <u>43/9</u> )		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported	Recalculated
	MS	MSD		MS	MSD	Reported	Recalc	Reported	Recalc	RPD	RPD
2,3,7,8-TCDD	213	205	41	283	276	114	114	115	115	2.8	2.5
1,2,3,7,8-PeCDD	106	102	11	136	123	108	108	109	110	2.8	2.4
1,2,3,4,7,8-HxCDD	↓	↓	64	114	133	102	102	114	114	7.2	7.6
1,2,3,4,7,8,9-HpCDF	213	205	310	286	626	0.0	0.0	305	310	0.0	7.5
OCDF			3100	1270	4390	0.0	0.0	594	580	2.0	10.9

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0126326

Compound	Spike Added (PPG)		Spiked Sample Concentration		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	20.0	NA	22.8	NA	114	114	114	114						
1,2,3,7,8-PeCDD	100	/	111	/	111	111	111	111						
1,2,3,4,7,8-HxCDD	/	/	102	/	102	102	102	102						
1,2,3,4,7,8,9-HpCDF	/	/	112	/	112	112	112	112						
OCDF	200	/	227	/	114	114	114	114						

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</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+2	C <sub>12</sub> H <sub>7</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>7</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>7</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>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> H <sub>9</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>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF (S)		459.7348	M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>9</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> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> H <sub>9</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>9</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	DCDFE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>9</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDFE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>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>4</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(e) The following 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 30, 2010

**LDC Report Date:** June 17, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

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

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E030471

**Sample Identification**

SSAK7-05-1BPC\*\*

SSAK7-05-1BPC\_FD

\*\*Indicates sample underwent Stage 4 review

## 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.

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
0127206MB	5/7/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.053 pg/g 0.35 pg/g 0.13 pg/g 0.095 pg/g 0.24 pg/g	SSAK7-05-1BPC**
0126326MB	5/6/10	2,3,7,8-TCDD 1,2,3,4,7,8-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	0.030 pg/g 0.021 pg/g 0.030 pg/g 0.043 pg/g 0.28 pg/g 0.061 pg/g 0.032 pg/g 0.046 pg/g 0.063 pg/g 0.081 pg/g 0.035 pg/g 0.046 pg/g 0.11 pg/g 0.079 pg/g 0.21 pg/g	SSAK7-05-1BPC_FD

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

Sample EB-04302010-RZD (from SDG G0E030473) 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
EB04302010-RZD	4/30/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.7 pg/L 1.4 pg/L 1.5 pg/L 2.8 pg/L 7.2 pg/L 1.5 pg/L 2.2 pg/L 1.2 pg/L 1.9 pg/L 1.6 pg/L 2.4 pg/L 1.4 pg/L 5.0 pg/L	All samples in SDG G0E030471

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 G0E030471

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 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
SSAK7-05-1BPC**	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E030471	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 G0E030471	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 SSAK7-05-1BPC\*\* and SSAK7-05-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK7-05-1BPC**	SSAK7-05-1BPC_FD				
2,3,7,8-TCDD	7.6	4.9	43 (≤50)	-	-	-
1,2,3,7,8-PeCDD	26	16	48 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAK7-05-1BPC**	SSAK7-05-1BPC_FD				
1,2,3,4,7,8-HxCDD	17	9.7	-	7.3 (≤2.6)	J (all detects)	A
1,2,3,6,7,8-HxCDD	39	23	52 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	37	19	64 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	160	72	76 (≤50)	-	J (all detects)	A
OCDD	150	58	88 (≤50)	-	J (all detects)	A
2,3,7,8-TCDF	210	140	40 (≤50)	-	-	-
1,2,3,7,8-PeCDF	410	260	45 (≤50)	-	-	-
2,3,4,7,8-PeCDF	230	140	49 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	800	440	58 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	580	310	61 (≤50)	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	150	84	56 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	84	45	60 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	1900	840	77 (≤50)	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	880	400	75 (≤50)	-	J (all detects)	A
OCDF	7000	2000	111 (≤50)	-	J (all detects)	A

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0E030471**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E030471	SSAK7-05-1BPC**	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E030471	SSAK7-05-1BPC** SSAK7-05-1BPC_FD	All compounds reported below the PQL	J (all detects)	A	Project Quantitation Limit (sp)
G0E030471	SSAK7-05-1BPC** SSAK7-05-1BPC_FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0E030471	SSAK7-05-1BPC** SSAK7-05-1BPC_FD	1,2,3,4,7,8-HxCDD	J (all detects)	A	Field duplicates (Difference) (fd)
G0E030471	SSAK7-05-1BPC** SSAK7-05-1BPC_FD	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0E030471**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0E030471**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E030471**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23307H21  
 SDG #: G0E030471  
 Laboratory: Test America

Stage 2B/4

Date: 6/10/10  
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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: 4/30/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/10V	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No used sp - No Anal
VII.	Laboratory control samples	A	LC9
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	TW	D = 1 + 2
XV.	Field blanks	TW	FB-04072010-RZD(F0D090441), EB-04302010-RZD(F01203473)

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	SSAK7-05-1BPC ** S	11	0127206MB	21		31
2	SSAK7-05-1BPC_FD ↓	12	0126326MB	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 $< 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound $\geq 2.5$ and for each recovery and internal standard $> 10$ ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Continuing calibration</b>				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

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 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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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:

# VALIDATION FINDINGS WORKSHEET Blanks

LDC #: 2330742  
SDG #: 200000

**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: 5/11/10 Blank analysis date: 5/19/10  
Conc. units: pg Associated Samples: 1 (75X)

Compound	Blank ID	Sample Identification
	<u>0.0206/13</u>	
<u>F</u>	<u>0.053</u>	
<u>G</u>	<u>0.75</u>	
<u>O</u>	<u>0.13</u>	
<u>P</u>	<u>0.095</u>	
<u>A</u>	<u>0.54</u>	

Blank extraction date: \_\_\_\_\_ 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".

Blanks

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: 5/6/10    Blank analysis date: 5/18/10

Conc. units: pg/g    Associated samples: 2 (>5X)

Compound	Blank ID	Sample Identification			
	0126326MR	5X			
A	0.030	0.15			
C	0.021	0.105			
E	0.030	0.15			
F	0.043	0.215			
G	0.28	1.4			
H	0.061	0.305			
I	0.032	0.16			
J	0.046	0.23			
K	0.063	0.315			
L	0.081	0.405			
M	0.035	0.175			
N	0.046	0.23			
O	0.11	0.55			
P	0.079	0.395			
Q	0.21	1.05			

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

### VALIDATION FINDINGS WORKSHEET

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:** 4/30/10

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

Compound	Blank ID	Sample Identification				
	EB-04302010-RZD					
C	1.7	5X				
D	1.4	0.0085				
E	1.5	0.007				
F	1.5	0.0075				
G	2.8	0.014				
H	7.2	0.036				
I	1.5	0.0075				
J	2.2	0.011				
K	1.2	0.006				
L	1.9	0.0095				
M	1.6	0.008				
N	2.4	0.012				
O	1.4	0.007				
P	5.0	0.025				
Q						
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: 4/7/10

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

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".

LDC #: 2330741  
 SDG #: See Cover

# 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
		1	H.O.R. > calcs range		Notes / F(e)
		NA	ENPC results	NA	(K)(K)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23307H21  
 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 Were field duplicate pairs identified in this SDG?  
Y N NA Were target analytes detected in the field duplicate pairs?

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	1	2				
A	7.6	4.9	43			
B	26	16	48			
C	17	9.7		7.3	(≤2.6)	↓ detects/A (fd)
D	39	23	52			↓
E	37	19	64			
F	160	72	76			
G	150	58	88			↓
H	210	140	40			
I	410	260	45			
J	230	140	49			
K	800	440	58			↓ detects/A (fd)
L	580	310	61			↓
M	150	84	56			
N	84	45	60			
O	1900	840	77			
P	880	400	75			↓
Q	7000	2000	111			↓

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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:

$RRF = (A_x)(C_{is}) / (A_{is})(C_x)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$

$A_x$  = Area of compound,  $A_{is}$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_{is}$  = Concentration of internal standard  
 $S$  = Standard deviation of the RRFs,  $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	RRF (Initial)	Average RRF (Initial)	RRF (CS std)	RRF (CS std)	%RSD	RRF (CS 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		
			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		
			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	4.93847	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		
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.32988	1.26224	1.32988	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		
			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 #: 23307H21  
 SDG #: 281CDU2

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**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 = continuing calibration RRF  
 $\text{RRF} = (A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	17M10A3DS	5/18/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.92135	0.99756	8.3	0.99756	8.3
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.03870	1.12695	8.5	1.12695	8.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.09904	1.18764	8.1	1.18764	8.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.99684	1.05134	5.5	1.05134	5.5
			OCDF ( <sup>13</sup> C-OCDD)	1.26224	1.50957	19.6	1.50957	19.6
2	19M10552	5/19/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.13	4.3	1.13	4.3
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					
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.

LDC #: 2330TH21  
 SDG #: 221 cover

## VALIDATION FINDINGS WORKSHEET

### Sample Calculation Verification

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

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

Y  N  N/A  
 Y  N  N/A

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

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

- 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. 1, 2:

$$\text{Conc.} = \frac{(452998)(2000)}{(2181473)(1.09904)(10.43)(6.94)}$$

= 38.5 pg/g

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

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

LDC #: 23307A21  
 SDG #: SHLCOVH

**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: 01276206

Compound	Spike Added ( <u>155g</u> )		Spiked Sample Concentration ( <u>827g</u> )		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	22.6	NA	113	113	113	113					
1,2,3,7,8-PeCDD	100	/	108	/	108	108	108	108						
1,2,3,4,7,8-HxCDD	✓	/	105	/	105	105	105	105						
1,2,3,4,7,8,9-HpCDF	✓	/	119	/	119	119	119	119						
OCDF	200	✓	245	✓	123	123	123	123						

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>7</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>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>3</sub> <sup>37</sup> Cl <sub>10</sub> <sub>2</sub>	TCDD		425.7737	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O <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+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8964	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFE		479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>10</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>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		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</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>5</sub> <sup>37</sup> ClO	HxCDFE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> O	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> O	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> 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  
 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 30, 2010  
**LDC Report Date:** June 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0E030477

**Sample Identification**

SSAQ5-01-2BPC  
SSAQ5-01-1BPC  
SSAQ5-01-1BPC\_FD  
SSAQ5-01-2BPCMS  
SSAQ5-01-2BPCMSD

## 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.
- 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
0127206MB	5/7/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.053 pg/g 0.35 pg/g 0.13 pg/g 0.095 pg/g 0.24 pg/g	All samples in SDG G0E030477



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

Samples EB-04302010-RZB-1 and EB-04302010-RZB-2 (both from SDG G0E030473) 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
EB04302010-RZB-1	4/30/10	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	0.58 pg/L 2.9 pg/L 15 pg/L 5.5 pg/L 3.5 pg/L 7.1 pg/L 7.0 pg/L 2.0 pg/L 0.81 pg/L 20 pg/L 6.1 pg/L 40 pg/L	All samples in SDG G0E030477
EB04302010-RZB-2	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 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	0.67 pg/L 0.46 pg/L 1.2 pg/L 5.7 pg/L 0.91 pg/L 0.78 pg/L 0.44 pg/L 0.85 pg/L 0.74 pg/L 0.68 pg/L 2.0 pg/L	All samples in SDG G0E030477

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

Sample 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 G0E030477

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

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

All internal standard recoveries were within QC limits.

### 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
SSAQ5-01-1BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAQ5-01-1BPC_FD	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E030477	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 G0E030477	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 SSAQ5-01-1BPC and SSAQ5-01-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAQ5-01-1BPC	SSAQ5-01-1BPC_FD				
2,3,7,8-TCDD	95	300	104 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDD	290	980	109 (≤50)	-	J (all detects)	A
1,2,3,4,7,8-HxCDD	180	900	133 (≤50)	-	J (all detects)	A
1,2,3,6,7,8-HxCDD	410	1600	118 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	390	1600	122 (≤50)	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDD	1500	5500	114 (≤50)	-	J (all detects)	A
OCDD	1600	5400	109 (≤50)	-	J (all detects)	A
2,3,7,8-TCDF	1700	5600	107 (≤50)	-	J (all detects)	A
1,2,3,7,8-PeCDF	4000	14000	111 (≤50)	-	J (all detects)	A

Compound	Concentration (ug/L)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SSAQ5-01-1BPC	SSAQ5-01-1BPC_FD				
2,3,4,7,8-PeCDF	2200	7900	113 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,4,7,8-HxCDF	8700	31000	112 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,6,7,8-HxCDF	5400	20000	115 ( $\leq 50$ )	-	J (all detects)	A
2,3,4,6,7,8-HxCDF	1500	5700	117 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,7,8,9-HxCDF	910	3800	123 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,4,6,7,8-HpCDF	18000	72000	120 ( $\leq 50$ )	-	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	8100	30000	115 ( $\leq 50$ )	-	J (all detects)	A
OCDF	62000	250000	121 ( $\leq 50$ )	-	J (all detects)	A

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0E030477**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E030477	SSAQ5-01-1BPC	OCDF	J (all detects)	P	Project Quantitation Limit (e)
G0E030477	SSAQ5-01-1BPC_FD	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E030477	SSAQ5-01-2BPC SSAQ5-01-1BPC SSAQ5-01-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E030477	SSAQ5-01-2BPC SSAQ5-01-1BPC SSAQ5-01-1BPC_FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0E030477	SSAQ5-01-1BPC SSAQ5-01-1BPC_FD	2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects)	A	Field duplicates (RPD) (fd)

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0E030477**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0E030477**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada**  
**Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E030477**

No Sample Data Qualified in this SDG

LDC #: 23307121  
 SDG #: G0E030477  
 Laboratory: Test America

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

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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: 4/30/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	ASW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 2+3
XV.	Field blanks	SW	FB04062010-R2B (CF0D120488) EB-04302010-R2B! EB-04302010-R (G02030473)

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	SSAQ5-01-2BPC	S	11	0127206MB	21		31	
2	SSAQ5-01-1BPC		12		22		32	
3	SSAQ5-01-1BPC_FD		13		23		33	
4	SSAQ5-01-2BPCMS		14		24		34	
5	SSAQ5-01-2BPCMSD		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:



VALIDATION FINDINGS WORKSHEET

Blanks

Page: 6 of 9  
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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".

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: 5/12/10 Blank analysis date: 5/19/10

Conc. units: PS/g Associated Samples: MM (C>Sx)

Compound	Blank ID	Sample Identification
[Shaded]	013 7206 MAB	
F	0.053	
G	0.35	
O	0.13	
P	0.095	
R	0.24	

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: 4/30/10  
 Field blank type: (circle one) Field Blank / Rinsate / Other: Field Blank Associated Samples: All (>5X)

Compound	Blank ID	5X	Sample Identification
	EB-04302010-BZB-1		
D	0.58	0.0029	
F	2.9	0.0145	
G	15	0.075	
I	5.5	0.0275	
J	3.5	0.0175	
K	7.1	0.0355	
L	7.0	0.035	
M	2.0	0.01	
N	0.81	0.00405	
O	20	0.1	
P	6.1	0.0305	
Q	40	0.2	
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)

N/A Were field blanks identified in this SDG?

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

Sampling date: 4/30/10

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

Associated Samples: All (>5X)

Compound	Blank ID	5X	Sample Identification
	ER-04302010-RZB-2	5X	
D	0.67	0.00335	
E	0.46	0.0023	
F	1.2	0.006	
G	5.7	0.0285	
K	0.91	0.00455	
L	0.78	0.0039	
M	0.44	0.0022	
N	0.85	0.00425	
O	0.74	0.0037	
P	0.68	0.0034	
Q	2.0	0.01	
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)

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

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

Sampling date: 4/6/10

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

Compound	Blank ID	Sample Identification				
	ER04062010-RZR					5X
E	0.68					0.0034
F	2.5					0.0125
G	6.2					0.031
H	2.7					0.0135
K	1.4					0.007
L	0.82					0.0041
N	0.94					0.0047
O	1.8					0.009
P	1.2					0.006
Q	4.4					0.022
CRQL						

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

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

LDC #: 23307121  
SDG #: See copy

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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 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 Was a MS/MSD analyzed every 20 samples of each matrix?
- N 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	Tox out for several spots	( )	( )	( )	1	No Qual
			sample has high conc of targets (tox)	( )	( )	( )		(see in)
				( )	( )	( )		
				( )	( )	( )		
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LDC #: 23307121  
SDG #: *Re Cover*

VALIDATION FINDINGS WORKSHEET  
Compound Quantitation and Reported CRQLs

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		<i>2</i>	<i>&amp; &gt; calib range</i>	<i>2</i>	<i>None (E)</i>
		<i>3</i>	<i>D.R. ↓</i>	<i>3</i>	<i>↓</i>
		<i>All</i>	<i>EMPC results</i>	<i>all</i>	<i>None (E)</i>

Comments: See sample calculation verification worksheet for recalculations

LDC#: 23307121  
 SDG#: See Cover

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

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

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

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

Compound	Concentration (pg/g)		(<50) RPD	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	2	3				
A	95	300	104			dots/A (fd)
B	290	980	109			
C	180	900	133			
D	410	1600	118			
E	390	1600	122			
F	1500	5500	114			
G	1600	5400	109			
H	1700	5600	107			
I	4000	14000	111			
J	2200	7900	113			
K	8700	31000	112			
L	5400	20000	115			
M	1500	5700	117			
N	910	3800	123			
O	18000	72000	120			
P	8100	30000	115			
Q	62000	250000	121			

V:\FIELD DUPLICATES\23307121.wpd



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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** April 30, 2010  
**LDC Report Date:** June 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E030478

**Sample Identification**

SSAK6-02-5BPC  
SSAK6-03-3BPC

## 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
0127206MB	5/7/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.053 pg/g 0.35 pg/g 0.13 pg/g 0.095 pg/g 0.24 pg/g	SSAK6-03-3BPC

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0130380MB	5/10/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.41 pg/g 1.6 pg/g 0.65 pg/g 1.3 pg/g 0.67 pg/g 0.28 pg/g 2.0 pg/g 0.82 pg/g 4.0 pg/g	SSAK6-02-5BPC

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

Sample EB-04302010-RZD (from SDG G0E030473) 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
EB04302010-RZD	4/30/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.7 pg/L 1.4 pg/L 1.5 pg/L 2.8 pg/L 7.2 pg/L 1.5 pg/L 2.2 pg/L 1.2 pg/L 1.9 pg/L 1.6 pg/L 2.4 pg/L 1.4 pg/L 5.0 pg/L	All samples in SDG G0E030478

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 GOE030478

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
SSAK6-02-5BPC	<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
SSAK6-03-3BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E030478	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 G0E030478	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 G0E030478**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E030478	SSAK6-02-5BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E030478	SSAK6-03-3BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E030478	SSAK6-02-5BPC SSAK6-03-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E030478	SSAK6-02-5BPC SSAK6-03-3BPC	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  
G0E030478**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0E030478**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E030478**

No Sample Data Qualified in this SDG



**Tronox Northgate Henderson  
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23307J21  
SDG #: G0E030478  
Laboratory: Test America

Stage 2B

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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: 4/30/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/NA	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	No ass'd sample - No level
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	RN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB-04072010-R2D (F0D0904#1) ZB-04302010-R2D (F020304#3)

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	SSAK6-02-5BPC	S	11	0130380MB	21	31
2	SSAK6-03-3BPC	W	12	012T206MB	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: 4/30/10

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

Associated Samples: All (>5X)

Compound	Blank ID	5X	Sample Identification
	EB-04302010-RZD		
C	1.7	0.0085	
D	1.4	0.007	
E	1.5	0.0075	
F	2.8	0.014	
G	7.2	0.036	
I	1.5	0.0075	
K	2.2	0.011	
L	1.2	0.006	
M	1.9	0.0095	
N	1.6	0.008	
O	2.4	0.012	
P	1.4	0.007	
Q	5.0	0.025	
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 > 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
			I	87 (40-135)	✓ N/A P (SE-R)
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
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				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC # 2330712  
 SDG # See EDW

# 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)  
 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
		2	H. o. p. & z calc	range	dotz P (E)
		All	ZNFC results	all	JK (K)

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:** May 3, 2010  
**LDC Report Date:** June 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E040578

**Sample Identification**

SSAK7-06-1BPC  
SSAK8-07-1BPC  
SSAL7-04-1BPC  
SSAL2-03-1BPC  
SSAK7-03-1BPCMS  
SSAK7-03-1BPCMSD

## Introduction

This data review covers 6 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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
0131390MB	5/11/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.073 pg/g 0.40 pg/g 0.12 pg/g 0.12 pg/g 0.075 pg/g 0.18 pg/g 0.42 pg/g	All samples in SDG G0E040578

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 G0E040578

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
SSAK7-06-1BPC	<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	36 (40-135) 33 (40-135) 25 (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
SSAL7-04-1BPC	<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
SSAK8-07-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E040578	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 G0E040578	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 G0E040578**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E040578	SSAK7-06-1BPC	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)
G0E040578	SSAL7-04-1BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E040578	SSAK8-07-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E040578	SSAK7-06-1BPC SSAK8-07-1BPC SSAL7-04-1BPC SSAL2-03-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E040578	SSAK7-06-1BPC SSAK8-07-1BPC SSAL7-04-1BPC SSAL2-03-1BPC	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  
G0E040578**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E040578**

No Sample Data Qualified in this SDG



**Tronox Northgate Henderson  
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23307K21  
SDG #: G0E040578  
Laboratory: Test America

Stage 2B

Date: 5/5/10  
Page: 1 of 1  
Reviewer: \_\_\_\_\_  
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>5/3/10</u>
II.	HRGC/HRMS Instrument performance check	A
III.	Initial calibration	A
IV.	Routine calibration/ITV	A
V.	Blanks	TW
VI.	Matrix spike/Matrix spike duplicates	A
VII.	Laboratory control samples	A <u>LOS</u>
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 / <u>FB-04072010-R2D (FAD090441)</u>

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

Validated Samples:

1	SSAK7-03-1BPC <sup>06</sup>	5	11	<u>0131390MB</u>	21	31
2	SSAK8-07-1BPC		12		22	32
3	SSAL7-04-1BPC		13		23	33
4	SSAL2-03-1BPC		14		24	34
5	SSAK7-03-1BPCMS		15		25	35
6	SSAK7-03-1BPCMSD		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:









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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** May 3, 2010  
**LDC Report Date:** June 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0E040582

**Sample Identification**

SSAK8-06-4BPC  
SSAK8-06-5BPC  
SSAL2-02-3BPC

## **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
0134189MB	5/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.41 pg/g 0.12 pg/g 0.11 pg/g 0.15 pg/g 0.14 pg/g 0.35 pg/g	SSAK8-06-4BPC SSAK8-06-5BPC
0131390MB	5/11/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.073 pg/g 0.40 pg/g 0.12 pg/g 0.12 pg/g 0.075 pg/g 0.18 pg/g 0.42 pg/g	SSAL2-02-3BPC

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 G0E040582

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
SSAK8-06-4BPC SSAL2-02-3BPC	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)	P
SSAK8-06-5BPC	1,2,3,4,7,8-HxCDF 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E040582	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 G0E040582	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 G0E040582**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E040582	SSAK8-06-4BPC SSAL2-02-3BPC	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)
G0E040582	SSAK8-06-5BPC	1,2,3,4,7,8-HxCDF 2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E040582	SSAK8-06-4BPC SSAK8-06-5BPC SSAL2-02-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E040582	SSAK8-06-4BPC SSAK8-06-5BPC SSAL2-02-3BPC	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 G0E040582**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E040582**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson  
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23307L21  
SDG #: G0E040582  
Laboratory: Test America

Stage 2B

Date: 6/15/10  
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>5/3/10</u>
II.	HRGC/HRMS instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	<u>No ass'd sample - No level</u>
VII.	Laboratory control samples	A	<u>CCS</u>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	<del>X</del> N	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	<u>FB-04072010-R2D (G0D090441)</u>

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

Validated Samples:

1	SSAK8-06-4BPC	<u>5</u>	11	<u>0134-189NB</u>	21	31
2	SSAK8-06-5BPC	V	12		22	32
3	SSAL2-02-3BPC		13		23	33
4			14		24	34
5			15		25	35
6			16		26	36
7			17		27	37
8			18		28	38
9			19		29	39
10			20		30	40

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

# 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 #: 233072-1  
 SDG #: See above

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

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: 2

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		<u>1, 3</u>	<u>spds &gt; calibration</u>	<u>1, 3</u>	<u>Identified (e)</u>
		<u>2</u>	<u>K.H.O.P.R</u>	<u>2</u>	<u>✓</u>
		<u>nil</u>	<u>ZMPC results</u>	<u>nil</u>	<u>✓ (e)</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:** May 4, 2010  
**LDC Report Date:** June 17, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B & 4  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0E050582

**Sample Identification**

SSAL3-05-1BPC  
SSAM5-03-4BPC\*\*

\*\*Indicates sample underwent Stage 4 review

## 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.

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
0127206MB	5/7/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.053 pg/g 0.35 pg/g 0.13 pg/g 0.095 pg/g 0.24 pg/g	SSAL3-05-1BPC
0134189MB	5/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.41 pg/g 0.12 pg/g 0.11 pg/g 0.15 pg/g 0.14 pg/g 0.35 pg/g	SSAM5-03-4BPC**

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
SSAL3-05-1BPC	OCDD	0.44 pg/g	0.44U pg/g

Sample EB05042010-RZE (from SDG G0E050610) 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
EB05042010-RZE	5/4/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.1 pg/L 3.6 pg/L 2.2 pg/L 2.8 pg/L 1.3 pg/L 0.99 pg/L 0.88 pg/L 4.6 pg/L 2.2 pg/L 8.8 pg/L	SSAM5-03-4BPC**

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

Samples FB-0407210-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-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	SSAM5-03-4BPC**
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	SSAL3-05-1BPC

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
SSAM5-03-4BPC**	<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

## 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
SSAM5-03-4BPC**	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 G0E050582	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 G0E050582	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 G0E050582**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E050582	SSAM5-03-4BPC**	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E050582	SSAM5-03-4BPC**	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E050582	SSAL3-05-1BPC SSAM5-03-4BPC**	All compounds reported below the PQL	J (all detects)	A	Project Quantitation Limit (sp)
G0E050582	SSAL3-05-1BPC SSAM5-03-4BPC**	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  
G0E050582**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0E050582	SSAL3-05-1BPC	OCDD	0.44U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0E050582**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E050582**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23307M21  
 SDG #: G0E050582  
 Laboratory: Test America

Stage 2B/4

Date: 4/15/10  
 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: 5/4/10
II.	HRGC/HRMS Instrument performance check	A
III.	Initial calibration	A
IV.	Routine calibration/NDV	A
V.	Blanks	EW
VI.	Matrix spike/Matrix spike duplicates	EW No spike/duplicate - No Anal
VII.	Laboratory control samples	EW LCG
VIII.	Regional quality assurance and quality control	N
IX.	Internal standards	EW
X.	Target compound identifications	A
XI.	Compound quantitation and CRQLs	KN
XII.	System performance	A
XIII.	Overall assessment of data	A
XIV.	Field duplicates	N
XV.	Field blanks	EW FB-0407210 R22 (F0D090441) - FB-04132010 R142-R2E ZB05042010-R22 (F0D050610) (F0D150582)

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	SSAL3-05-1BPC	5	11	0127206MB	21	31
2	SSAM5-03-4BPC	**	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 $< 30\%$ 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

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

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:















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

LDC #: 23207M2  
 SDG #: 2c 2062

Page: 1 of 1  
 Reviewer: 9  
 2nd Reviewer: 2

**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 (Initial)	Average RRF (Initial)	RRF (0.53 std)	RRF (0.53 std)	%RSD	RRF (0.53 std)	%RSD
1	1CAR (905)	3/24/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.936	0.99	0.936	0.99	0.99	5.24	0.99	5.16
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.033	1.05	1.033	1.05	1.05	7.77	1.05	7.82
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.16	1.112	1.16	1.16	4.39	1.16	4.38
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.957	1.02	0.957	1.02	1.02	9.00	1.02	9.08
			OCDF ( <sup>13</sup> C-OCDF)	1.347	1.47	1.347	1.47	1.47	9.57	1.47	9.61
2	1CAR	4/24/10	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			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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

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

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

LDC #: 2320712  
 SDG #: Set 52127

**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	21M/105P5	5/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.936	0.94	0.5	0.94	0.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.033	0.84	18.2	0.84	18.2
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.112	1.19	6.8	1.19	6.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.957	0.98	2.6	0.98	2.6
			OCDF ( <sup>13</sup> C-OCDD)	1.347	1.55	14.9	1.55	14.9
2	22M/105P2	5/23/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.00	8.0	1.00	8.0
			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			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.



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

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

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

Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $\frac{|LCS - LCSD| * 2}{LCS + LCSD}$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0134189

Compound	Spike Added (PPM)		Spiked Sample Concentration (PPM)		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	200	NA	213	NA	107	107	107	107						
1,2,3,7,8-PeCDD	100		109		109	109	109	109						
1,2,3,4,7,8-HxCDD			101		101	101	101	101						
1,2,3,4,7,8,9-HpCDF			108		108	108	108	108						
OCDF	200		218		109	109	109	109						

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte			
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>10</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>10</sub> O	TCDF		M+4	409.7788	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>10</sub> O	TCDF (S)		M	417.8250	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>10</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)		M+2	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>10</sub> O <sub>2</sub>	TCDD		M+2	423.7767	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD		M+4	425.7737	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</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>10</sub> O <sub>2</sub>	TCDD (S)		M+2	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>35</sub> Cl <sub>9</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>35</sub> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	375.8964	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>10</sub> <sup>37</sup> ClO	HxCDFE		M+4	479.7165	M+4	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> <sup>37</sup> Cl <sub>10</sub> O	NCDFE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		LOCK	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF	
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		M+4	443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF (S)		M+2	457.7377	M+2	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)		M+4	459.7348	M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>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>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> ClO <sub>2</sub>	PeCDD	M+4	471.7750		M+4	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	PeCDD (S)	M+2	513.6775		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)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	M+4	[422.9278]		M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDFE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO	HxCDFE	LOCK	[430.9728]		LOCK	C <sub>10</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK	LOCK			LOCK				
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>1</sub> <sup>37</sup> ClO	HxCDF							
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>1</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>1</sub> <sup>37</sup> ClO	HxCDF (S)							
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>1</sub> <sup>37</sup> ClO	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO	HxCDD								
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>1</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>1</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>1</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>1</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:** May 4, 2010

**LDC Report Date:** June 14, 2010

**Matrix:** Water

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E050610

**Sample Identification**

EB05042010-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.

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
0127403MB	5/7/10	1,2,3,4,6,7,8-HpCDD OCDD	0.63 pg/L 2.0 pg/L	All samples in SDG G0E050610

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
EB05042010-RZE	1,2,3,4,6,7,8-HpCDD OCDD	1.1 pg/L 3.6 pg/L	1.1U pg/L 3.6U pg/L

Sample EB05042010-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
EB05042010-RZE	5/4/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.1 pg/L 3.6 pg/L 2.2 pg/L 2.8 pg/L 1.3 pg/L 0.99 pg/L 0.88 pg/L 4.6 pg/L 2.2 pg/L 8.8 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) and relative percent differences (RPD) were within QC limits.

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

## 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
EB05042010-RZE	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 G0E050610	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 G0E050610	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 G0E050610**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E050610	EB05042010-RZE	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0E050610	EB05042010-RZE	All compounds reported below the PQL	J (all detects)	A	Project Quantitation Limit (sp)
G0E050610	EB05042010-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 G0E050610**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0E050610	EB05042010-RZE	1,2,3,4,6,7,8-HpCDD OCDD	1.1U pg/L 3.6U pg/L	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0E050610**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson  
VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23307N21  
SDG #: G0E050610  
Laboratory: Test America

Stage 2B

Date: 6/10/10  
Page: 1 of 1  
Reviewer: \_\_\_\_\_  
2nd Reviewer: \_\_\_\_\_

**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>5/4/10</u>
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ <del>IQX</del>	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	N	<u>client provided</u>
VII.	Laboratory control samples	A	<u>LCSD</u>
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	<u>EB = 1</u>

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	EB05042010-RZE	W	11	<u>0127403MB</u>	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:







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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** May 5, 2010  
**LDC Report Date:** June 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E060621

**Sample Identification**

SSAM7-05-1BPC  
SSAO3-02-1BPC  
SSAO7-03-1BPC  
SSAQ5-02-1BPC  
SSAO3-03-9BPC



## 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.
- 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
0134189MB	5/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.41 pg/g 0.12 pg/g 0.11 pg/g 0.15 pg/g 0.14 pg/g 0.35 pg/g	SSAO3-03-9BPC
0130380MB	5/10/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.41 pg/g 1.6 pg/g 0.65 pg/g 1.3 pg/g 0.67 pg/g 0.28 pg/g 2.0 pg/g 0.82 pg/g 4.0 pg/g	SSAM7-05-1BPC SSAO3-02-1BPC SSAO7-03-1BPC SSAQ5-02-1BPC

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
SSAQ5-02-1BPC	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.30 pg/g 1.4 pg/g 0.72 pg/g 1.6 pg/g 1.1 pg/g 0.35 pg/g 3.3 pg/g 1.6 pg/g 10 pg/g	0.30U pg/g 1.4U pg/g 0.72U pg/g 1.6U pg/g 1.1U pg/g 0.35U pg/g 3.3U pg/g 1.6U pg/g 10U pg/g

Samples FB04062010-RZB (from SDG G0D120488), FB-04072010-RZC (from SDG G0D130519), 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-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	SSAM7-05-1BPC

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	SSAO3-02-1BPC SSAO7-03-1BPC SSAO3-03-9BPC
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	SSAQ5-02-1BPC

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
SSAM7-05-1BPC	<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) 28 (40-135) 18 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SSAO7-03-1BPC	<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) 29 (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
SSAQ5-02-1BPC	<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	27 (40-135) 27 (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
SSAO3-03-9BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	28 (40-135) 12 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 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
SSAO3-02-1BPC	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAO7-03-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E060621	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 G0E060621	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 G0E060621**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E060621	SSAM7-05-1BPC SSAO7-03-1BPC SSAQ5-02-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E060621	SSAO3-03-9BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E060621	SSAO3-02-1BPC	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (e)
G0E060621	SSAO7-03-1BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E060621	SSAM7-05-1BPC SSAO3-02-1BPC SSAO7-03-1BPC SSAQ5-02-1BPC SSAO3-03-9BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E060621	SSAM7-05-1BPC SSAO3-02-1BPC SSAO7-03-1BPC SSAQ5-02-1BPC SSAO3-03-9BPC	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  
G0E060621**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0E060621	SSAQ5-02-1BPC	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.30U pg/g 1.4U pg/g 0.72U pg/g 1.6U pg/g 1.1U pg/g 0.35U pg/g 3.3U pg/g 1.6U pg/g 10U pg/g	A	bl



**Tronox LLC Facility, PCS, Henderson, Nevada**  
**Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E060621**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**

**VALIDATION COMPLETENESS WORKSHEET**

Stage 2B

LDC #: 23307O21  
 SDG #: G0E060621  
 Laboratory: Test America

Date: 6/15/10  
 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		Findings	Comments
I.	Technical holding times	A	Sampling dates: <u>5/5/10</u>
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	<u>No sp ass'd - No dual</u>
VII.	Laboratory control samples	A	<u>ICS</u>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB04062010-RZB (G0D120488) FB-04072010-RZC (G0D130519) FB-04132010-RIG2-RZE (G0D150582)

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	SSAM7-05-1BPC	<u>S</u>	11	<u>0130380MB</u>	21		31
2	SSAO3-02-1BPC		12	<u>0131189MB</u>	22		32
3	SSAO7-03-1BPC		13		23		33
4	SSAQ5-02-1BPC		14		24		34
5	SSAO3-03-9BPC		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**  **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: 2, 3, 5 (>5X)

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

**CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:**

Samples with compound concentrations within five times the associated field blank concentration are listed above, these sample results were qualified as not detected, "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?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	F	33	Y/N/A (F-F, O-R)
			H	28	
			I	18	
		3	F	31	
			H	29	
			I	13	
		4	F	27	
			H	27	
			I	17	
		5	H	28	(F-F, O-R)
			I	12	

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

LDC #: 233010-1  
 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
			<u>CRQLs &gt; calcs range</u>		
		<u>2</u>	<u>H</u>	<u>2</u>	<u>detected (e)</u>
		<u>3</u>	<u>H, K, O, P &amp;</u>	<u>3</u>	<u>↓</u>
		<u>M</u>	<u>ZMPC results</u>	<u>M</u>	<u>detected (e)</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 16, 2010

**Matrix:** Soil

**Parameters:** Dioxins/Dibenzofurans

**Validation Level:** Stage 2B

**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0E100428

**Sample Identification**

SSAK3-01-2BPC

SSAK3-01-3BPC

## 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
0130380MB	5/10/10	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.41 pg/g 1.6 pg/g 0.65 pg/g 1.3 pg/g 0.67 pg/g 0.28 pg/g 2.0 pg/g 0.82 pg/g 4.0 pg/g	All samples in SDG G0E100428

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

Sample EB-04132010-RIG3-RZD (from SDG G0D150582) was identified as a field 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 G0E100428

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 G0E100428

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
SSAK3-01-2BPC	<sup>13</sup> C-OCDD	23 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAK3-01-3BPC	<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



## 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
SSAK3-01-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAK3-01-3BPC	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 G0E100428	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 G0E100428	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 G0E100428**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E100428	SSAK3-01-2BPC SSAK3-01-3BPC	OCDD  OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E100428	SSAK3-01-2BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E100428	SSAK3-01-3BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (e)
G0E100428	SSAK3-01-2BPC SSAK3-01-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E100428	SSAK3-01-2BPC SSAK3-01-3BPC	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  
G0E100428**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0E100428**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E100428**

No Sample Data Qualified in this SDG

LDC #: 23307P21  
 SDG #: G0E100428  
 Laboratory: Test America

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

Date: 6/15/10  
 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/ICX	A	
V.	Blanks	TW	
VI.	Matrix spike/Matrix spike duplicates	TW	No ass'd sample - 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	SW	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	2B-04132010-R1&3-R2D (F0D150582)
XV.	Field blanks	TW	FB-0402010-R2D (F0D090441)

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	SSAK3-01-2BPC	S	11	0130380MB	21		31	
2	SSAK3-01-3BPC	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: 4/13/10

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

Compound	Blank ID	Blank Concentration	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".





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

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (I)
		1	I	83 (40-135)	<i>[Handwritten]</i>
		2	I	82	<i>[Handwritten]</i>

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

LDC #: 233072  
SDG #: 2ER CONEY

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

Page: 10/1  
Reviewer: 9  
2nd Reviewer: 2

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

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

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	<i>spds &gt; calib range</i>		
			H. I. K. L. O. P. R.	1	<i>RRFs P (e)</i>
		2	<i>o. # Q</i>	2	<i>↓</i>
		all	<i>ZMP = results</i>	all	<i>JK (K)</i>

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 16, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 2B  
**Laboratory:** TestAmerica, Inc.  
**Sample Delivery Group (SDG):** G0E140423

**Sample Identification**

SSAI3-01-3BPC  
SSAI3-01-3BPCMS  
SSAI3-01-3BPCMSD

## **Introduction**

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

## III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0137352MB	5/17/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.24 pg/g 0.40 pg/g 0.067 pg/g 0.055 pg/g 0.18 pg/g 0.26 pg/g	All samples in SDG G0E140423

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

Samples EB-04152010-1-RZD (from SDG G0D170492) and EB-04152010-2-RZD (from SDG G0D200558) 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-04152010-1-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 G0E140423
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 73 pg/L 7.6 pg/L 180 pg/L 58 pg/L 450 pg/L	All samples in SDG G0E140423

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 G0E140423

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

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) were not within QC limits for 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
SSAI3-01-3BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	30 (40-135) 30 (40-135) 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 compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E140423	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 G0E140423	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 G0E140423**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E140423	SSAI3-01-3BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E140423	SSAI3-01-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E140423	SSAI3-01-3BPC	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  
G0E140423**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG  
G0E140423**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E140423**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

Stage 2B

LDC #: 23307Q21  
 SDG #: G0E140423  
 Laboratory: Test America

Date: 6/15/10  
 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	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	ICS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	SN	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB-04072010-R2D (F0D090441), EB-04152010-1-R2D EB-04152010-2-R2D (F0D20058) (F0D170492)

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	SSAI3-01-3BPC	S	11	013735=MB	21	31
2	SSAI3-01-3BPCMS	↓	12		22	32
3	SSAI3-01-3BPCMSD	↓	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: 5/17/10 Blank analysis date: 5/27/10

Conc. units: pg/g Associated Samples: All (>5x)

Compound	Blank ID	Sample Identification
	<u>0137352M13</u>	
<u>F</u>	<u>0.24</u>	
<u>G</u>	<u>0.40</u>	
<u>K</u>	<u>0.067</u>	
<u>L</u>	<u>0.055</u>	
<u>O</u>	<u>0.18</u>	
<u>R</u>	<u>0.26</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".

**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: 4/16/10

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

Compound	Blank ID	Blank ID	Blank ID	Blank ID	Blank ID	Sample Identification
	EB-04152010-1-RZD	5X				
A	0.34	0.0017				
D	0.41	0.00205				
E	0.41	0.00205				
F	7.3	0.0365				
G	60	0.3				
H	0.19	0.00095				
I	0.39	0.00195				
K	1.3	0.0065				
L	0.33	0.00165				
N	0.63	0.00315				
O	2.2	0.011				
P	0.62	0.0031				
Q	6.8	0.034				
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: 4/16/10

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

Compound	Blank ID	5X	Sample Identification
	FB-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".







# VALIDATION FINDINGS WORKSHEET

## Internal Standards

**METHOD:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)  
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".  
 N/A Are all internal standard recoveries were 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 (i)
		1	F	30	NA (F-F.0-0)
			H	30	✓
			I	82	✓
		2(MSD)	F	29	No Qual
			H	28	✓
			I	19	✓
		3(MSD)	F	28	✓
			H	27	✓
			I	18	✓

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

LDC #: 83307821  
SDG #: [Signature]

### VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: 6 of 7  
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
		MU	2MPC results	MU	N/E (K)

Comments: See sample calculation verification worksheet for recalculations