

## LABORATORY DATA CONSULTANTS, INC.

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Northgate Environmental Management, Inc.

June 4, 2010

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

SUBJECT: Tronox LLC Facility, PCS, Henderson, Nevada,

**Data Validation** 

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on May 14, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

## **LDC Project # 23188:**

## SDG#

## Fraction

G0D100461, G0D130435, G0D140422 G0D140526, G0D140534, G0D140543

00D140320, G0D140334, G0D1

G0D160437, G0D170488

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** 

Attachment 1

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LDC #: 23188

SDG #: <u>G0D100461</u>, <u>G0D130435</u>, <u>G0D140422</u>, <u>G0D140526</u> <u>G0D140534</u>, <u>G0D140543</u>, <u>G0D160437</u>, <u>G0D170488</u> Page: 1 of 1 Reviewer: JE 2nd Reviewer: BC

## Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	X		-	
II. EDD Qualifier Population			4 4	
Were all qualifiers from the validation report populated into the EDD?	Х			
III. EDD Lab Anomaties				The second of th
Were EDD anomalies identified?	X			
If yes, were they corrected or documented for the client?	Х			See EDD_discrepancy_ form_LDC23188_060310.doc
IV. EDD Delivery	ill lij			
Was the final EDD sent to the client?	X			

## Tronox LLC Facility, PCS, Henderson, Nevada Data Validation Reports LDC #23188

Dioxins/Dibenzofurans



## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 8, 2010

LDC Report Date:

June 3, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D100461

Sample Identification

SSAO5-02-1BPC

SSAO5-03-1BPC\*\*

SSAO5-02-1BPC FD

SSAO5-03-1BPC FD

SSAO4-02-3BPC

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/20/10	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	30.5	SSAO5-03-1BPC** SSAO5-03-1BPC_FD	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	Р

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

## V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0103408MB	4/13/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.040 pg/g 0.13 pg/g 0.71 pg/g 0.033 pg/g 0.048 pg/g 0.033 pg/g 0.13 pg/g 0.068 pg/g 0.32 pg/g	SSAO5-02-1 BPC SSAO5-03-1 BPC** SSAO5-02-1 BPC_FD SSAO5-03-1 BPC_FD

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

Sample EB-04072010-RZC (from SDG G0D130519) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04072010-RZC	4/8/10	2,3,7,8-TCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	1.0 pg/L 0.69 pg/L 0.65 pg/L 5.5 pg/L 53 pg/L 2.6 pg/L 1.5 pg/L 1.0 pg/L 1.8 pg/L 1.1 pg/L 0.97 pg/L 4.5 pg/L 1.1 pg/L 1.1 pg/L 1.2 pg/L	All samples in SDG G0D100461

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

Sample FB-04072010-RZC (from SDG G0D130519) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZC	4/8/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 0CDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.0 pg/L 1.5 pg/L 6.7 pg/L	All samples in SDG G0D100461

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
SSA05-02-1BPC	<sup>13</sup> C-OCDD	31 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAO5-03-1BPC**	13C-OCDD	38 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAO4-02-3BPC	<sup>13</sup> C-OCDD	36 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

## 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D100461	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 G0D100461	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	А

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 SSAO5-02-1BPC and SSAO5-02-1BPC\_FD and samples SSAO5-03-1BPC\*\* and SSAO5-03-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentra	ition (pg/g)				
Compound	SSA05-02-1BPC	SSAO5-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	2.5	2.4	-	0.1 (≤0.53)	-	-
1,2,3,7,8-PeCDD	7.7	6,8	-	0.9 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	4.7	4.4	-	0.3 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	10	8.7	-	1.3 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	9.2	8.4	•	0.8 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	33	30	10 (≤50)	-	-	-
OCDD	38	40	5 (≤50)	-	•	-
2,3,7,8-TCDF	75	71	5 (≤50)	-	-	-
1,2,3,7,8-PeCDF	130	120	8 (≤50)	-	-	-
2,3,4,7,8-PeCDF	69	66	4 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	250	240	4 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	120	120	0 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	32	28	13 (≤50)	-	-	•
1,2,3,7,8,9-HxCDF	31	29	7 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	530	490	8 (≤50)	-	-	_
1,2,3,4,7,8,9-HpCDF	290	260	11 (≤50)	-	-	-

	Concentra	ition (pg/g)				
Compound	SSAO5-02-1BPC	SSAO5-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
OCDF	1900	1800	5 (≤50)	-	-	_

	Concentr	ation (pg/g)				
Compound	SSA05-03-1BPC**	SSAO5-03-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	31	33	-	2 (≤0.11)	-	_
1,2,3,7,8-PeCDD	110	110	-	0 (≤53)	-	-
1,2,3,4,7,8-HxCDD	66	84	-	18 (≤53)	-	-
1,2,3,6,7,8-HxCDD	150	150	-	0 (≤53)	-	-
1,2,3,7,8,9-HxCDD	130	150	-	20 (≤53)	<u>.</u>	-
1,2,3,4,6,7,8-HpCDD	530	630	17 (≤50)	-	-	-
OCDD	560	680	19 (≤50)	-	-	-
2,3,7,8-TCDF	730	780	7 (≤50)	-	<u>-</u>	-
1,2,3,7,8-PeCDF	1500	1600	6 (≤50)	-	-	-
2,3,4,7,8-PeCDF	800	820	2 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	3400	3600	6 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	1700	1800	6 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	310	350	12 (≤50)	-	-	_
1,2,3,7,8,9-HxCDF	350	370	6 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	7600	8200	8 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	3600	4000	11 (≤50)		-	-
OCDF	24000	28000	15 (≤50)	-		-

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D100461

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D100461	SSAO5-03-1BPC** SSAO5-03-1BPC_FD	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J+ (all detects) J+ (all detects) J+ (all detects) J+ (all detects)	Р	Routine calibration (%D) (c)
G0D100461	SSAO5-02-1BPC SSAO5-03-1BPC** SSAO4-02-3BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0D100461	SSAO5-02-1BPC SSAO5-03-1BPC** SSAO5-02-1BPC_FD SSAO5-03-1BPC_FD SSAO4-02-3BPC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0D100461	SSAO5-02-1BPC SSAO5-03-1BPC** SSAO5-02-1BPC_FD SSAO5-03-1BPC_FD SSAO4-02-3BPC	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 G0D100461

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D100461

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D100461

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 23188A21 VALIDATION COMPLETENESS WORK
SDG #: G0D100461 Stage 2B
Laboratory: Test America

Date: 40/10
Page: 10f1
Reviewer: 9
2nd Reviewer: 1

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 4/8/10
II.	HRGC/HRMS Instrument performance check	A	/
III.	Initial calibration	A	
IV.	Routine calibration/I	W	
V.	Blanks	w	
VI.	Matrix spike/Matrix spike duplicates	W	NO SDI assid - No Cenal
VII.	Laboratory control samples	$\triangle$	109
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	an	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	N	
XIII.	Overall assessment of data	<del>-</del> #	
XIV.	Field duplicates	W	D=1+3. 2+4
XV.	Field blanks	W	B-040790-KZC. ZB-040790-RZC(40DB0519

Note: A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

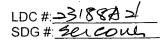
TB = Trip blank

EB = Equipment blank

Validated Samples: x | Laul (V

1	SSAO5-02-1BPC	5 11	0103408MB	21		31
2	SSAO5-03-1BPC <b>**</b>	1 12	0103408MB 0105361M13	22	1	32
3	SSAO5-02-1 BPC_FD	13		23		33
4	SSAO5-03-1BPC_FDX	14		24		34
5	SSAO4-02-3BPC	15		25		35
6	`	16		26		36
7		17		27		37
8		18		28		38
9		19		29		39
10		20		30		40

Notes:	 		
-			

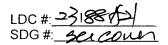


## **VALIDATION FINDINGS CHECKLIST**

Page: /of >
Reviewer: 9
2nd Reviewer: \_\_\_\_\_\_

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

Validation Area	Yes	No	NA	Findings/Comments '
I. Technical holding times	<u> </u>			
All technical holding times were met.				-
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check		<del>,</del>		
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?	1			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration	·			
Was the initial calibration performed at 5 concentration levels?	/			The state of the s
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?				
IV. Continuing calibration				
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?				
V. Blanks				
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?				
VI. Matrix spike/Matrix spike duplicates			7.3	
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.		D		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VII. Laboratory control samples	1111			
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



## **VALIDATION FINDINGS CHECKLIST**

Page:_	<u> &gt;of &gt;</u>
Reviewer:_	
2nd Reviewer:_	

VIII. Regional Quality Assurance and Quality Control	<u></u> .		<u> </u>	<b>P</b>
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	]			<u></u>
IX. Internal standards	<u>, i</u>	·		
Were internal standard recoveries within the 40-135% criteria?		/	<u> </u>	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?			<u> </u>	
X. Target compound identification	7		1	
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?	<u> </u>			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?				
Was an acceptable lock mass recorded and monitored?				
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII. System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				·
Target compounds were detected in the field duplicates.				
XV: Field blanks		Na		
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. ocbb	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#:23/88/4> SDG #:See Cover

## **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Page:\_ Reviewer: 2nd Reviewer:

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

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

Sampling date: 4/8/10

N D

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

Compound	OI Vacia							
31334	Dialin ID			Sai	Sample Identification	ıtion		
	FR-04072010-RZC	5X						
А	1.0	0.005						
D	69:0	0.00345						
E	0.65	0.00325						
<u>L</u>	5.5	0.0275						
9	53	0.265						
工	2.6	0.013						
-	1.5	0.0075						
70	1.0	0.005						
*	1.8	600.0						
	1.1	0.0055						
M	0.97	0.00485						
0	4.5	0.0225						
۵	1.1	0.0055						
Ø	12	90.0						
CROL								

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

LDC# 23/88/4 SDG #: See Cover

## VALIDATION FINDINGS WORKSHEET

Field Blanks

2nd Reviewer: Reviewer:

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

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

Associated sample units: Blank units: pg/L Sampling date: 4/8/10

Field blank tyne: /circle

		7	7		T	T	T		<u> </u>	Ī	T		T	Τ	T	T	T	T	T	T
~ N ~ )	e di te	ation																		
M	ample Identifie	Dample Identification																		
amples:		ř																		
Associated Samples:																				
/ Other:			28	0.00385	0.0037	0.0041	0.021	0.185	0.00285	0.0048	0.00335	0.0055	0.0048	0.005	0.005	0.0105	0.0075	0.0335		
XField Blank≯Rinsate	Blank ID	0100000000	FB-0407010-R70	0.77	0.74	0.82	4.2	37	0.57	0.96	0.67	1.1	0.96	1.0	1.0	2.1	1.5	6.7		
Field blank type: (circle pre) Field Blank Rinsate / Other:	Compound				D	Ш	14.	ව	Ŧ			~		M	Z	0	Д.	٥		CROL

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

LDC#:<u>23188A21</u> SDG#:<u>See Cover</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: of Reviewer: 2nd Reviewer:

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

YN NA YN NA

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

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	1	3	RPD	Difference	Limits	(Parent Only)
А	2.5	2.4		0.1	( <u>&lt;</u> 0.53)	
В	7.7	6.8		0.9	( <u>&lt;</u> 2.6)	
С	4.7	4.4		0.3	( <u>&lt;</u> 2.6)	
D	10	8.7		1.3	( ≤2.6)	
E	9.2	8.4		0.8	( <u>≤</u> 2.6)	
F	33	30	10			
G	38	40	5			
Н	75	71	5			
1	130	120	8			
J	69	66	4			
к	250	240	4			
L	120	120	0			
М	32	28	13			
N	31	29	7			
0	530	490	8			
Р	290	260	11	·		
Q	1900	1800	5			

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	2	4	RPD	Difference	Limits	(Parent Only)
А	31	33		2	( <u>&lt;</u> 11)	
В	110	110		0	( ≤53)	
С	66	84		18	( ≤53)	
D	150	150		0	( <u>&lt;</u> 53)	
E	130	150		20	( ≤53)	
F	530	630	17			
G	560	680	19			

LDC#: 23188A21 SDG#: See Cover

## VALIDATION FINDINGS WORKSHEET <u>Field Duplicates</u>

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

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

YNNA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concentra	tion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	2	4	RPD	Difference	Limits	(Parent Only)
Н	730	780	7			
1	1500	1600	6			
J	800	820	2			
к	3400	3600	6			
L	1700	1800	6			
М	310	350	12			
Z	350	370	6			
0	7600	8200	8			
Р	3600	4000	11			
Q	24000	28000	15			

V:\FIELD DUPLICATES\23188A21.wpd

SDG #:200 COND LDC #:00/Cox/47

## VALIDATION FINDINGS WORKSHEET Routine Calibration

2nd Reviewer: Reviewer:

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

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

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

Were all percent differences (%D) of RRFs  $\leq$  20% for unlabeled compounds and  $\leq$  30% for labeled? Did all routine calibration standards meet the Ion Abundance Ratio criteria?

*	Date	Standard ID	Compound	Finding %D d (Limit: ≤30.0%)	D )%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications (	6
	alpetr	1944-110405	40 13C-1	508 >	١,		2.4	1+4+1/P(K-N	( N
	,	,						_	
T	-								
T									
T									
		PCDDs	Selected ions (m/z)	Ion Abundance Ratio		PCDFs	Selected ions (m/z)	/z)   Ion Abundance Ratio	atio
	Tetra-		M/M+2	0.65-0.89		Tetra-	M/M+2	0.65-0.89	
	Penta-		M+2/M+4	1.32-1.78		Penta-	M+2/M+4	1.32-1.78	
	Hexa-		M+2/M+4	1.05-1.43		Неха-	M+2/M+4	1.05-1.43	
	Hexa- <sup>13</sup> C-HxC	Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59	
I	Hepta-¹³C-Hp	Hepta-¹³C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51	
	Hepta-		M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	0.88-1.20	
	Octa-		M+2/M+4	0.76-1.02		Octa-	M+2/M+4	0.76-1.02	
									-

LDC #: 23188A21

SDG #: See Cover

## VALIDATION FINDINGS WORKSHEET Blanks

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

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? If yes, please see qualification below. AN Z

への人 Associated samples: Blank analysis date: 4/20/10 Blank extraction date: 4/13/10 Conc. units: pg/g

Sample Identification															
Sample															
	5X	0.2	0.65	3.55	0.165	0.24	0.165	0.65	0.34	1.6					
Blank ID	0103408MB	0.040	0.13	0.71	0.033	0.048	0.033	0.13	0.068	0.32					
Compound															
		ш	Ц.	၅	I	ᅩ		0	<u>σ</u>	ø					

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".

SDG #5ex GOVI LDC #:03/202/4

## **VALIDATION FINDINGS WORKSHEET**

Internal Standards

Page: /of\_

2nd Reviewer: Reviewer:\_\_

**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 IN N/A

Are all internal standard recoveries were 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%)	: 40-135%)	Qualifications ( )
		/	#		<u>/</u> @	(561-07)	July (4.2)
					,	( ' ' )	1
		ב	T	W	38	( )	
						( )	
		5	Ŧ	W	8		Ł
						( )	
						( )	
						( )	
						( )	
П						( )	
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						( )	
						( )	
						( )	
						( )	
					-	( )	
						( )	
						( )	
						( )	
						(	
1							
						(	
		Internal Standards	Check Standard Used		In	Internal Standards	Check Standard Used
Ą.	13C-2,3,7,8-TCDF	JDF		G.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	IPCDF	
В.	<sup>13</sup> C-2,3,7,8-TCDD	CDD		Ξ	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	рсор	
ن	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF			13C-OCDD		
Ö.	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		Ϋ́	13C-1,2,3,4-TCDD		
ш	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	3-HxCDF		نـ	13C-1,2,3,7,8,9-HxCDD	CDD	
Ч	13C-123678-HxCDD	3-HxCDD					

LDC #: 33/8817/ SDG #: 20. 2000/

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of viewer:

Reviewer:

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

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

X X X X

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).

Qualifications	(F) F)								
Associated Samples									
Finding	ZHO MSULTE	(8 tag)							
Sample ID	M								
Date						0			
*									

Comments: See sample calculation verification worksheet for recalculations

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

/ of / q 2nd Reviewer: Reviewer:\_\_ Page:

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

 $A_{\mathbf{k}} = Area$  of associated internal standard  $C_{\mathbf{k}} = Concentration$  of internal standard X = Mean of the RRFs 
$$\begin{split} A_x &= \text{Area of compound,} \\ C_x &= \text{Concentration of compound,} \\ S &= \text{Standard deviation of the RRFs,} \end{split}$$

<u> </u>				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF ( <<<> std)	RRF ( CSC Seed)	%RSD	%RSD
-	1946	,	2,3,7,8-TCDF ( <sup>18</sup> C-2,3,7,8-TCDF)	0.945	0.945	86.0	0.98	4.4	4 23
	(405)	14/10	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1-0-1	1.02	40.1	F.0.7	80 M	2.07
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	4.1.4		1.19	0	5.33	50.5
			1,2,3,4,6,7,8-HpCDD (¹³C-1,2,4,6,7,8,-HpCDD)	1.072	1.072	1.1.1		3.60	3.75
			OCDF (13C-OCDD)	1.445	1.445	1.5	1.5	5.85	68.8
Ø	19/2	3/0/3	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	000:	000.	1.00	001	N.	25.1
		1/2/2	2,3,7,8-TCDD ( <sup>13</sup> C-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 ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDF (1ºC-OCDD)						
၈	1chz.	/ / [	2,3,7,8-TCDF (¹C-2,3,7,8-TCDF)	820.1	1.088	1.10	0   .	65.1	04
	(EDS)	4/21/10	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
	`		1,2,3,6,7,8-HxCDD ( <sup>18</sup> C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (*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.

LDC #: 23/88/2/

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: /of / 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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_a)(C_a)/(A_a)(C_a)$ 

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

 $A_x$  = Area of compound,  $A_x$  = Area of associated internal standard  $C_x$  = Concentration of compound,  $C_x$  = Concentration of internal standard

L					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	<b>0%</b>
	2942194P1		2,3,7,8-TCDF (1°C-2,3,7,8-TCDF)	0.945	(0.1	ao:/	6.1	1.9
	. / . / .	1/1/2	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.001	1.02	1.02	V.0	4.0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.114	611	617	6.8	<i>M</i>
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1072	1.07	1.07	1:0	1.0
			OCDF (3c-OCDD)	1.445	/: 5/	1.51	4,8	4.8
7	24 Profts	1/20/1	2,3,7,8-TCDF (1ºC-2,3,7,8-TCDF)	2460	1.06	1.06	2.51	5.01
		0/6/1	2,3,7,8-TCDD ( <sup>3</sup> C-2,3,7,8-TCDD)	1.001	1.06	90.1	3.9	3.9
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1-114	6/:/	1.19	7.5	ソン
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	1.12	4/1	4,4	49
			OCDF (13C-OCDD)	1.445	1.57	1.57	8,3	8.3
က	SOFFICEDS	*	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	0001	1.05	59:1	4.8	A. 80
	`	441	2,3,7,8-TCDD (13C-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 (*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.

1DC#23188/12/ SDG#: 281.00m

# 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 laboratoy control sample duplicate (if applicable) were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 \* SSC/SA Where:

Where: SSC = Spiked sample concentration SA = Spike added

RPD = 1 LCS - LCSD 1 \* 2/(LCS + LCSD)

LCS ID: 0103408

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

108   Perce			-1	S bodies	amula		85	USDI	ູນ	I CS/I CSD	CSD
TCDD         22.0         NA         1.CS         1.CS         1.0         1.0         Reported         Recalc         Reported         Rep	Compound	Ağ a	ike Jed 5/<	Concent Concent	ration (2)	Percent F	Recovery	Percent R	ecovery	RPD	۵
TCDD		100	Lesn	108	I CSD	Renorted	Recalc	Reported	Recalc	Reported	Recalculated
8-PeCDD 10-2 10-8 10-8 10-8 10-8 10-8 10-8 10-8 10-8	2.3.7.8-TCDD	9	NĀ	0,55	*7	011	011				
7,8-HXCDD	1,2,3,7,8-PeCDD	201		108	`	108	108				
7,8,9-HpCDF	1.2.3.4.7.8-HxCDD			911		911					
1	1,2,3,4,7,8,9-HpCDF			811		811					
	OCDF	N.	->	Sce	->	<u></u>	113				
							\		-		
						2012					
									-		

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.

Analyte	HPCDF HPCDF HPCDF HPCDD HPCDD HPCDD HPCDD SO NCDPE SO	OCDF OCDF OCDD OCDD (S) OCDD (S) DCDPE PFK	
Elemental Composition	C12 H <sup>35</sup> C1, <sup>37</sup> C1O C12 H <sup>35</sup> C1, <sup>37</sup> C1, O 13C1, H <sup>35</sup> C1, <sup>37</sup> C1, O 13C1, H <sup>35</sup> C1, <sup>37</sup> C1O C12 H <sup>35</sup> C1, <sup>37</sup> C1O <sub>2</sub> C12 H <sup>35</sup> C1, <sup>37</sup> C1O <sub>2</sub> 13C1, H <sup>35</sup> C1, <sup>37</sup> C1, O <sub>2</sub> 13C1, H <sup>35</sup> C1, <sup>37</sup> C1, O <sub>2</sub> C12 H <sup>35</sup> C1, <sup>37</sup> C1, O <sub>2</sub> C12 H <sup>35</sup> C1, <sup>37</sup> C1, O <sub>2</sub>	C, 36C, 37ClO C, 36Cl, 37ClO C, 36Cl, 37ClO <sub>2</sub> C, 36Cl, 37ClO <sub>2</sub> 13C, 28Cl, 37ClO <sub>2</sub> 13C, 28Cl, 37ClO <sub>2</sub> C, 28Cl, 37Cl <sub>2</sub> O C, 28Cl, 37Cl <sub>2</sub> O C, 18Cl, 37Cl <sub>2</sub> O	
Ion ID	M M M M M M M H H H H H H H H H H H H H	M+2 M+4 M+4 M+4 M+4 LOCK	
Accurate Mass <sup>(a)</sup>	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	441.7428 443.7399 457.7377 459.7348 469.7780 513.6775 [422.9278]	
Descriptor	4	ഗ	
Analyte	TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HXCDPE	Pecde Pecde Pecde (S) Pecde Pecde Pecde (S) Pecde (S) Pecde (S) Pecde (S) Pecde (S)	HXCDF HXCDF (S) HXCDF (S) HXCDD (S) HXCDD (S) HXCDD (S) HXCDD (S) HXCDD (S)
Elemental Composition	C <sub>12</sub> H, 35Cl <sub>4</sub> O C <sub>12</sub> H, 35Cl <sub>4</sub> O	C <sub>12</sub> H <sub>3</sub> 3C  <sub>4</sub> 7C O C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O 13C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O 13C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O <sub>2</sub> 13C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O <sub>2</sub> C <sub>12</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O C <sub>2</sub> H <sub>3</sub> 3C  <sub>3</sub> 7C O	C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO (1 <sub>2</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO (1 <sub>2</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO (1 <sub>2</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO (1 <sub>3</sub> C <sub>1</sub> +1*Cl <sub>2</sub> *ClO (1 <sub>3</sub> C <sub>1</sub> +1*Cl <sub>2</sub> *ClO (1 <sub>3</sub> C <sub>1</sub> +1*Cl <sub>2</sub> *ClO (1 <sub>3</sub> C <sub>1</sub> +1*Cl <sub>3</sub> *Cl <sub>2</sub> O (1 <sub>4</sub> C <sub>1</sub> +1*Cl <sub>3</sub> *Cl <sub>2</sub> O (1 <sub>5</sub> C <sub>1</sub> +1*Cl <sub>3</sub> *Cl <sub>2</sub> O (1 <sub>5</sub> C <sub>1</sub> +1*Cl <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O
Ol nol	C C C C C C C C C C C C C C C C C C C	M W W W W W W W W W W W W W W W W W W W	M+2 M+2 M+2 M+2 M+4 M+4 M+4
Accurate mass <sup>(s)</sup>	303.9016 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 445.7555 [430.9728]
Descriptor	-	2	8

The following nuclidic masses were used: **®** 

H = 1.007825 C = 12.000000 <sup>13</sup>C = 13.003355 F = 18.9984

O = 15.994915  $^{36}Cl = 34.968853$   $^{37}Cl = 36.965903$ 

S = internal/recovery standard

LDC #: 23188AZ/ SDG #: 20 CONEY

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:_	/of
Reviewer:	Q+
2nd reviewer:	0/
_	7

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

/	V	Ŋ	N/A
	YZ	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?

Concen	tration	$= (A_s)(l_s)(DF)  (A_k)(RRF)(V_o)(%S)$
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
$V_{\circ}$	=	Volume or weight of sample extract in milliliters (ml

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:
Conc. = $(130240)(2000)($ (855590)(1.02))(10.53)(0.92)
= 30.8 P3/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		**************************************			
·					
		76100011			
			·		

## Laboratory Data Consultants, Inc. Data Validation Report

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

Collection Date: April 9, 2010

LDC Report Date: May 21, 2010

Matrix: Soil

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

**Laboratory:** TestAmerica, Inc.

Sample Delivery Group (SDG): G0D130435

## Sample Identification

SSAM6-01-1BPC

SA60-3BPC

SSAN6-04-1BPC

SSAN6-04-1BPC FD

SSAN5-01-1BPC

SSAI2-02-2BPC\*\*

SSAJ3-01-1BPC

SSAO6-04-1BPC FD

SSAO6-04-1BPC

SSA07-01-1BPC

SSAO7-01-1BPC FD

SSAM6-01-1BPCMS

SSAM6-01-1BPCMSD

<sup>\*\*</sup>Indicates sample underwent Stage 4 review

## Introduction

This data review covers 13 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a Stage 4 review. A Stage 2B review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Stage 2B criteria since this review is based on QC data.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition) for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/28/10	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	30.3	SSAO6-04-1BPC_FD SSAO6-04-1BPC SSAO7-01-1BPC	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD	J+ (all detects) J+ (all detects) J+ (all detects)	Р

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
0104236MB	4/14/10	1,2,3,6,7,8-HxCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.11 pg/g 0.68 pg/g 0.19 pg/g 0.13 pg/g 0.098 pg/g 0.13 pg/g 0.15 pg/g 0.41 pg/g	SSAM6-01-1BPC SA60-3BPC SSAN6-04-1BPC SSAN6-04-1BPC_FD SSAN5-01-1BPC SSAI2-02-2BPC** SSAJ3-01-1BPC

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

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAN6-04-1BPC	1,2,3,6,7,8-HxCDD	0.22 pg/g	0.22U pg/g
	2,3,4,6,7,8-HxCDF	0.27 pg/g	0.27U pg/g
	1,2,3,7,8,9-HxCDF	0.34 pg/g	0.34U pg/g
SSAN6-04-1BPC_FD	1,2,3,6,7,8-HxCDD	0.19 pg/g	0.19U pg/g
	2,3,4,6,7,8-HxCDF	0.22 pg/g	0.22U pg/g
	1,2,3,7,8,9-HxCDF	0.19 pg/g	0.19U pg/g

Samples FB-04072010-RZD (from SDG G0D090441) and FB-04072010-RZC (from SDG G0D130519) were identified as field blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	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 1.6 pg/L 1.7 pg/L 1.9 pg/L 1.9 pg/L 1.1 pg/L	SSAI2-02-2BPC** SSAJ3-01-1BPC

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 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 0CDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.5 pg/L 6.7 pg/L	SSAM6-01-1BPC SA60-3BPC SSAN6-04-1BPC SSAN6-04-1BPC SSAN5-01-1BPC SSAO6-04-1BPC_FD SSAO6-04-1BPC SSAO7-01-1BPC SSAO7-01-1BPC SSAO7-01-1BPC_FD

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 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
SSAM6-01-1BPC	18C-OCDD	38 (40-135)	OCDD	J (all detects) UJ (all non-detects)	Р
			OCDF	J (all detects) UJ (all non-detects)	

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-04-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	23 (40-135) 27 (40-135) 15 (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)	Р
SSAN6-04-1BPC_FD	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	36 (40-135) 27 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р
SSAI2-02-2BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	33 (40-135) 37 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-01-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	38 (40-135) 25 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р
SSAO7-01-1BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAO7-01-1BPC_FD	<sup>13</sup> C-OCDD	22 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

## 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
SSAM6-01-1BPC SA60-3BPC	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)	P
SSAI2-02-2BPC**	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)	Р
SSAJ3-01-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample Finding		Flag	A or P
All samples in SDG G0D130435	All samples in SDG G0D130435 All compounds reported below the PQL.		А

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D130435	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	А

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 SSAN6-04-1BPC and SSAN6-04-1BPC\_FD, samples SSAO6-04-1BPC\_FD and SSAO6-04-1BPC, and samples SSAO7-01-1BPC and SSAO7-01-1BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentration (pg/g)					
Compound	SSAN6-04-1BPC	SSAN6-04-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.50U	0.041	-	0.459 (≤0.50)	-	-
1,2,3,4,7,8-HxCDD	2.5U	0.081	-	2.419 (≤2.5)	-	-
1,2,3,6,7,8-HxCDD	0.22	0.19	-	0.03 (≤2.5)	_	-
1,2,3,7,8,9-HxCDD	0.30	0.32	-	0.02 (≤2.5)	-	-
1,2,3,4,6,7,8-HpCDD	0.80	1.6	-	0.8 (≤2.5)	-	-
OCDD	3.7	8.6	-	4.9 (≤5.0)	-	-
2,3,7,8-TCDF	0.45	0.46	-	0.01 (≤0.50)	-	-
1,2,3,7,8-PeCDF	0.70	0.73	•	0.03 (≤2.5)	-	-
2,3,4,7,8-PeCDF	0.45	0.45	-	0 (≤2.5)	-	-
1,2,3,4,7,8-HxCDF	1.3	1.5	-	0.2 (≤2.5)	-	•
1,2,3,6,7,8-HxCDF	0.68	0.91	-	0.23 (≤2.5)	•	-
2,3,4,6,7,8-HxCDF	0.27	0.22	-	0.05 (≤2.5)	-	-
1,2,3,7,8,9-HxCDF	0.34	0.19	-	0.15 (≤2.5)	•	_
1,2,3,4,6,7,8-HpCDF	2.9	2.8	-	0.1 (≤2.5)	-	-
1,2,3,4,7,8,9-HpCDF	1.5	1.6	-	0.1 (≤2.5)	-	•
OCDF	5.5	7.3	-	1.8 (≤5.0)	-	-

	Concentration (pg/g)			<b></b>		
Compound	SSAO6-04-1BPC_FD	SSAO6-04-1BPC	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.86	0.80	-	0.06 (≤0.53)	-	-
1,2,3,7,8-PeCDD	2.1	2.0	-	0.1 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	1.8	1.6	-	0.2 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	3.2	3.1	-	0.1 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	2.8	2.6	•	0.2 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	18	12	40 (≤50)	-	-	-
OCDD	140	30	129 (≤50)	•	J (all detects)	А
2,3,7,8-TCDF	13	13	0 (≤50)	-	-	-
1,2,3,7,8-PeCDF	27	25	8 (≤50)	-	-	-
2,3,4,7,8-PeCDF	13	12	-	1 (≤2.6)	-	-
1,2,3,4,7,8-HxCDF	52	47	10 (≤50)	<u>.</u>	-	-
1,2,3,6,7,8-HxCDF	35	32	9 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	8.0	7.6	-	0.4 (≤2.6)	-	-
1,2,3,7,8,9-HxCDF	4.5	4.2	-	0.3 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDF	130	120	8 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	45	44	2 (≤50)	-	-	
OCDF	300	250	18 (≤50)	-	-	

	Concentration (pg/g)			5.77		
Compound	SSAO7-01-1BPC	SSAO7-01-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	1.5	1.6		0.1 (≤0.52)	-	-

	Concentration (pg/g)			D.44		
Compound	SSAO7-01-1BPC	SSAO7-01-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,7,8-PeCDD	3.4	4.0	-	0.6 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	2.4	2.6	-	0.2 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	4.2	4.8	•	0.6 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	3.6	4.1	-	0.5 (≤2.6)	-	<u>-</u>
1,2,3,4,6,7,8-HpCDD	14	16	13 (≤50)	-	-	-
OCDD	22	22	0 (≤50)	-	-	-
2,3,7,8-TCDF	160	100	46 (≤50)	-	-	-
1,2,3,7,8-PeCDF	58	62	7 (≤50)	-	•	-
2,3,4,7,8-PeCDF	24	26	8 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	76	96	23 (≤50)	-	-	_
1,2,3,6,7,8-HxCDF	50	63	23 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	12	15	22 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	8.1	9.2	-	1.1 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDF	170	200	16 (≤50)		-	-
1,2,3,4,7,8,9-HpCDF	110	94	16 (≤50)	-	-	-
OCDF	480	940	65 (≤50)	-	J (all detects)	А

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D130435

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D130435	SSAO6-04-1BPC_FD SSAO6-04-1BPC SSAO7-01-1BPC	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD	J+ (all detects) J+ (all detects) J+ (all detects)	Р	Routine calibration (%D) (c)
G0D130435	SSAM6-01-1BPC SSAO7-01-1BPC SSAO7-01-1BPC_FD	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	internal standards (%R) (i)
G0D130435	SSAN6-04-1BPC SSAI2-02-2BPC**	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р	internal standards (%R) (i)
G0D130435	SSAN6-04-1BPC_FD SSAJ3-01-1BPC	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0D130435	SSAM6-01-1BPC SA60-3BPC	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)	Р	Compound quantitation and CRQLs (e)
G0D130435	SSAI2-02-2BPC**	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)	Р	Compound quantitation and CRQLs (e)
G0D130435	SSAJ3-01-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Compound quantitation and CRQLs (e)
G0D130435	SSAM6-01-1BPC SA60-3BPC SSAN6-04-1BPC_FD SSAN5-01-1BPC SSAI2-02-2BPC** SSAJ3-01-1BPC SSAO6-04-1BPC_FD SSAO6-04-1BPC SSAO7-01-1BPC SSAO7-01-1BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D130435	SSAM6-01-1BPC SA60-3BPC SSAN6-04-1BPC SSAN6-04-1BPC FD SSAN5-01-1BPC SSAI2-02-2BPC** SSAJ3-01-1BPC SSAO6-04-1BPC FD SSAO6-04-1BPC SSAO7-01-1BPC SSAO7-01-1BPC FD	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D130435	SSAO6-04-1BPC_FD SSAO6-04-1BPC	OCDD	J (all detects)	А	Field duplicates (RPD) (fd)
G0D130435	SSAO7-01-1BPC SSAO7-01-1BPC_FD	OCDF	J (all detects)	A	Field duplicates (RPD) (fd)

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D130435

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D130435	SSAN6-04-1BPC	1,2,3,6,7,8-HxCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.22U pg/g 0.27U pg/g 0.34U pg/g	А	bl
G0D130435	SSAN6-04-1BPC_FD	1,2,3,6,7,8-HxCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.19U pg/g 0.22U pg/g 0.19U pg/g	А	bl

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D130435

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

LDC #: 23188B21	VALIDATION COMPLETENESS WORKSHEET
SDG #: G0D130435	Stage 2B/4
Laboratory: Test America	

	Date: 🗲	20/10
	Page: 7/	of /
	Reviewer: C	)
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
1.	Technical holding times	A	Sampling dates: 4/9/10
11.	HRGC/HRMS Instrument performance check	4	/ /
111.	Initial calibration	4	
IV.	Routine calibration/IX	w.	
V.	Blanks	my	
VI.	Matrix spike/Matrix spike duplicates	W	
VII.	Laboratory control samples	A	100
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
Χ.	Target compound identifications	1	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	m	Not reviewed for Stage 2B validation.
XII.	System performance		Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	W	D=3+4.8+9.10+11
XV.	Field blanks	W	D=3+4.8+9.10+1/ FBO4072010-RZD(40D090441), FB-040710-RZC(40D130514

Note: A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank

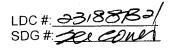
FB = Field blank

EB = Equipment blank

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

M	50115		·			
1	SSAM6-01-1BPC	11	SSAO7-01-1BPC_FD	21	0/04236MB	31
2	SA60-3BPC	12	SSAM6-01-1BPCMS	22	0/0536/ME	32
3	SSAN6-04-1BPC	13	SSAM6-01-1BPCMSD	23		33
4	SSAN6-04-1BPC_FD	14		24		34
5	SSAN5-01-1BPC	15		25		35
6	SSAI2-02-2BPC**	<b>1</b> 16		26		36
7	SSAJ3-01-1BPC	<b>D</b> 17		27		37
87	SSAO6-04-1BPC_FD	18		28		38
9	SSAO6-04-1BPC	19		29		39
10	SSA07-01-1BPC	20		30		40

Notes:	 	

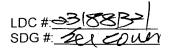


## **VALIDATION FINDINGS CHECKLIST**

Page: / of 36'
Reviewer: / 2nd Reviewer: \_ \_\_\_

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

Validation Area	Yes	No	NA	Findings/Comments '
I. Technical holding times				
All technical holding times were met.		Ĺ'	'	-
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?		'	!	
Were the retention time windows established for all homologues?		<u> </u>		
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)?	1	<u> </u>	!	
Was the mass resolution adequately check with PFK?	1/	<b></b> '	<u> </u>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	/	<u> </u>		
III. Initial calibration	<del></del>	L	· ·	1
Was the initial calibration performed at 5 concentration levels?		<u> </u>	<u>                                     </u>	
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?		<u> </u>		
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?		<u></u>		
IV. Continuing calibration	-		<u> </u>	
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?		<u> </u>	1	
V. Blanks		I I	1999 - 1999 1990 - 1999 1	
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?		· · · · · · · · · · · · · · · · · · ·		
VI. Matrix spike/Matrix spike duplicates			<u> </u>	T
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?				
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?		<u> </u>		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



## **VALIDATION FINDINGS CHECKLIST**

Page: of Pag

VIII. Regional Quality Assurance and Quality Control	•			· · · · · · · · · · · · · · · · · · ·
Were performance evaluation (PE) samples performed?	<u> </u>		1_	
Were the performance evaluation (PE) samples within the acceptance limits?				
IX. Internal standards		·		
Were internal standard recoveries within the 40-135% criteria?			<u> </u>	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?				
X. Target compound identification	1	т	1	
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?	/	<u> </u>		
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?	_	<u> </u>		
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?		]	ļ	
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	/		ļ	
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?				
Was an acceptable lock mass recorded and monitored?			<u> </u>	
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.		1		
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	a. 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	1. 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 #: ~2/8889 SDG #: Decam

## VALIDATION FINDINGS WORKSHEET Routine Calibration

Page: Reviewer: 2nd Reviewer:

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 N

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

*	Date	Standard ID	Compound		Finding %D (Limit: <30.0%)	Finding Ion Abundance Ratio	Associated Samples		Qualifications (
	0//25/10	28.4P10375.2	2 13C-D	30	. W		21-8		1748/P(C-2
		\							
									-
1									
			-						
		PCDDs	Selected ions (m/z)	lon Abundance Ratio	Ratio	PCDFs	Selected ions (m/z)	(m/z)	Ion Abundance Ratio
Ī	Tetra-		M/M+2	0.65-0.89		Tetra-	M/M+2		0.65-0.89
T	Penta-		M+2/M+4	1.32-1.78		Penta-	M+2/M+4	4	1.32-1.78
	Hexa-		M+2/M+4	1.05-1.43		Неха-	M+2/M+4	4	1.05-1.43
	Hexa- <sup>13</sup> C-HxC	Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-13C-HxCDF (IS) only	M/M+2		0.43-0.59
	Hepta- <sup>13</sup> C-Hp	Hepta-¹3C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-13C-HpCDF (IS) only	/ M/M+2		0.37-0.51
	Hepta-		M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	4	0.88-1.20
	Octa-		M+2/M+4	0.76-1.02		Octa-	M+2/M+4	4	0.76-1.02

SDG #: See Cover LDC #: 23188B21

VALIDATION FINDINGS WORKSHEET **Blanks** 

Reviewer: 2nd Reviewer:

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

Were all samples associated with a method blank?

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

Associated samples: 1-7 ( 6 ( Blank analysis date: 4/24/10 Blank extraction date: 4/14/10 Conc. units: pg/レゲー

Sample Identification 0.22/∪ 0.19∕∪ 0.19/U 0.27/U 0.22/U 0.34/∪ 0.55 0.95 0.65 0.49 0.65 0.75 2.05 ž 3.4 0104236MB Blank ID 0.098 0.68 0.19 0.13 0.15 0.11 0.13 0.41 Compound G

LDC #: 2/2008 > SDG #:See Cover

## **VALIDATION FINDINGS WORKSHEET** Field Blanks

Reviewer: 2nd Reviewer: Page:\_

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

Associated sample units:

Sampling date: 4/8/10

Field blank type: (circle one) Field Blank) Rinsate / Other:	e) Field Blank Rinsate	/ Other:	Associated Samples:	les:	1-5	1-8,8-11	
Compound	Blank ID				Sample Identification		
	EB-04072010-RZC						
ပ	0.77	0.00385					
D	0.74	0.0037					
Ш	0.82	0.0041					
ш	4.2	0.021					
9	37	0.185					
Ι	0.57	0.00285					
	96.0	0.0048					
ŗ	0.67	0.00335					
×	1.1	0.0055					
-1	0.96	0.0048					
M	1.0	0.005					
Z	1.0	0.005					
0	2.1	0.0105					
<u>d</u>	1.5	0.0075					
Ö	6.7	0.0335					
Cac							

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

SDG #:See Cover LDC #: 23188B21

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

2nd Reviewer: Reviewer:

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

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

Associated sample units: Blank units: pg/L

Associated Samples: Sampling date: 4/7/10
Field blank type: (circle one) Field Blank / Rinsate / Other:

Dinomod Plant II	Ol Angla			Sample Identification	ation		
	FB-04072010-RZD	X					
S	0.89	0.00445					
ш	1.5	0.0075					
ш	2.2	0.011					
တ	8.3	0.0415					
¥	1.4	0.007					
7	1.6	0.008					
W	1.5	0.0075				:	
Z	1.6	0.008					
0	1.3	0.0065					
Ь	1.4	0.007					
Ö	4.1	0.0205					
CROL							

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

424 CINCIC CLICCLI F. (1)

VALIDATION FIINDIINGO WORNONEET Matrix Spike/Matrix Spike Duplicates

-UCD | CU: # CUI

rage: / or / Reviewer: 2nd Reviewer:

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

N N/A Y (N N/A

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

L				Me	No.			
*	Date	MS/MSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		8/21	XX	ar		1 2/200	/	No Gas
		/	•	( / )	\( \)	( )		
				(	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				)	)	( )		
						( )		
				)	(	(		
				( )		( )		
				( )	( )	( )		
				( )	( )	( )		
			-	( )	( )	( )		-
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
	-			( )	( )	( )		
				( )	( )	( )		
				( )	)	(		

## **VALIDATION FINDINGS WORKSHEET** Internal Standards

SDG # 200 CON LDC #: 33/88/82

Reviewer: Cand Reviewer: Page:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

X/N N/A

Are all internal standard recoveries 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 ( / )
		/	+	88	SE1-04) 8	( )	(N & (4. Q)
-					)	) (	
					)	) [	
		4)	+	€\ 	)	)	
			+	74	)	) [	
			+	15	)	) (	
$\vdash$					)	)	
		4	4	98	)	)	(4.0-R
$\vdash$			Н	27	)	)	
H					)	)	
-		9	Ą	₩ W		)	
			<b>T</b>	M)	)	) (	
T			+	20	Ú	)	
T					)	(	
		_	<b>\</b>	88	)	(	
			+	25	)		
╟─					)	)	
$\vdash$		01	7	75	)	)	
				/	)	7	
		//	T	22	<b>)</b>		<b>^</b>
					)		
					)	)	
		Internal Standards	Check Standard Used		Internal Standards		Check Standard Used
Ą	13C-2,3,7,8-TCDF	F)		O	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		
ю	<sup>13</sup> C-2,3,7,8-TCDD	QQ		H	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		
ن	<sup>13</sup> C-1,2,3,7,8-PeCDF	ecdf			<sup>13</sup> C-OCDD		
<u>.</u>	<sup>13</sup> C-1,2,3,7,8-PeCDD	CDD		Α.	<sup>13</sup> C-1,2,3,4-TCDD		
ші	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	HXCDF		ا ت	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
Щ	13C-123678-HxCDD	ЧХСDD					

LDC #: <u>33/88B</u>>/ SDG #: <u>58. GDW</u>PI

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

rage.

Reviewer:

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".

A N N N N

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).

Finding  L. O. P. & Associated Samples  K. L. O. P. & Associated Samples	
dang dang	
H.T. K. L. H. T. L. H. T. K. L. H. T. K. L. H. T. L. H. T. K. L. H. T. L. L. H. T. L. L. H. T. L. H	
Sample ID	
Date O	

Comments: See sample calculation verification worksheet for recalculations

LDC#:<u>23188B21</u> SDG#:<u>See Cover</u>

## VALIDATION FINDINGS WORKSHEET Field Duplicates

	Page:	Lof 3
	Reviewer:	9
2nd	Reviewer:	

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

YN NA YN NA

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

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	3	4	RPD	Difference	Limits	(Parent Only)
А	0.50U	0.041		0.459	( <u>&lt;</u> 0.50)	
С	2.5U	0.081		2.419	( <u>≤</u> 2.5)	
D	0.22	0.19		0.03	( <u>&lt;</u> 2.5)	
E	0.30	0.32		0.02	( <u>≤</u> 2.5)	
F	0.80	1.6		0.8	( <u>&lt;</u> 2.5)	
G	3.7	8.6		4.9	( <u>&lt;</u> 5.0)	
Н	0.45	0.46		0.01	( <u>&lt;</u> 0.50)	
ı	0.70	0.73		0.03	( ≤2.5)	
J	0.45	0.45		0	( <u>&lt;</u> 2.5)	
к	1.3	1.5		0.2	( <u>&lt;</u> 2.5)	
L	0.68	0.91		0.23	( ≤2.5)	
М	0.27	0.22		0.05	( <u>≤</u> 2.5)	
N	0.34	0.19		0.15	( <u>≤</u> 2.5)	
О	2.9	2.8		0.1	( <u>&lt;</u> 2.5)	
Р	1.5	1.6		0.1	( <2.5)	
Q	5.5	7.3		1.8	( <5.0)	

	Concentrat	tion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	8	9	RPD	Difference	Limits	(Parent Only)
Α	0.86	0.80		0.06	( ≤0.53)	
В	2.1	2.0		0.1	( <u>&lt;</u> 2.6)	
С	1.8	1.6		0.2	( <u>≤</u> 2.6)	
D	3.2	3.1		0.1	( <u>&lt;</u> 2.6)	
Е	2.8	2.6		0.2	( ≤2.6)	
F	18	12	40			
G	140	30	129		-	Ults/A
н	13	13	0			

Ital)

LDC#: 23188B21 SDG#: See Cover

## VALIDATION FINDINGS WORKSHEET <u>Field Duplicates</u>

Page: 🗻	_of <u>∠</u> ≥_
Reviewer:	9
2nd Reviewer:	

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

YN NA YN NA

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

	Concentra	tion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	8	9	RPD	Difference	Limits	(Parent Only)
ı	27	25	8			
J	13	12		1	( ≤2.6)	
κ	52	47	10			
L	35	32	9			
м	8.0	7.6		0.4	( ≤2.6)	
N	4.5	4.2		0.3	( <u>&lt;</u> 2.6)	
0	130	120	8			
Р	45	44	2			
Q	300	250	18			

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	10	11	RPD	Difference	Limits	(Parent Only)
Α	1.5	1.6		0.1	( ≤0.52)	
В	3.4	4.0		0.6	( <2.6)	
С	2.4	2.6		0.2	( ≤2.6)	
D	4.2	4.8		0.6	( <u>&lt;</u> 2.6)	
Е	3.6	4.1		0.5	( <u>&lt;</u> 2.6)	
F	14	16	13			
G	22	22	0			
н	160	100	46			
I	58	62	7			
J .	24	26	8			
К	76	96	23			
L	50	63	23			
М	12	15	22			
N	8.1	9.2		1.1	( <u>&lt;</u> 2.6)	
0	170	200	16			

LDC#: 23188B21 SDG#: See Cover

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

Page: <u>→ of →</u> Reviewer: <u>→</u> 2nd Reviewer:

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

Y N NA YNNA

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

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	10	11	RPD	Difference	Limits	(Parent Only)
Р	110	94	16			
Q	480	940	65			Sty A

V:\FIELD DUPLICATES\23188B21.wpd

## LDC #: -710 072-4 SDG #: 20 CONE

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer:

/ of /

Page:

2nd Reviewer:

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 =  $\langle A_{\nu} \rangle (C_{\mu})/\langle A_{\mu} \rangle (C_{\nu})$ average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

C<sub>x</sub> = Concentration of compound, S = Standard deviation of the RRFs,  $A_x = Area of compound,$ 

 $A_{\rm b} = {\rm Area~of~associated~internal~standard~} \\ C_{\rm b} = {\rm Concentration~of~internal~standard~} \\ X = {\rm Mean~of~the~RRFs} \\$ 

Recalculated 50.5 08.5 3.75 2.07 9 %RSD 3.03 5.85 Reported 4.4 4 %RSD BANK OF Recalculated .98 0 9 CS3 std) Reported Ţ 2 a 0 Ø Average RRF Recalculated 000 94C 0 (initial) 00 Average RRF oas 243 449 Reported 10 000 (initial) Compound (Reference Internal Standard) 1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD) 1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (19C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD) 2,3,7,8-TCDD (13C-2,3,7,8-TCDD) 2,3,7,8-TCDD (13C-2,3,7,8-TCDD) 2,3,7,8-TCDF (13C-2,3,7,8-TCDF) 2,3,7,8-TCDD (13C-2,3,7,8-TCDD) 2,3,7,8-TCDF (13C-2,3,7,8-TCDF) 2,3,7,8-TCDF (13C-2,3,7,8-TCDF) OCDF (13C-OCDD) OCDF (13C-OCDD) OCDF (13C-OCDD) 3/0/10 Calibration Date Standard ID るでで 194 レを 5 (a) \*

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

LDC #: 23/28/24

# VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of 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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$ 

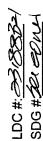
Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF

A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound,

 $A_k = Area$  of associated internal standard  $C_k = Concentration of internal standard$ 

<u></u>					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
	Tapa Hypo	/, /,	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.945	76.0	76.0	V.0	0.7
		01/40/4	74/10 2,3,7,8-TCDD (3C-2,3,7,8-TCDD)	1.021	0.96	29.0	5.6	6.0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.114	1.07	1.07	Ŋ. Ø.	W.
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	1.04	7001	3.6	3.4
			OCDF (1°C-OCDD)	1.445	1.4.1	1.4/	S. S	J. W.
7	Sahorthe	1/0/1	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	2460	0.90	0.90	K.A	4.3
	6	as /2/2	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	25.0	76.0	N.O	6.3
	\		1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.08	1.08	w	w.
			1,2,3,4,6,7,8-HpCDD (°C-1,2,4,6,7,8,-HpCDD)	1.072	801	1.03	a	8.8
			OCDF (12-OCDD)	1.445	136	1.36	5.19	5.8
က	A44108512 1	01/92/1	2,3,7,8-TCDF (°3C-2,3,7,8-TCDF)	880.1	1.02	20.1	5.9	50
	_	. / / /	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
		`	1,2,3,6,7,8-HxCDD (°C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (3c-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.



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

rage:_ Reviewer: nd Reviewer:
<u> </u>

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

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

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

MS/MSD samples: \_\_\_/

RPD = I MSR - MSDR I \* 2/(MSR + MSDR)

	os	ike	Sample	Spiked Sample	ample	Matrix	Matrix Spike	Matrix Spike Duplicate	a Duplicate	Reported	Recalculated
Compound	Adj.	Added	Concentration (アダタ)	Concentration ( PS/9)	tration	Percent F	Percent Recovery	Percent Recovery	Recovery	RPD	RPD
	MS	MSD	/ /	/ SW	MSD	Reported	Recalc	Reported	Recalc	water a	
2,3,7,8-TCDD	200	19.9	'	14 1	29.2	7	7:1	93	87	7	(3
1,2,3,7,8-PeCDD	786	99.3	7	<u>~</u>	178	7	(1)	011	107	V.	۲.
1,2,3,4,7,8-HxCDD	1		4	291	89	251	124	021	<b>8</b>	o.	3.0
1,2,3,4,7,8,9-HpCDF	>	_	0000	2740	240	843	255	440	342	4.7	2.6
OCDF	196	66	00091	00961	17800	2002	1837	1110	2905	a,	96
				_					_		
						Ŧ					
											·

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

LDC #:33/2333/ SDG #:3224000

## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: Reviewer:

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy 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 = ILCS - LCSD | \* 2/(LCS + LCSD)

LCS ID: 0/04336

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

csp	0	Recalculated									
TCS/I CSD	RPD	Reported									
an as	Recovery	Recalc				·					
I CSD	Percent Recovery	Reported									
S	ecovery	Recalc	97	100	103	100	00				
SO	Percent Recovery	Reported	97	100	103	100	de				
elume	()	l CSD	MA	,			-				-
Spiked	Concentration	) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) ) )	19.5	566	801	6.66	197				
iko	Added (A)	I CSD	Ϋ́N				->				
5	¥Ŷ	1.08	0.8	20)		_>	200				
	Сотроила		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF				

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

1   303.8016	Descriptor	Accurate mass <sup>(a)</sup>	Ol nol	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ol nol	Elemental Composition	Analyte
409.7289   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>2</sub> U <sup>2</sup> C <sub>1</sub> C   TODF   409.7789   M+4   HPODF   HPODF   HPODF   409.7889   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   409.7889   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   418.8220   M+2   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   418.8220   M+2   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   428.7389   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   428.7389   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C   HPODF   428.7389   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C <sub>2</sub> C <sub>2</sub> C   HPODF   428.7389   M+4   C <sub>2</sub> H <sup>4</sup> C <sub>1</sub> C <sub>2</sub> C <sub>1</sub> C <sub>2</sub>	-	303.9016	M	C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> O	TCDF	4	407.7818	M+2	C,H°CI,7CIO	HpCDF
315.9419   M   "C_AH_WCI_"CIO   TODF (S)   417.8250   M   TO_AH_WCI_"CIO   TODF (S)   417.8250   M   TODE		305.8987	M+2	C12H,36C1,37C10	TCDF		409.7788	M+4	C <sub>12</sub> H³cnj³7cn₂o	HpCDF
317.3839   M+2   C_0H_4C_0C_0C_0C_0C_0C_0C_0C_0C_0C_0C_0C_0C_0C		315.9419	Σ	0,12#,4,20,10	TCDF (S)		417.8250	Σ	13C12H3C1,O	HpCDF (S)
319.8865   M + 2		317,9389	M+2	13C12H4**CI3**CIO	TCDF (S)		419.8220	M+2	13C <sub>12</sub> H <sup>35</sup> CI <sub>6</sub> 37CIO	HPCDF
321,8386   M+2   C <sub>1</sub> <sub>1</sub> <sup>1</sup> <sup>1</sup> <sup>1</sup> C <sub>1</sub> <sup>1</sup> <sup>2</sup> C <sub>1</sub> <sup>2</sup> <sup>2</sup> C <sub>1</sub> <sup>2</sup> <sup></sup>		319.8965	Σ	C12H, #C1_O2	TCDD		423.7767	M+2	C12H35C1837C1O2	Ньсвр
331 9388		321.8936	M+2	C <sub>12</sub> H <sub>4</sub> **C <sub>13</sub> **C10 <sub>2</sub>	TCDD		425.7737	M+4	C <sub>1</sub> ,H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>3</sub>	HpCDD
333 8358   M+2		331.9368	Σ	13C12H, 25C1, O2	TCDD (S)		435.8169	M+2	13Č;,H³Č[,³7ČlŌ,	HPCDD (S)
375.8364   MA-2   C_n^H_wCl_v^TClO   HyCDPE   479.7165   MA+4   C_n^H^WCl_v^TClO   PRK   430.9728    LOCK   C_n^H^WCl_v^TClO   PRK   430.9728    LOCK   C_n^H^WCl_v^TClO   PRK   430.9728    LOCK   C_n^H^WCl_v^TClO   PRK   A41.7428   MA+4   C_n^H^WCl_v^TClO   PRCDF   A41.7397   MA+4   C_n^H^WCl_v^TClO   PRCDF   A41.7790   MA+2   C_n^H^WCl_v^TClO   PRCDF   A41.7790   MA+4   C_n^H^WCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v^TCl_v		333.9338	M+2	13C <sub>12</sub> H <sub>2</sub> 35Cl <sub>3</sub> 37ČlO <sub>2</sub>	TCDD (S)		437.8140	M+4	13C, H*C  37C ,O,	HpCDD (S)
1354.972  LOCK   C <sub>2</sub> F <sub>1</sub>   C <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F <sub>2</sub>   C <sub>2</sub> F		375.8364	M+2	C <sub>12</sub> H <sub>4</sub> **Cl <sub>5</sub> **ClO	HXCDPE		479.7165	M+4	C, H*CI, 37CI, Ô	
339.8597   M+2   C_12H_3^1 Cl_1^1 Cl_2   DeCDF   5   441.7428   M+2   C_12^2 Cl_2^1 Cl_2   C_2^2 Cl_2^2		[354.9792]	Lock	C,F <sub>13</sub>	PFK		[430.9728]	LOCK	ָרְיָּרְיֶּ הְיִּרְיֶּ	PFK
339.8597   M+2   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   5   441.7428   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   Cl <sub>2</sub>   PeCDF   5   441.7428   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> *Cl <sub>2</sub>   PeCDF   Sist 1867   PeCDF   Sist 1867   PeCDF   Sist 1867   PeCDF										
341.8667   M+4   C <sub>12</sub> H <sub>2</sub> H <sub>2</sub> C <sub>13</sub> C <sub>12</sub> C <sub>12</sub>   PeOPF   PeOPP   PeOPF   PeOP	2	339.8597	M+2	C.,H,35CI,37CIO	PeCDF	ιΩ	441,7428		0,350,3700	OCDF
351,9000   M+2   10,24,3c0,47c10   PeoDF (8)   457,7377   M+2   C <sub>12</sub> 2c0,7c0 <sub>2</sub>   A57,7377   M+4   C <sub>12</sub> 2c0,7c0 <sub>2</sub>   A71,7750   M+4   C <sub>12</sub> 2c0,7c0 <sub>2</sub>   A72,7c0 <sub>2</sub>   A7		341.8567	M+4	C, H, 301, 3701, O	PecDF		443.7399		0,13,501,0	OCDF
353.8970         M+4         "C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PeCDF (S)         459.7348         M+4         C <sub>12</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           355.896         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PeCDD         471.7750         M+2         O <sub>12</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           357.896         M+4         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PeCDD         471.7750         M+4         O <sub>12</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           367.8949         M+4         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PeCDD (S)         518.775         M+4         O <sub>12</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           409.7974         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PeCDD (S)         518.775         M+4         O <sub>12</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           409.7974         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PFK         PFK         C <sub>12</sub> Cl <sub>3</sub> *Cl <sub>2</sub> O           409.7974         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PFK         PFK         C <sub>12</sub> Cl <sub>3</sub> *Cl <sub>2</sub> O           409.7974         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PFK         PFK         C <sub>12</sub> Cl <sub>3</sub> *Cl <sub>2</sub> O           375.8178         M+4         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PFK         PFK         C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O           403.859         M+2         O <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O         PFK         PFK         C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> O           418.7555         M+4<		351,9000	M+2	13C, H, 3CI, 37C10	PeCDF (S)		457,7377		C,35C 37C O,	OCDD
355.8546   M+2   C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>1</sub> <sup>3</sup> Cl <sub>1</sub> <sup>2</sup>   PeCDD   469.7780   M+2   16 <sub>1</sub> (13.6C) <sup>2</sup>   C <sub>1</sub> Cl <sub>2</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   469.7780   M+4   16 <sub>1</sub> (13.6C) <sup>2</sup>   C <sub>1</sub> Cl <sub>2</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub>   PeCDD   471.7750   M+4   16 <sub>1</sub> C <sub>1</sub> M <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>3</sub>   PFK		353.8970	M+4	13C12H3 35C13O	PeCDF (S)		459.7348		C,3*C ,3*C ,0,	OCDD
357.8516         M+4         C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> PeCDD         471.750         M+4         1°C <sub>1</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> 367.8949         M+2         1°C <sub>1</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> PeCDD (S)         513.6775         M+4         C <sub>1</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> 367.8949         M+2         1°C <sub>1</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> PeCDD (S)         513.6775         M+4         C <sub>1</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> C <sub>2</sub> 403.7974         M+4         C <sub>1</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> PFK         M+4         C <sub>1</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> Cl <sub>2</sub> C <sub>2</sub> 373.8208         M+2         C <sub>1</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> HXCDF         HXCDF           375.8178         M+4         C <sub>1</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> HXCDF           385.8610         M+2         C <sub>1</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> HXCDF           385.8156         M+2         C <sub>1</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> Cl <sub>2</sub> C <sub>2</sub> HXCDD           401.8559         M+4         C <sub>1</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> Cl <sub>2</sub> C <sub>2</sub> HXCDD           445.7555         M+4         C <sub>2</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> C <sub>2</sub> 445.7555         M+4         C <sub>2</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> C <sub>2</sub> C <sub>2</sub>		355.8546	M+2	C12H3*C127CIO2	PeCDD		469.7780		13Č, 35ČI, 37ČIO,	OCDD (S)
367.8949         M+2         19C <sub>12</sub> H <sub>3</sub> sCl <sub>3</sub> TCl <sub>2</sub> Cl <sub>2</sub> PeCDD (S)         513.6775         M+4         Cr <sub>2</sub> sCl <sub>3</sub> TCl <sub>2</sub> Cl <sub>2</sub> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>2</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>2</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>3</sub>		357.8516	M+4	C <sub>1</sub> ,H <sub>3</sub> ,3C <sub>1</sub> ,37C <sub>1</sub> ,O <sub>2</sub>	PeCDD		471.7750		13C, 35C  37C , O,	ocpp (s)
369.8919         M+4         'iG_1H_3mCl_3^MCl_2^L_2         PeCDD (S)         [422.9278]         LOCK         C_nEH_3mCl_3^MCl_2^L_2         PeCDD (S)         PeCD (S)         PeCDD (S)         <		367.8949	M+2	13C1, H, 45C1, 37C1O,	PeCDD (S)		513.6775		C,,3°CI,3°CI,0°	DCDPE
409.7974         M+2         C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> *Cl         HpCDPE           [354.3792]         LOCK         C <sub>2</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> *Cl         PFK           373.8208         M+2         C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> *Cl         HxCDF           375.8178         M+4         C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>3</sub> *Cl         HxCDF           383.8639         M         InC12H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl         HxCDF           386.8156         M+2         C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl         HxCDD           391.8127         M+4         C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl <sub>2</sub> HxCDD           401.8559         M+4         InC <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl <sub>2</sub> HxCDD           403.8529         M+4         InC <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl <sub>2</sub> HxCDD           445.7555         M+4         C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> *Cl <sub>2</sub> PFK		369.8919	M+4	13C1,H,36C1,7C1,O,	PeCDD (S)		[422.9278]		, IT.	PFK
[354.9792] LOCK C <sub>9</sub> F <sub>13</sub> 373.8208 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **TClO 375.8178 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **TClO 383.8639 M  M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **TClO 389.8156 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **TClO 389.8156 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **TClO 391.8127 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> **Cl 401.8559 M+4 (**Cl <sub>3</sub> ***Cl <sub>3</sub> ***Cl <sub>2</sub> ***Cl 445.7555 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> **Cl (**2**********************************		409.7974	M+2	C <sub>12</sub> H <sub>3</sub> *Cl <sub>8</sub> 7ClO	HPCDPE					
373.8208 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> 7*ClO 375.8178 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> 7*ClO 383.8639 M C <sub>12</sub> H <sub>2</sub> **Cl <sub>4</sub> **Cl <sub>5</sub> O 385.8610 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **ClO 389.8156 M+2 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **ClO 391.8127 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **ClO 401.8559 M+2 G <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **ClO 445.7555 M+4 C <sub>12</sub> H <sub>2</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> O (430.9728] LOCK C <sub>2</sub> H <sub>2</sub> **Cl <sub>3</sub> **Cl <sub>2</sub> O		[354.9792]	LOCK	n 0	PFK					
373.8208     M+2     C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO       375.8178     M+4     C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO       383.8639     M <sup>13</sup> C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO       385.8610     M+2 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO       389.8156     M+2     C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO <sub>2</sub> 401.8559     M+4     C <sub>12</sub> H <sub>2</sub> *sCl <sub>5</sub> *7ClO <sub>2</sub> 403.8559     M+4 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> *sCl <sub>4</sub> *7Cl <sub>2</sub> O <sub>2</sub> 445.7555     M+4     C <sub>12</sub> H <sub>2</sub> *sCl <sub>4</sub> *7Cl <sub>2</sub> O <sub>2</sub> [430.9728]     LOCK     C <sub>12</sub> H <sub>2</sub> *Cl <sub>6</sub> *7Cl <sub>2</sub> O										
M+4 C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+2 (3C <sub>12</sub> H <sub>2</sub> **c(1,0) <sub>2</sub> O M+2 (3C <sub>12</sub> H <sub>2</sub> **c(1,0) <sub>2</sub> O M+4 (C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+4 (C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+4 (10C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+4 (10C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+4 (10C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+5 (10C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O M+6 (10C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> **c(1,37C) <sub>2</sub> O	ო	373.8208		C, H, **CI, **CIO	HXCDF					
M 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>0</sub> O M+2 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO M+2 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> 3]		375.8178		C;H2**O[3*Cl2O	HXCDF					
M+2 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO M+2 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+2 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 13C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VClO <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> &Cl <sub>3</sub> VCl <sub>2</sub> O <sub>2</sub>	<del></del>	383.8639		<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>8</sub> O	HXCDF (S)		_			
M+2 C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> s <sup>1</sup> Cl <sub>2</sub> Cl <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> s <sup>1</sup> Cl <sub>2</sub> C <sub>2</sub> M+2 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> s <sup>1</sup> Cl <sub>2</sub> C <sub>2</sub> M+4 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> s <sup>1</sup> Cl <sub>2</sub> C <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> s <sup>2</sup> C <sub>2</sub> C <sub>2</sub>		385.8610		13C12H23CI537CIO	HXCDF (S)					
M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub> M+2 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *Cl <sub>0</sub> M+4 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>4</sub> *Cl <sub>2</sub> O <sub>2</sub> 3] LOCK C <sub>2</sub> F <sub>7</sub>		389.8156		C <sub>12</sub> H <sub>2</sub> 35Cl <sub>2</sub> 37ClO <sub>2</sub>	HXCDD					
M+2 13C <sub>12</sub> H <sub>2</sub> sC <sub>10</sub> C <sub>10</sub> C <sub>10</sub> M+4 13C <sub>12</sub> H <sub>2</sub> sC <sub>1</sub> 3C <sub>10</sub> C <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> sC <sub>13</sub> C <sub>10</sub> C <sub>1</sub> S] LOCK C <sub>0</sub> F <sub>17</sub>		391.8127		C <sub>12</sub> H <sub>2</sub> <sup>32</sup> Cl <sub>2</sub> <sup>43</sup> Cl <sub>2</sub> O <sub>2</sub>	HXCDD					
M+4 ''C <sub>12</sub> H <sub>2</sub> <sup>2</sup> C <sub>1</sub> C <sub>1</sub> O <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> <sup>2</sup> C <sub>1</sub> (1C <sub>1</sub> O 3) LOCK C <sub>9</sub> F <sub>17</sub>		401.8559		13C12H23C157C1O2	HXCDD (S)					
3] LOCK C <sub>2</sub> H <sub>2</sub> 7, C <sub>4</sub> , C <sub>2</sub> C <sub>2</sub> C <sub>3</sub> C <sub>3</sub> C <sub>4</sub>		403.8529			HXCDD (S)					
		445.755		C_2 Z_2 Z_2 Z_2 Z_2 Z_2 Z_2 Z_2 Z_2 Z_2 Z	יין מיליקייי					
		[07/6:06+]		7. 7.	٠. ــــــــــــــــــــــــــــــــــــ					

# (a) The following nuclidic masses were used:

O = 15.994915 $^{36}CI = 34.968853$  $^{37}CI = 36.965903$ 

H = 1.007825 C = 12.000000 <sup>13</sup>C = 13.003355 F = 18.9984

S = internal/recovery standard

LDC #: 23/88B2/ SDG #: 52 COW

only.

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:	/of/_
Reviewer:	0
2nd reviewer:	

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

/	$\widehat{Y}$	N	N/A
ļ	У	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?

Conce	ntration	$= \frac{(A_{\bullet})(I_{\bullet})(DF)}{(A_{\bullet})(RRF)(V_{\bullet})(\%S)}$	Example:
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard	,
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = (16053/20) (2000) ( ) (6104400) (1.021) (0.7) (0.95)
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	= 50.6 pg/g
Df	=	Dilution Factor.	
%S	_	Percent solids, applicable to soil and solid matrices	i .

#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
						·
				7.		
			_ <u>i</u>			
		· · · · · · · · · · · · · · · · · · ·				

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 12, 2010

LDC Report Date:

May 24, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D140422

Sample Identification

SSAJ3-02-1BPC\*\*

SSAH3-01-1BPC\*\*

SSAM7-03-1BPC

SSAM7-04-1BPC

SSAN7-02-1BPC

SSAN6-06-1BPC

<sup>\*\*</sup>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.

Sample EB-04122010-RIG3-RZB (from SDG G0D140534) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG3-RZB	4/12/10	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 1,2,3,4,6,7,8-HpCDF 0CDF	0.64 pg/L 0.47 pg/L 1.9 pg/L 5.0 pg/L 4.6 pg/L 2.2 pg/L 7.7 pg/L 4.8 pg/L 1.2 pg/L 0.59 pg/L 5.0 pg/L 27 pg/L	SSAJ3-02-1BPC** SSAH3-01-1BPC**

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

Samples FB-04072010-RZD (from SDG G0D090441) and FB-04072010-RZC (from SDG G0D130519) were identified as field blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.89 pg/L 1.5 pg/L 2.2 pg/L 8.3 pg/L 1.4 pg/L 1.6 pg/L 1.5 pg/L 1.6 pg/L 1.4 pg/L 1.4 pg/L 4.1 pg/L 4.1 pg/L	SSAJ3-02-1BPC** SSAH3-01-1BPC**

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 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.0 pg/L 2.1 pg/L 6.7 pg/L	SSAM7-03-1BPC SSAM7-04-1BPC SSAN7-02-1BPC SSAN6-06-1BPC

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
SSAJ3-02-1BPC**	<sup>13</sup> C-OCDD	29 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAH3-01-1BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD	27 (40-135) 29 (40-135) 15 (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)	Р
SSAM7-03-1BPC	<sup>13</sup> C-OCDD	25 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAN7-02-1BPC	<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	35 (40-135) 36 (40-135) 26 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р
SSAN6-06-1BPC	<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	31 (40-135) 36 (40-135) 24 (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)	Р

## 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
SSAM7-03-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	Р
SSAM7-04-1BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р
SSAN6-06-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140422	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140422	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	А

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 SSAJ3-02-1BPC\*\* and SSAJ3-02-1BPC\_FD (from SDG G0D140453) and samples SSAN7-02-1BPC and SSAN7-02-1BPC\_FD (from SDG (G0D140453) and were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentration (pg/g)			D."		
Compound	SSAJ3-02-1BPC**	SSAJ3-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.22	1.1U	-	0.88 (≤1.1)	-	-
1,2,3,7,8-PeCDD	0.90	5.3U	-	4.4 (≤5.3)	-	-
1,2,3,4,7,8-HxCDD	0.99	5,3U	-	4.31 (≤5.3)	-	_
1,2,3,6,7,8-HxCDD	1.2	0.13	-	1.07 (≤5.3)	-	-
1,2,3,7,8,9-HxCDD	1.1	0.12	-	0.98 (≤5.3)	-	-

	Concentration (pg/g)		RPD	Difference		
Compound	SSAJ3-02-1BPC**	SSAJ3-02-1BPC_FD	(Limits)	(Limits)	Flags	A or P
1,2,3,4,6,7,8-HpCDD	1.6	0.41	-	1.19 (≤5.3)	<del>-</del>	-
OCDD	5.9	1.4	-	4.5 (≤11)	-	-
2,3,7,8-TCDF	0.90	0.60	<del>-</del>	0.3 (≤1.1)	-	-
1,2,3,7,8-PeCDF	2.1	0.69	-	1.41 (≤5.3)	<del>-</del>	-
2,3,4,7,8-PeCDF	1.5	0.39	-	1.11 (≤5.3)	•	-
1,2,3,4,7,8-HxCDF	1.7	1.2	-	0.5 (≤5.3)	-	-
1,2,3,6,7,8-HxCDF	2.2	0.76		1.44 (≤5.3)	<del>-</del>	-
2,3,4,6,7,8-HxCDF	1.5	0.16	-	1.34 (≤5.3)	<u>-</u>	-
1,2,3,7,8,9-HxCDF	1.4	0.18	_	1.22 (≤5.3)	-	-
1,2,3,4,6,7,8-HpCDF	3.2	2.7	-	0.5 (≤5.3)	-	-
1,2,3,4,7,8,9-HpCDF	2.1	0.99	_	1.11 (≤5.3)	-	-
OCDF	9.1	7.8	-	1.3 (≤11)	-	-

	Concentration (pg/g)		DDD	D.24		
Compound	SSAN7-02-1BPC	SSAN7-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	2.4	2.4	•	0 (≤1.1)	-	-
1,2,3,7,8-PeCDD	11	10	-	1 (≤5.3)	-	-
1,2,3,4,7,8-HxCDD	6.6	5.3	-	1.3 (≤5.3)	-	-
1,2,3,6,7,8-HxCDD	15	12	-	3 (≤5.3)	-	-
1,2,3,7,8,9-HxCDD	11	8.6	-	2.4 (≤5.3)	-	-
1,2,3,4,6,7,8-HpCDD	39	35	11 (≤50)	-	-	-

	Concentra	RPD	Difference			
Compound	SSAN7-02-1BPC	SSAN7-02-1BPC_FD	(Limits)	(Limits)	Flags	A or P
OCDD	40	35	-	5 (≤11)	-	-
2,3,7,8-TCDF	130	170	27 (≤50)	-	-	-
1,2,3,7,8-PeCDF	160	210	27 (≤50)	-	-	-
2,3,4,7,8-PeCDF	77	82	6 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	230	250	8 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	200	160	22 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	44	35	23 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	42	21	-	21 (≤5.3)	J (all detects)	А
1,2,3,4,6,7,8-HpCDF	500	440	13 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	270	220	20 (≤50)	-	-	-
OCDF	1300	1200	8 (≤50)	_	-	-

#### Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D140422

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D140422	SSAJ3-02-1BPC** SSAM7-03-1BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0D140422	SSAH3-01-1BPC** SSAN7-02-1BPC SSAN6-06-1BPC	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)	Р	Internal standards (%R) (i)
G0D140422	SSAM7-03-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D140422	SSAM7-04-1BPC	OCDF	J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D140422	SSAN6-06-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D140422	SSAJ3-02-1BPC** SSAH3-01-1BPC** SSAM7-03-1BPC SSAM7-04-1BPC SSAN7-02-1BPC SSAN6-06-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D140422	SSAJ3-02-1BPC** SSAH3-01-1BPC** SSAM7-03-1BPC SSAM7-04-1BPC SSAN7-02-1BPC SSAN6-06-1BPC	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)
G0D140422	SSAN7-02-1BPC	1,2,3,7,8,9-HxCDF	J (all detects)	А	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D140422

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D140422

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D140422

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: 23188C21	VALIDATION COMPLETENESS WORKSHEET
SDG #: G0D140422	Stage 2B/4
Laboratory: Test America	

Date:	5/11/10
Page:_	<u>/</u> of_/_
Reviewer:	Q
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
1.	Technical holding times	A	Sampling dates: 4/10
11.	HRGC/HRMS Instrument performance check	A	/
111.	Initial calibration	₩	
IV.	Routine calibration/i	A	
V.	Blanks	-A	2
VI.	Matrix spike/Matrix spike duplicates	SW	No spassid - No Gual
VII.	Laboratory control samples	<b>A</b>	209
VIII.	Regional quality assurance and quality control	N,	
IX.	Internal standards	W	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	w	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	1	
XIV.	Field duplicates	W	D=1+SSAJ3-02-1BPC_FD, 5+SSAN7-02-1BPC_FD(G0D140543)
XV.	Field blanks	W	ρο FB-04072010-RZD(G0D090441),FB-040710-RZC(G0D130519) EB-04122010-RIG3-RZD(G0D140534)

Note:

A = Acceptable

ND = No compounds detected

D = Duplicate

N = Not provided/applicable

R = Rinsate

TB = Trip blank

SW = See worksheet

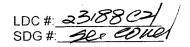
FB = Field blank

EB = Equipment blank

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

1 12	SSAJ3-02-1BPC**	11	0/0536MB	21	31	
2 <i>f</i> )	SSAH3-01-1BPC**	12		22	32	
3	SSAM7-03-1BPC	13		23	33	
4	SSAM7-04-1BPC	14		24	34	
5	SSAN7-02-1BPC	15		25	 35	
6	SSAN6-06-1BPC	16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	 39	
10	11-2	20		30	40	

Notes:		

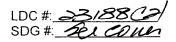


#### **VALIDATION FINDINGS CHECKLIST**

Page: /of AREVIEWER: 2nd Reviewer: \_\_\_\_

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

Validation Area	Yes	No	NA	Findings/Comments '
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?				
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?		,		
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?				
IV. Continuing calibration				
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?		اي		
V. Blanks				
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?		,		
VI. Matrix spike/Matrix spike duplicates			77.4	
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?				
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



#### **VALIDATION FINDINGS CHECKLIST**

Page:_	<u> 구of                                   </u>
Reviewer:	<u>q</u> _
2nd Reviewer:	· · · · · · · · · · · · · · · · · · ·

0				
VIII. Regional Quality Assurance and Quality Control	<del>,</del>	<del>,</del>	<u>, ,                                   </u>	T
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?			<u></u>	
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?	6			
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?	1			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	(			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?			/	
Was an acceptable lock mass recorded and monitored?				
XI. Compound quantitation/CRQLs	· · · · · ·			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII. System performance	مر			
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. ocpp	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:

SDG #: See Cover LDC #: 23188C21

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Page:\_ Reviewer: 2nd Reviewer:

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

Associated sample units: Y N/A Were field blanks identified in this SDG?

Blank units: pg/L / Sampling date: 4/12/10

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

Compound	Blank ID		S	Sample Identification	ion		
	FB-04122010-RIG3-RZD	5X					
D	0.64	0.0032					
ш	0.47	0.00235					
ш	1.9	0.0095					
9	5.0	0.025					
Ξ	4.6	0.023					
	3.8	0.019					
7	2.2	0.011					
<b>Y</b>	7.7	0.0385					
7	4.8	0.024					
M	1.2	900.0					
Z	0.59	0.00295					
0	14	0.07					
Ь	5.0	0.025					
Ø	27	0.135					
CRQL							

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

LDC #: 23188C21

SDG #:See Cover

**VALIDATION FINDINGS WORKSHEET** 

**Field Blanks** 

Page: of 2nd Reviewer:\_ Reviewer:

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

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

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

b/bd Sampling date: 4/7/10

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

Compound	Blank ID			Sai	Sample Identification	ıtion		
14 4 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	FB-04072010-RZD	2X						
	0.89	0.00445						
	1.5	0.0075						
	2.2	0.011						
	8.3	0.0415						
	1.4	0:00						
	1.6	800'0						
	1.5	0.0075						
	1.6	0.008						
	1.3	0.0065						
	1.4	200'0						
	1.4	0.0205						

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

WildellTransvIEOCIED DIOVINE D70 ....

LDC#23/8802 SDG #:See Cover

# VALIDATION FINDINGS WORKSHEET

Field Blanks

2nd Reviewer: Page: Reviewer:

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

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

Blank units: pg/L Associated sample units

Sampling date: 4/8/10

Associated sample units:\_

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

1 Associated Samples:

	אייייייייייייייייייייייייייייייייייייי	7 Olligi.	Associated Samples:	
Compound	Blank ID		Sample Identification	
Turk.	EB-04072010-RZC	5x		
O	0.77	0.00385		
D	0.74	0.0037		
Е	0.82	0.0041		
<b>4</b>	4.2	0.021		
9	37	0.185		
Н	0.57	0.00285		Ī
	96:0	0.0048		
	0.67	0.00335		
×	1.1	0.0055		
	0.96	0.0048		
M	1.0	0.005		
Z	1.0	0.005		
0	2.1	0.0105		
	1.5	0.0075		
٥	6.7	0.0335		
				Ī
CRQL				

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

LDC #: \$1886 SDG #:20

## **VALIDATION FINDINGS WORKSHEET** Internal Standards

Page: / of Reviewer: 0 2nd Reviewer:

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".

YAN N/A
Are all internal standard recoveries were within the 40-135% criteria?
Y N N/A
Was the S/N ratio all internal standard peaks > 10?

1						
**	Date	Lab ID/Reference	Internal Standard	% Recove	% Recovery (Limit: 40-135%)	Qualifications
-			eb	ba	(40-135	1 JULY (4. 8)
├-		0				
$\vdash$						
$\vdash$		٨	4		•	) (F-G. 0-K)
$\vdash$			<del>                                     </del>	00	)	
$\Vdash$				5	)	
$\vdash$					)	
$\vdash$		6	Н	755	)	
H						
					)	
$\vdash$		いな	<b>34</b>	35		
$\vdash$		1	#	36	)	)
+				2	)	
十					)	
$\vdash$		1	<i>A</i>	8	)	
$\vdash$			#	36		
╟			(	74	<b>^</b>	
T				,	)	
$\vdash$						
$\vdash$					)	
$\Box$						
$\dashv$					)	
		Internal Standards	Check Standard Used		Internal Standards	Check Standard Used
Ą	13C-2,3,7,8-TCDF	ODF		G. <sup>13</sup> C-1,2,3	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	
9	13C-2,3,7,8-TCDD	ada		H. <sup>13</sup> C-1,2,3	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	
ن	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF		1. 13C-OCDD	Q	
a	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		K. <sup>13</sup> C-1,2,3	<sup>13</sup> C-1,2,3,4-TCDD	
ш	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	8-HxCDF		L. 13C-1,2,3	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
П	13C-1,2,3,6,7,8-HxCDD	8-HxCDD				

LDC #: 35188027 SDG #: 2823000

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of / Reviewer:

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 We

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 Finding	Associated Samples	Qualifications
		<b>N</b>	0 8		1/2/4(e)
		4	X		
		9	4. I.K. 4.0 P. 8		
		M	zuto usulto		(#1 (F)
			(x toa)		
	0		_ / _		

Comments: See sample calculation verification worksheet for recalculations

LDC#:<u>23188C21</u> SDG#: <u>See Cover</u>

### VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: de_	
Reviewer:	
2nd Reviewer:	

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

YN NA YN NA

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

	Concentra	ation (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	1	SSAJ3-02-1BPC_FD	RPD	Difference	Limits	(Parent Only)
А	0.22	1.1U		0.88	( <u>&lt;</u> 1.1)	
В	0.90	5.3U		4.4	( ≤5.3)	
С	0.99	5.3U		4.31	( ≤5.3)	
D	1.2	0.13		1.07	( <5.3)	
E	1.1	0.12		0.98	( <5.3)	
F	1.6	0.41		1.19	( <u>&lt;</u> 5.3)	
G	5.9	1.4		4.5	( ≤11)	
Н	0.90	0.60		0.3	( ≤1.1)	
ı	2.1	0.69		1.41	( ≤5.3)	
J	1.5	0.39		1.11	( <5.3)	
к	1.7	1.2		0.5	( ≤5.3)	
L	2.2	0.76		1.44	( ≤5.3)	
м	1.5	0.16		1.34	( ≤5.3)	
N	1.4	0.18		1.22	( <u>&lt;</u> 5.3)	
0	3.2	2.7		0.5	( ≤5.3)	
Р	2.1	0.99		1.11	( ≤5.3)	
Q	9.1	7.8		1.3	( <u>&lt;</u> 11)	

	Conc	entration (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	5	SSAN7-02-1BPC_FD	RPD	Difference	Limits	(Parent Only)
А	2.4	2.4		0	( ≤1.1)	
В	11	10		1	( ≤5.3)	
С	6.6	5.3		1.3	( ≤5.3)	
D	15	12		3	( ≤5.3)	
E	11	8.6		2.4	( ≤5.3)	
F	39	35	11			
G	40	35		5	( ≤11)	

LDC#: <u>23188C21</u> SDG#: <u>See Cover</u>

### VALIDATION FINDINGS WORKSHEET <u>Field Duplicates</u>

Page: of	
Reviewer:	
2nd Reviewer:	

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

YN NA YN NA

Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concenti	ation (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	5	SSAN7-02-1BPC_FD	RPD	Difference	Limits	(Parent Only)
Н	130	170	27			
ı	160	210	27			
J	77	82	6	i		
к	230	250	8			
L	200	160	22			
М	44	35	23			
N	42	21		21	( <5.3)	deta/
0	500	440	13			/
Р	270	220	20			
α .	1300	1200	8			

(+d)

V:\FIELD DUPLICATES\23188C21.wpd

UC #: 0//5 SDG #: 767

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

₹ Page: Reviewer:

2nd Reviewer:

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 =  $(A_u)(C_u)/(A_u)(C_v)$ average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

A<sub>x</sub> = Area of compound,
C<sub>x</sub> = Concentration of compound,
S = Standard deviation of the RRFs,

 $A_{\bf k}$  = Area of associated internal standard  $C_{\bf k}$  = Concentration of internal standard X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF ( CSStd)	RRF (CS3 std)	%RSD	%RSD
	19th	0/10/1	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	0.860	0.860	18.0	0.87	10.4	106
	(105)	2/4//	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	4560	0.934	0.96	56.0	00	12.8
		-1	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.058	1.058	00.	601	7/,7	11.0
			1,2,3,4,6,7,8-HpCDD (¹³C-1,2,4,6,7,8,-HpCDD)	8660	0.998	20.1	501	٧.٩/	12.2
			OCDF (1°C-OCDD)	1.437	1.437	1.52	<5.1	141	07/
~	1012	1/2./	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.088	880./	1.10	01.1	& si	ar'
		1/1/2	2,3,7,8-TCDD ( <sup>13</sup> C-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 (1ºC-OCDD)						
၈	iste	/ //	2,3,7,8-TCDF ("C-2,3,7,8-TCDF)	3460	0,945	86.0	86.0	4.44	4 33
	(4BB)	01/2/14	2,3,7,8-TCDD ( <sup>18</sup> C-2,3,7,8-TCDD)	1001	1.07	1.04	104	202	780
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.114	0/:/	0 !:	533	3.2
		7	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	CL0:1	1.072	// : /	///	3.60	3.75
			OCDF ("C-OCDD)	11/12	11111	141	1	601	000 (

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



# VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF = (A,)(C<sub>b</sub>)/(A<sub>b</sub>)(C<sub>c</sub>)

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

 $A_x$  = Area of compound,  $A_k$  = Area of associated internal standard  $C_x$  = Concentration of compound,  $C_k$  = Concentration of internal standard

L					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>0%</b>	Q%
-	26APVOAIDS	04105 11,61.	2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	0.860	560	0.92	7.0	0.7
		4/10	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	0.934	0.93	0.93	1.0	0.
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	8501	1.15	1.15	<i>v</i> .	8.0
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	8660	1.06	1.06	1	6.2
			OCDF (3C-OCDD)	1.437	7.5.1	75.1	1.7	40
7	2/4P1465	1/22/11	2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	6.945	760	0.47	3./	.w./
		0///	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.02	760	76.0	7.57	13.7
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	7117	0/./	01:1	4.	7
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	40.1	1.04	0.0	8
			OCDF (1°C-OCDD)	1.445	1.4>	1 45	0.0	0.0
က	38 APICEDO	4/28/1°	2,3,7,8-TCDF (¹°C-2,3,7,8-TCDF)	1.084	1.09	601	0.3	4.0
	\	/	2,3,7,8-TCDD (13C-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 (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (°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.



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

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Page:	Reviewer:_	2nd Reviewer:

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy 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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: DID SON

RPD = ILCS - LCSD I \* 2/(LCS + LCSD)

		24	S Polico	olame	85-		uso i		I CS/I CSD	csp
Tri Control	Added	) let	Concentration	ration	Percent Recovery	PCOVERV	Percent Recovery	ecoverv	RPD	٥
	801	1.560	2	g I Csn	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	0.00	NA	4. R	本	201	102				
1,2,3,7,8-PeCDD	(0)		109		109	109				
1,2,3,4,7,8-HxCDD			7.88		68	89				
1,2,3,4,7,8,9-HpCDF			88.2		× × × × × × × × × × × × × × × × × × ×	88				
OCDF	SON		500		<b>(0)</b>	102				
				-						
	-			-						

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

Ol nol
M C <sub>12</sub> H <sub>3</sub> 3Cl <sub>4</sub> O M+2 C <sub>12</sub> H <sub>3</sub> 3Cl <sub>0</sub>
N
M   C <sub>12</sub> H <sub>2</sub> <sup>2</sup> Cl <sub>1</sub> O <sub>2</sub> M+2   C <sub>12</sub> H <sub>2</sub> <sup>2</sup> Cl <sub>3</sub> <sup>2</sup> C1 <sub>0</sub>
M+2 C <sub>12</sub> H <sub>3</sub> 3C <sub>1</sub> C <sub>1</sub>
M+2   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> 7'ClO M+4   C <sub>12</sub> H <sub>3</sub> *Cl <sub>4</sub> 7'Cl <sub>2</sub> O
M+2 C;H,3C[,1C],C
M+4 C <sub>12</sub> H <sub>3</sub> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub>
M+2 C12H3*C18,7C10
M+4   C <sub>12</sub> H <sub>2</sub> 3C  <sub>4</sub> 37C  <sub>2</sub> O M 13C <sub>2</sub> H <sub>3</sub> C  <sub>2</sub> O
M+2 C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO <sub>2</sub> M+4 C <sub>14</sub> *Cl <sub>3</sub> *ClO <sub>3</sub>
M+4 13C <sub>12</sub> H <sub>2</sub> 35C <sub>1</sub> 37C <sub>12</sub> O <sub>2</sub> M+4 C H 35C <sub>1</sub> 37C <sub>1</sub> O
:

The following nuclidic masses were used: Ē

O = 15.994915 $^{3}CI = 34.968853$  $^{3}CI = 36.965903$ 

H = 1.007825 C = 12.000000  $^{13}C = 13.003355$  F = 18.9984

S = internal/recovery standard

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	Page:

### VALIDATION FINDINGS WORKSHEET

SDC LDC #: 2388

.ylno

Verification

Sample Calculation	#: 268 CONO!
VACIDATION FINDINGS	10005P:#

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

	Percent solids, applicable to soil and solid matrices	=	S%
	Dilution Factor.	=	ΙO
B/4d 55 1=	Relative Response Factor (average) from the initial calibration		ARF
	Volume or weight of sample extract in milliliters (ml) or grams (g).		°۸
(0000) (0+829E) = .000	smangonan ni bəbba brabnata lanretni to truomA (gn)	=	s.l
725 72	Area of the characteristic ion (EICP) for the specific internal standard		<sub>ai</sub> A
Sample I.D.	Area of the characteristic ion (EICP) for the compound to be measured		×̈Υ
Example:	( <del>7</del> 0)(,)(, <u>A)</u> = (공%)(,V)(귀위기( <sub>a</sub> A)	= noitatr	neonoO
target compounds agree within 10.0% of the reported results?	Were all recalculated results for detected	<b>∀/</b> N	N N /
berified for all level IV samples?	Were all reported results recalculated and	A\v	1 N /

		·			
	* 12.118 A. N.				
noitsoitilsuD	Colculated Concentration (	Peported noitstinencO (	Сотроипа	Gl elqmε∂	#

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 12, 2010

LDC Report Date:

May 24, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D140526

Sample Identification

RSAI3-12BPC RSAI3-12BPCMS

RSAI3-12BPCMSD

#### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

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

Instrument performance was checked at the required daily frequency.

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

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition).

#### III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound for samples.

#### IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

#### V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0109260MB	4/19/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	0.14 pg/g 0.59 pg/g 0.28 pg/g 0.14 pg/g 0.24 pg/g 0.14 pg/g 0.086 pg/g 0.30 pg/g 0.13 pg/g 0.63 pg/g	All samples in SDG G0D140526

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

Sample EB-04122010-RIG3-RZD (from SDG G0D140534) was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG3-RZD	4/12/10	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-PeCDF 1,2,3,6,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	0.64 pg/L 0.47 pg/L 1.9 pg/L 5.0 pg/L 4.6 pg/L 2.2 pg/L 7.7 pg/L 4.8 pg/L 1.2 pg/L 0.59 pg/L 14 pg/L 5.0 pg/L 27 pg/L	All samples in SDG G0D140526

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 1,2,3,4,6,7,8-HpCDF	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 1.6 pg/L 1.7 pg/L 1.9 pg/L 1.9 pg/L 1.1 pg/L	All samples in SDG G0D140526

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

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

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

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

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

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

Not applicable.

#### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
RSAI3-12BPC	<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) 22 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P

#### X. Target Compound Identifications

All target compound identifications were within validation criteria.

#### 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
RSAI3-12BPC	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)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140526	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140526	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	А

#### XII. System Performance

The system performance was acceptable.

#### XIII. Overall Assessment of Data

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

#### XIV. Field Duplicates

No field duplicates were identified in this SDG.

#### Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D140526

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D140526	RSAI3-12BPC	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)	Р	Internal standards (%R) (i)
G0D140526	RSAI3-12BPC	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)	Р	Compound quantitation and CRQLs (e)
G0D140526	RSAI3-12BPC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0D140526	RSAI3-12BPC	All compounds reported as EMPC	JK (all detects)	А	Project Quantitation Limit (k)

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D140526

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D140526

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D140526

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson

DC #: 23188D21	VALIDATION COMPLETENESS WORKSHEET	
SDG #: G0D140526	Stage 4	
Laboratory: Test America		F
		2nd F

	Date:	5/1/10
	Page:_	/of
	Reviewer:_	9-
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	$\triangleleft$	Sampling dates: 4/13/16
11.	HRGC/HRMS Instrument performance check	1	/ /
III.	Initial calibration	1	
IV.	Routine calibration/)	A	
V.	Blanks	M	
VI.	Matrix spike/Matrix spike duplicates	M	
VII.	Laboratory control samples	A	109
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	w	
Χ.	Target compound identifications	\$	
XI.	Compound quantitation and CRQLs	m	
XII.	System performance	1	
XIII.	Overall assessment of data	1	
XIV.	Field duplicates	N	
XV.	Field blanks	W	ZB-04/226/0-RI43-RZD(GOD140534)
lote:	A = Acceptable ND = N	o compound	ZB-04/226/0-RIG3-RZD(G0D140534)  FB-040726/0-RZD(G0D090441)  S detected D = Duplicate  TR - Trip blank

N = Not provided/applicable SW = See worksheet

R = Rinsate

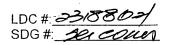
FB = Field blank

TB = Trip blank EB = Equipment blank

Validated Samples:

1	RSAI3-12BPC	S	11	0/09260143	21	31	
2	RSAI3-12BPCMS	}	12		22	32	
3	RSAI3-12BPCMSD	V	13		23	33	
4			14		24	34	
5			15		25	35	
6			16		26	36	
7			17		27	37	
8			18		28	38	
9			19		29	39	
10			20		30	40	

Notes:_					
		 •			

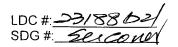


#### **VALIDATION FINDINGS CHECKLIST**

	Page:_	
	Reviewer:	9
2nd	Reviewer:	

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times	·	,		
All technical holding times were met.	/			·
Cooler temperature criteria was met.	/			
II. GC/MS Instrument performance check	·			
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?	/			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and < 30% for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?				
IV. Continuing calibration				
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?				
V Blanks				
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?		,		
VI. Matrix spike/Matrix spike duplicates				
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?			-	
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



#### **VALIDATION FINDINGS CHECKLIST**

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

T				
VIII. Regional Quality Assurance and Quality Control		· · · · ·	<del>, .</del>	
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?			$\perp$	ł
IX. Internal standards		· · · · ·	<del></del>	<b>T</b>
Were internal standard recoveries within the 40-135% criteria?	<u> </u>	/	<u> </u>	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	/	<u> </u>	<u> </u>	
X. Target compound identification	Т	т	1	Ţ
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?	(		<u> </u>	
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?	/		ļ	
Does the maximum intensity of each specified characteristic ion coincide within $\pm2$ seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?		-		
Was an acceptable lock mass recorded and monitored?	/_			
XI. Compound quantitation/CRQLs			1	
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.		٠		
XIII. Overall assessment of data			7 e	
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.		<i></i>		
Target compounds were detected in the field blanks.			-	

# VALIDATION FINDINGS WORKSHEET

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

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

### Notes:

LDC #: 23188D21

SDG #: See Cover

### VALIDATION FINDINGS WORKSHEET **Blanks**

Page: 1 of 1

Reviewer: 2nd Reviewer:

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

Pease see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Were all samples associated with a method blank? Y 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. N/N N/A

Associated samples: Blank analysis date: 4/25/10 Blank extraction date: 4/19/10 Conc. units:  $pg/L \, \ell_{\gamma}$ 

AII (>5X)

Sample Identification 3.15 2.95 0.43 0.65 0.7 1.2 0.7 1.5 0.7 섫 0109260MB Blank ID 0.086 0.14 0.59 0.28 0.14 0.14 0.30 0.13 0.63 0.24 Compound 0 z

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 #: 23188D21 SDG #: See Cover

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Reviewer: Page:\_ 2nd Reviewer:

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

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

Blank units: pg/L Associated sample units

b/gd Associated sample units:

Sampling date: 4/12/10

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

Associated Samples:

Sample Identification 0.00295 0.0032 0.00235 0.0095 0.0385 0.025 0.019 0.023 0.011 0.024 900.0 0.025 0.135 0.07 X EB-04122010-RIG3-RZD Blank ID 0.64 0.47 0.59 1.9 4.6 5.0 3.8 4.8 5.0 2.2 1,2 7.7 4 27 Compound G Ш Z

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

LDC #: 23188D21 SDG #:See Cover

# VALIDATION FINDINGS WORKSHEET

Field Blanks

Reviewer:\_ 2nd Reviewer:

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

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

b/bd Associated sample units: Blank units: pg/L

Sampling date: 4/7/10

メタイプ Sample Identification Associated Samples: 0.00445 0.0415 0.0075 0.0075 0.0205 0.0065 0.011 0.007 0.008 0.008 0.007 Field blank type: (circle one) Field Blank / Rinsate / Other: 젊 FR-04072010-RZD Blank ID 0.89 5. 1.6 7.5 1.6 2.2 8.3 1.4 <u>ს</u> 4.1 4.1 Compound മ

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

+ 0::

LDC #: 23/8862/ SDG #: 20x 2011

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: of Reviewer: 2nd Reviewer:

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

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

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

associated MS/MSD. Soil / Water.

Was a MS/MSD analyzed every 20 samples of each matrix?

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

		/								_		<u> </u>		Π	Ī				Ī	Ī	<del>-</del>	Π	Γ
Qualifications	Notains																						
Associated Samples	1	-																					
RPD (Limits)	de to to	( )	( )	_	( )	( )		( )	( )	(		( )	( )	( )	(	( )	( )	(	)	( )	<u> </u>	( )	
MSD %R (Limits)	all ( and s)	( )	(	( )	( )	)		( )	( )	(	_	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	(	
MS %R (Limits)	`	( / )	( )	( )	( )	( )	( )	( )	<u> </u>	-		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
Compound	70 K																						
ai asw/sm	· 3/3																						
# Date																							

LDC #23680 SDG # W CON

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

Page: / Reviewer:\_ 2nd Reviewer:\_

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".

YAN 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
			¥	(S)	LOF)	1351	1/N A (F-40-A
T			¥	3	)	)	
T			/	20	)	1	•
					)	/ )	
		SMSD)	4	36	/ )	)	Noteax
			<del>/</del>	7	$A \rightarrow$	(	7
T					)	(	
T					)	(	
T					)	(	
T					)	(	
					)	,	
T					)	ſ	
1					)	)	
T					)	^	
Γ					)		
T						)	
					. )	)	
П							
					)	7	
					)		
					)	)	
		Internal Standards	Check Standard Used		Internal Standards	dards	Check Standard Used
Ą	13C-2,3,7,8-TCDF	ODF		G.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF		
æi	<sup>13</sup> C-2,3,7,8-TCDD	CDD		Ξ	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD		
رن ن	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF			13C-OCDD		
o.	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		작	<sup>13</sup> C-1,2,3,4-TCDD		
ய்	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	8-HxCDF		ا د	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
ц	13C-123678-HxCDD	8-нхСПП					

LDC #:023/88/80

### Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Reviewer:

2nd Reviewer: \_\_

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

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

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

	Date	Sample ID	Spales > Called Marso	Associated Samples	Qualifications
FI 1		/	X, O, B, &	\	John A(e)
		Rel	ZNPC LEWE		(4) 47
			(8/-1/ac)		
. 7					
1					
	0				
-					
1					

Comments: See sample calculation verification worksheet for recalculations

### SDG #: 56 COND

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

	4	
Page:	Reviewer:	2nd Reviewer
		2nd

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 =  $(A_{\nu})(C_{\mu})/(A_{\mu})(C_{\nu})$ 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,

 $A_{\bf k} = {\sf Area~of~associated~internal~standard~C_{\bf k}} = {\sf Concentration~of~internal~standard~X} = {\sf Mean~of~the~RRFs}$ 

L									
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (A)	RRF	%BSD	
-	1941		2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0 945	0 945	800	0 100	777	40 V
	(40E)	1/1/2	23.7.8-TCDD ( <sup>13</sup> C-23.7.8-TCDD)		1	200	0000	1.1	4 2/2
		1770	יייייייייייייייייייייייייייייייייייייי	- 0	.02	+0.	1.04	3.03	2.97
		. /	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	4   1	1.11	6-	0	5.33	50.5
			1,2,3,4,6,7,8-HpCDD (19C-1,2,4,6,7,8,-HpCDD)	1.0T2	1.072	1.1.1		3.60	3.75
			OCDF (13C-OCDD)	1.445	1.45	ſ.	.4	1 8C	08.8
7	1str	3/0/2	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	000	000	201	00-	R	X 55 /
		1/2/2	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 (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (%C-OCDD)						
က	10A2	/ - / .	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.088	1.10	01.1	60-	04
	(SD2)	4/1/10	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 (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF ("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.

LDC #: 2/88024

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of 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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$ 

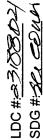
Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF  $A_x = Area$  of compound,  $C_x = Concentration$  of compound,

 $A_{\mathbf{k}} = Area$  of associated internal standard  $C_{\mathbf{k}} = Concentration$  of internal standard

<u> </u>					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
	21 PIONE	/ / / /	2,3,7,8-TCDF (18C-2,3,7,8-TCDF)	0.945	0.90	0.90	<i>4</i> . <i>y</i>	4.3
	,	4/2/10	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.02	0.96	26:0	Ø. W	NO W
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.114	1.08	80/	W.	8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1072	1.03	1.03	8.0	3.9
		-	OCDF ("C-OCDD)	1.445	1.36	1.36	2.0	5. X
7	KNEO	14 C/3/1)	2,3,7,8-TCDF (*3C-2,3,7,8-TCDF)	0.945	293	6.6.0	V.Y	ď,
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.02	095	26.0	000	XX
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	7.11	1.15	/:/ \$/	13.4	3.6
			1,2,3,4,6,7,8-HpCDD (°C-1,2,4,6,7,8,-HpCDD)	1.072	1.0.1	1.0.1	44	75
			OCDF (1°C-OCDD)	1.445	1.40	1.40	24	34
က	O/M/JOACT	4410	2,3,7,8-TCDF ( <sup>3</sup> C-2,3,7,8-TCDF)	1.088	7//	7.7	2,8	N
	/_/	1.1	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-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.



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

Reviewer: 9 Page:∠ 2nd Reviewer:\_\_

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

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

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

MS/MSD samples: \_

V)

RPD = I MSR - MSDR I \* 2/(MSR + MSDR)

	S	ike	Sample	Spiked Sample	ample	Matrix Spike	Snike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Compound	A A	Added (22/9)	Concentration (PS/Q)	Concentration (FF)	tration	Percent Recovery	Recovery	Percent Recovery	ecovery	RPD	RPD
	MS	MSD		/ SM	MSD	Reported	Recalc	Reported	Recalc	***************************************	3 P 4 A
2,3,7,8-TCDD	1./6	20.3	5/0	488	436	0	0	0	0	0	0
1,2,3,7,8-PeCDD	501	101	1700	07/21	15-30	\$33	38	0	0	0	13
1,2,3,4,7,8-HxCDD	1		doo	1570	e& //	161	163	0	0	0	28
1,2,3,4,7,8,9-HpCDF	1	1	62000	00	64100	0911	_	2430	800	71	14
OCDF	1/6	203	3/0000	4/000	9681 000 pag 58 000	2000	1896	0	0	0	6
											_

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.

Willalidation Modelshaptal Dissingmine Michigan Con 34

LDC #: 2318807 SDG #46. COND

# 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 laboratoy 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: SS(

Where: SSC = Spiked sample concentration SA = Spike added

RPD = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0/09>60

SD		Recalculated									
I CS/I CSD	RPD	Reported						***			
Q	ecovery	Recalc									
ICSD	Percent Recovery	Reported									
<i>S</i>	ecovery	Recalc	60	102	108	104	101				
ICS	Percent Recovery	Reported	66	102	8 01	104	101				
ample	a tion	I CSD	NA					_			
Spiked S	Concentration	108	19.8	102	801	104	400				
, ik	Added'	l CSD	NA				->				
8	Adç	1.08	0'02	001	1		2191)				
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF				

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

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Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte
-	303.9016	Σ	O,D*,H,30	TCDF	4	407.7818	M+2	C.H <sup>®</sup> Cl. <sup>37</sup> ClO	HDCDF
	305.8987	M+2	C <sub>12</sub> H <sub>3</sub> 35Cl <sub>3</sub> 37C10	TCDF		409.7788	₩ + ₩	C,17**C ,3'C ,0	HDCDF
•	315.9419	Σ	0,10 %,H 2,10 %	TCDF (S)		417.8250	Σ	13C,H2CI,O	HpCDF (S)
	317.9389	M+2	*3C12H4*5C13*7CIO	TCDF (S)		419.8220	M+2	13C1,H35C1,37C1O	HPCDF
	319.8965	<b>∑</b>	C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C, Hack Too	HPCDD
	321.8936	M+2	C12H4**C13**C102	TCDD		425.7737	M+4	C,H*Cl, 37Cl,O,	Hecdo
	331.9368	Σ	13C12H, 35C1, O2	TCDD (S)		435.8169	M+2	13C, H <sup>ac</sup> Cl, 37ClO,	HPCDD (S)
	333.9338	M+2	13C <sub>12</sub> H <sub>4</sub> 35Cl <sub>3</sub> 37ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	13C,174c,12,17C,1.0,	HeCDD (S)
	375.8364	M+2	C <sub>1</sub> ,H, <sup>36</sup> Cl, <sup>37</sup> ClO	HXCDPE		479.7165	M+4	O. H. C. 17 C. O	NCDPF
	[354.9792]	LOCK	C,FT,2	PFK		[430.9728]	LOCK	C 15	PFK
2	339.8597	M+2	C,,H,35CI,37CIO	PeCDF	5	441,7428	M+2	0.350137010	# COO
	341.8567	M+4	C,H,**C1,*7C1,O	PeCDF		443.7399		C12 37 C1 C	- LOCO
	351,9000	M+2	<sup>13</sup> C, H, <sup>35</sup> Cl, <sup>37</sup> C10	PeCDF (S)		457.7377		C12 C16 C12 C. 35CL 37CIO.	0000
	353.8970	M+4	13C, H, 35Cl, 37Cl, O	PeCDF (S)		459.7348		C12 C1 C102	ממט
	355.8546	M+2	C, H, scl, sclo,	PeCDD		469.7780		13C 35C 37CIO	(8) (4)
	357.8516	M+4	C,H,*Cl,*7Cl,O,	PecDD		471.7750	. + +	13C, 35C, 37C, O.	(5)
	367.8949	M+2	13C, H, 45Cl, 37ClO,	PeCDD (S)		513.6775		C. 36[-370].O	DCOPE (C)
	369.8919	M+4	13C1,H,36C1,37C1,O,	PecDD (S)		[422.9278]		C.F. (120)	PFK
	409.7974	M+2	C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> C <sub>1</sub>	HPCDPE				-10.12	-
	[354.9792]	LOCK	0 F 1	PFK					
ဗ	373.8208	M+2	C,H, #Cl, 7/CIO	HXCDF					
	375.8178	4	C12H235CI_37CI_O	HXCDF					
	383.8639		13C <sub>12</sub> H <sub>2</sub> 3Cl <sub>8</sub> O	HxCDF (S)					
	385.8610		13C12H235CI537CIO	HXCDF (S)		•			
	389.8156		C <sub>12</sub> H <sub>2</sub> 35Cl <sub>5</sub> 37ClO <sub>2</sub>	HXCDD				-	
	391.8127	¥+₩	C <sub>12</sub> H <sub>2</sub> *C  <sub>4</sub> *C  <sub>2</sub> O <sub>2</sub>	HXCDD					
	401.8559		<sup>13</sup> C <sub>12</sub> H <sub>2</sub> *Cl <sub>5</sub> *7ClO <sub>2</sub>	HXCDD (S)					-
	403.8529 445.7555	M+4	1.C <sub>12</sub> H <sub>2</sub> .3C <sub>1</sub> 3C <sub>1</sub> O <sub>2</sub>	HXCDD (S)		-		_	
	[430,9728]		O. 12 12 O.	מיני אינים					
	[02 (0:001]		C <sub>8</sub> -17	_ <b>_</b>					
									=

The following nuclidic masses were used: <u>@</u>

 $^{36}Cl = 34.968915$  $^{37}Cl = 34.968953$ 

H = 1.007825 C = 12.000000 <sup>13</sup>C = 13.003355 F = 18.9984

S = internal/recovery standard

LDC #: 03/8802/ SDG #: 988 COM

### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:	/of /
Reviewer:	4
2nd reviewer:_	

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

Y	N	N/A
Y	N	N/A
V		

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?

Example:

Concent	ration	$= \frac{(A_s)(I_s)(DF)}{(A_h)(RRF)(V_o)(\%S)}$
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <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.

Sample I.D/	A	:
Conc. = <u>4/134</u>	3 (102)	10.56 10.98
	1 pg/g	•

I			····		0-11-4	
				Reported Concentration	Calculated Concentration	
#	Sample ID	Compound		( )	( )	Qualification
		·		· · · · · · · · · · · · · · · · · · ·		
						****
						<u> </u>
			<u>-</u>			

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 12, 2010

LDC Report Date:

May 26, 2010

Matrix:

Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D140534

Sample Identification

EB-04122010-RIG3-RZD

EB-04122010-RIG2-RZB

EB-04122010-RIG1-RZB

### Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

### III. Initial Calibration

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

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

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

### IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
4/26/10	1,2,3,4,7,8-HxCDD	20.4	0110418MB	1,2,3,4,7,8-HxCDD	J- (all detects) UJ (all non-detects)	Р

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
0110418MB	4/20/10	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 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HyCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 0CDF	1.3 pg/L 1.3 pg/L 1.8 pg/L 2.7 pg/L 11 pg/L 1.8 pg/L 1.8 pg/L 2.2 pg/L 1.6 pg/L 1.4 pg/L 1.8 pg/L 1.7 pg/L 1.9 pg/L 2.0 pg/L 4.2 pg/L	All samples in SDG G0D140534

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04122010-RIG3-RZD	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 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	0.64 pg/L 0.47 pg/L 1.9 pg/L 5.0 pg/L 4.6 pg/L 3.8 pg/L 2.2 pg/L 7.7 pg/L 4.8 pg/L 1.2 pg/L 0.59 pg/L 5.0 pg/L	0.64U pg/L 0.47U pg/L 1.9U pg/L 5.0U pg/L 4.6U pg/L 3.8U pg/L 2.2U pg/L 7.7U pg/L 4.8U pg/L 1.2U pg/L 0.59U pg/L 5.0U pg/L
EB-04122010-RIG2-RZB	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 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,7,8,9-HyCDF 1,2,3,7,8,9-HpCDF	0.31 pg/L 0.35 pg/L 0.60 pg/L 1.8 pg/L 4.0 pg/L 4.8 pg/L 4.3 pg/L 2.2 pg/L 7.3 pg/L 1.3 pg/L 0.85 pg/L 5.6 pg/L	0.31U pg/L 0.35U pg/L 0.60U pg/L 1.8U pg/L 4.0U pg/L 4.8U pg/L 4.3U pg/L 2.2U pg/L 7.3U pg/L 4.6U pg/L 1.3U pg/L 0.85U pg/L 5.6U pg/L

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-04122010-RIG1-RZB	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 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	0.57 pg/L 0.59 pg/L 0.82 pg/L 2.8 pg/L 10 pg/L 2.0 pg/L 1.2 pg/L 1.3 pg/L 1.7 pg/L 1.2 pg/L 1.1 pg/L 2.0 pg/L 3.6 pg/L 3.6 pg/L	0.57U pg/L 0.59U pg/L 0.82U pg/L 2.8U pg/L 10U pg/L 2.0U pg/L 1.2U pg/L 1.3U pg/L 1.7U pg/L 1.2U pg/L 1.1U pg/L 2.0U pg/L 3.6U pg/L 3.6U pg/L

Samples EB-04122010-RIG3-RZD, EB-04122010-RIG2-RZB, and EB-04122010-RIG1-RZB were identified as equipment blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG3-RZD	4/12/10	1,2,3,6,7,8-HxCDD 1,2,3,4,6,7,8-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-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-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	0.64 pg/L 0.47 pg/L 1.9 pg/L 5.0 pg/L 4.6 pg/L 2.2 pg/L 7.7 pg/L 4.8 pg/L 1.2 pg/L 0.59 pg/L 14 pg/L 5.0 pg/L 27 pg/L	No associated samples in this SDG
EB-04122010-RIG2-RZB	4/12/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 0CDF	0.31 pg/L 0.35 pg/L 0.960 pg/L 1.8 pg/L 4.0 pg/L 4.8 pg/L 4.3 pg/L 2.2 pg/L 7.3 pg/L 1.3 pg/L 1.3 pg/L 1.3 pg/L 0.85 pg/L 13 pg/L 5.6 pg/L 26 pg/L	No associated samples in this SDG

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-04122010-RIG1-RZB	4/12/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HyCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	0.57 pg/L 0.59 pg/L 0.82 pg/L 2.8 pg/L 10 pg/L 2.0 pg/L 1.2 pg/L 1.7 pg/L 1.2 pg/L 1.1 pg/L 2.0 pg/L 3.6 pg/L 3.6 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 G0D140534	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140534	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	А

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 G0D140534

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D140534	EB-04122010-RIG3-RZD EB-04122010-RIG2-RZB EB-04122010-RIG1-RZB	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D140534	EB-04122010-RIG3-RZD EB-04122010-RIG2-RZB EB-04122010-RIG1-RZB	All compounds reported as EMPC	JK (all detects)	А	Project Quantitation Limit (k)

### Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D140534

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D140534	EB-04122010-RIG3-RZD	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 1,2,3,7,8,9-HpCDF	0.64U pg/L 0.47U pg/L 1.9U pg/L 5.0U pg/L 4.6U pg/L 3.8U pg/L 2.2U pg/L 7.7U pg/L 4.8U pg/L 1.2U pg/L 0.59U pg/L 5.0U pg/L	А	bl
G0D140534	EB-04122010-RIG2-RZB	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 1,2,3,7,8,9-HpCDF	0.31U pg/L 0.35U pg/L 0.60U pg/L 1.8U pg/L 4.0U pg/L 4.8U pg/L 4.3U pg/L 2.2U pg/L 7.3U pg/L 4.6U pg/L 1.3U pg/L 0.85U pg/L 5.6U pg/L	A	ы

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D140534	EB-04122010-RIG1-RZB	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	0.57U pg/L 0.59U pg/L 0.82U pg/L 2.8U pg/L 10U pg/L 2.0U pg/L 1.2U pg/L 1.3U pg/L 1.7U pg/L 1.2U pg/L 1.1U pg/L 2.0U pg/L 3.6U pg/L 3.6U pg/L	4	bl

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D140534

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23188E21 Stage 2B SDG #: G0D140534 Laboratory: Test America

/ ,	
Date: <u>\$\$20</u> /	0
Page: <u>//</u> o <u>f /</u>	
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
1.	Technical holding times	A	Sampling dates: 4/12/10
11.	HRGC/HRMS Instrument performance check	A	/ /
111,	Initial calibration	<u> </u>	
IV.	Routine calibration/IX	1	
V.	Blanks	ÂW	
VI.	Matrix spike/Matrix spike duplicates	$\wedge$	client efertised
VII.	Laboratory control samples	A	LCS /
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	*	
X	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	9N	
XII.	System performance	N	
XIII.	Overall assessment of data	1	
XIV.	Field duplicates	N	
XV.	Field blanks	w	2B=/.2.3

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:

r====							
1	EB-04122010-RIG3-RZD	W	11	0110418MB	21	31	
2	EB-04122010-RIG2-RZB		12	• • • • • • • • • • • • • • • • • • • •	22	32	
3	EB-04122010-RIG1-RZB	1	13		23	33	
4			14		24	34	
5			15		25	35	
6			16		26	36	
7			17		27	37	
8			18		28	38	
9			19		29	39	
10			20		30	40	

Notes:		 	

## VALIDATION FINDINGS WORKSHEET

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

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1.2.3.4.7.8.9.Hp.CDE	
00000				o. Total npcoo
O. 1,5,5,7,9-PBCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	// Total TODE
1000				v. 10tal 10th
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	14/ Total D. O. C.
1				VY. TOTAL FBCDF
U. 1,2,3,6,7,8-HxCDD	1. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HXCDF	S Total Bacon	# 40 C T T T T T
1000			2008 1 800 1 10	A. Iotal HXCUF
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	
				TOO 20 18 10

Notes:

## VALIDATION FINDINGS WORKSHEET

Routine Calibration

Page:

2nd Reviewer: Reviewer:

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

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

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

. *	Date	Standard ID	Compound	Findl (Limit:	Finding %D 20/	Finding Ion Abundance Ratio	Associated Samples	80	Qualifications (a)
	4/26/10	26APro4D5.	0 /	20.4	7		來		1744 P (C)
	/ /	,							
			-						
			,						
	-								
		PCDDs	Selected ions (m/z)	Ion Abundance Ratio	atio	PCDFs	Selected ions (m/z)	(z/m) suo	ion Abundance Ratio
	Tetra-		M/M+2	0.65-0.89		Tetra-	M/N	M/M+2	0.65-0.89
	Penta-		M+2/M+4	1.32-1.78		Penta-	M+2/M+4	/M+4	1.32-1.78
	Неха-		M+2/M+4	1.05-1.43		Неха-	M+2/M+4	M+4	1.05-1.43
	Hexa-13C-Hx(	Hexa-¹3C-HxCDF (IS) only	M/M+2	0.43-0,59		Hexa-13C-HxCDF (IS) only	M/M+2	1+2	0.43-0.59
	Hepta- <sup>13</sup> C-Hp	Hepta-13C-HpCDF (IS) only	, M/M+2	0.37-0.51		Hepta-13C-HpCDF (IS) only	y M/M+2	1+2	0.37-0.51
	Hepta-		M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	M+4	0.88-1.20
	Octa-		M+2/M+4	0.76-1.02		Octa-	M+2/M+4	M+4	0.76-1.02

LDC #: 23188E21

SDG #: See Cover

VALIDATION FINDINGS WORKSHEET

Blanks

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

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? If yes, please see qualification below. Y/N N/A

Blank analysis date: 4/26/10 Blank extraction date: 4/20/10

Associated samples:

AII ( ) ( ) Sample Identification 0.59/U 0.82/U 2.0/∩ 0.95/U 1.2/U 3.6/∪ 2.8/U 2.0/U 1.3/U 1.7/U 1.1/0 0.57/U 1.2/U 10/0 0.31/U 0.35/U 0.60/U 0.85/U 4.8/U 7.3/U 5.6/∪ 1.8/U 4.0/N 4.3/U 2.2/U 4.6/U 1.3/U 0.47/U 0.59/U 0.64/∪ 1.9/U 5.0/U 4.6/U 3.8/∪ 2.2/∪ 7.7/ 4.8/U 1.2/∪ 5.0/U 13.5 6.5 8.5 9.5 6.5 9 섫 55 7 O 7 တ 6 O 8 0110418MB Blank ID 1.3 <u>~</u> ∞ <u>6</u> 2.2 6.1 2.0 <del>2</del>. 2.7 1.6 <u>6</u>. 4.2 Ξ Compound Conc. units: pg/L G Ø O Ω Σ z 0 ш ш

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".

SDG #:See Cover LDC #: 23188E21

## VALIDATION FINDINGS WORKSHEET

**Field Blanks** 

Page: of Reviewer:\_ 2nd Reviewer:

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

Were field blanks identified in this SDG?

Associated sample units: Blank units: pg/L / Sampling date: 4/12/10

Compound Blank ID	Blank ID			Sample Identification	ıtion		
	ER-04122010-RIG3-RZD	5X					
D	0.64	0.0032					
Ш	0.47	0.00235					
ш.	1.9	0.0095					
<b>5</b>	5.0	0.025					
Ι	4.6	0.023					
	3.8	0.019					
ر	2.2	0.011					
¥	7.7	0.0385					
	4.8	0.024					
Z	1.2	0.006					
Z	0.59	0.00295					
0	14	0.07					
۵	5.0	0.025					
Ø	27	0.135					
			1				
CROL							

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

SDG #:See Cover LDC #: 23188E21

## **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Reviewer: 2nd Reviewer:

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

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

b/bd Associated sample units: Blank units: pg/L

Sampling date: 4/12/10

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

Associated Samples:

Sample Identification 0.00425 0.00175 0.00155 0.0048 0.0215 0.0065 0.0365 0.009 0.065 0.028 0.023 0.024 0.011 0.02 0.13 শ্ব EB-04122010-RIG2-RZB Blank ID 0.35 0.960 0.85 0.31 4.8 4.3 4.6 5.6 4.0 2.2 13 56 Compound ტ

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

ב-... המקרם הואועהות הדולהדגייי-דוג-הואו

SDG #: See Cover LDC #: 23188E21

## VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: 3 of 3 Reviewer: 2nd Reviewer:

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

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

Associated sample units:\_ Blank units: pg/L Sampling date: 4/12/10

b/bd

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

Associated Samples:

Compound	Blank ID		Sa	Sample Identification	tion		
	FB-04122010-RIG1-RZB	5X					
O	0.57	0.00285					
D	0.59	0.00295					
ш	0.82	0.0041		7			
L	2.8	0.014			ļ		
9	10	0.05				:	
H	2.0	0.01		-			
-	1.2	900.0					
7	1.3	0.0065					
Х	1.7	0.0085					
7	1.2	0.006					
M	1.1	0.0055					
0	2.0	0.01				. )	
Ь	0.95	0.00475					
۵	3.6	0.018					
CRQL							

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

SDG #: 28/8822/ SDG #: 28. CONEN

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: \_\_of\_

Reviewer: 4

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".

A N N

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).

Qualifications	(a)*		C	NONS						
Associated Samples										
Finding	ZMPC MSWAS	(& Has )		No castismation CM	8.37.8-TeaF					
Sample ID	m			M						
Date										
*										

Comments: See sample calculation verification worksheet for recalculations

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 12, 2010

LDC Report Date:

May 24, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D140543

### Sample Identification

SSAN6-02-3BPC SSAN7-02-1BPC\_FD SSAJ3-02-1BPC\_FD SA156-3BPC\_FD\*\* SSAN6-02-3BPCMS

SSAN6-02-3BPCMSD

<sup>\*\*</sup>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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	SSAN6-02-3BPC
0109260MB	4/19/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.59 pg/g 0.28 pg/g 0.14 pg/g 0.24 pg/g 0.14 pg/g 0.086 pg/g 0.30 pg/g 0.13 pg/g 0.63 pg/g	SSAN7-02-1BPC_FD SSAJ3-02-1BPC_FD SA156-3BPC_FD**

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

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAJ3-02-1BPC_FD	1,2,3,4,6,7,8-HpCDD	0.41 pg/g	0.41U pg/g
	OCDD	1.4 pg/g	1.4U pg/g
	2,3,7,8-TCDF	0.60 pg/g	0.60U pg/g
	1,2,3,7,8-PeCDF	0.69 pg/g	0.69U pg/g
	1,2,3,4,7,8-HxCDF	1.2 pg/g	1.2U pg/g
	1,2,3,7,8,9-HxCDF	0.18 pg/g	0.18U pg/g
SA156-3BPC_FD**	2,3,7,8-TCDF	0.16 pg/g	0.16U pg/g
	1,2,3,4,6,7,8-HpCDF	1.2 pg/g	1.2U pg/g

Samples EB-04122010-RIG1-RZB, EB-04122010-RIG2-RZB, and EB-04122010-RIG3-RZB (all from SDG G0D140534) 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-04122010-RIG1-RZB	4/12/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.57 pg/L 0.59 pg/L 0.82 pg/L 2.8 pg/L 10 pg/L 2.0 pg/L 1.2 pg/L 1.3 pg/L 1.7 pg/L 1.2 pg/L 1.1 pg/L 2.0 pg/L 3.6 pg/L 3.6 pg/L	SA156-3BPC_FD**
EB-04122010-RIG2-RZB	4/12/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.31 pg/L 0.35 pg/L 0.60 pg/L 1.8 pg/L 4.0 pg/L 4.8 pg/L 4.3 pg/L 2.2 pg/L 7.3 pg/L 4.6 pg/L 1.3 pg/L 0.85 pg/L 13 pg/L 5.6 pg/L 26 pg/L	SA156-3BPC_FD**
EB-04122010-RIG3-RZB	4/12/10	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 1,2,3,4,6,7,8-HpCDF	0.64 pg/L 0.47 pg/L 1.9 pg/L 5.0 pg/L 4.6 pg/L 3.8 pg/L 2.2 pg/L 7.7 pg/L 4.8 pg/L 1.2 pg/L 0.59 pg/L 14 pg/L 5.0 pg/L 27 pg/L	SSAJ3-02-1BPC_FD

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

Samples FB04062010-RZB (from SDG G0D120488), FB-04072010-RZD (from SDG G0D090441), and FB-04072010-RZC (from SDG G0D130519) were identified as field blanks. No polychlorinated dioxin/dibenzofuran contaminants were found in these blanks with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	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	SSAJ3-02-1BPC_FD
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	0.68 pg/L 2.5 pg/L 6.2 pg/L 2.7 pg/L 1.4 pg/L 0.82 pg/L 0.94 pg/L 1.8 pg/L 1.2 pg/L 4.4 pg/L	SA156-3BPC_FD**
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 1,2,3,4,6,7,8-HpCDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.0 pg/L 2.1 pg/L 6.7 pg/L	SSAN6-02-3BPC SSAN7-02-1BPC_FD

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 with the following exceptions:

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-02-3BPC	<sup>13</sup> C-OCDD	34 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA156-3BPC_FD**	<sup>13</sup> C-OCDD	32 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

### X. Target Compound Identifications

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

### XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN6-02-3BPC	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)	Р
SSAN7-02-1BPC_FD	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140543	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D140543	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	Α

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 SSAN7-02-1BPC\_FD and SSAN7-02-1BPC (from SDG (G0D140422), samples SSAJ3-02-1BPC\_FD and SSAJ3-02-1BPC (From SDG G0D140422), and samples SA156-3BPC\_FD\*\* and SA156-3BPC (from SDG G0D140435) were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentra	ation (pg/g)	DDD	222		
Compound	SSAJ3-02-1BPC	SSAJ3-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.22	1.1U	-	0.88 (≤1.1)	-	-
1,2,3,7,8-PeCDD	0.90	5.3U	-	4.4 (≤5.3)	-	-
1,2,3,4,7,8-HxCDD	0,99	5.3U	-	4.31 (≤5.3)	-	-
1,2,3,6,7,8-HxCDD	1.2	0.13	-	1.07 (≤5.3)	-	-
1,2,3,7,8,9-HxCDD	1.1	0.12	-	0.98 (≤5.3)	-	-
1,2,3,4,6,7,8-HpCDD	1.6	0.41	-	1.19 (≤5.3)	-	-
OCDD	5.9	1.4	-	4.5 (≤11)	-	-
2,3,7,8-TCDF	0.90	0.60	-	0.3 (≤1.1)	-	-

Concentration (pg/g)		ation (pg/g)	RPD	Difference		
Compound	SSAJ3-02-1BPC	SSAJ3-02-1BPC_FD	(Limits)	(Limits)	Flags	A or P
1,2,3,7,8-PeCDF	2.1	0.69	-	1.41 (≤5.3)	-	-
2,3,4,7,8-PeCDF	1.5	0.39	-	1.11 (≤5.3)	-	-
1,2,3,4,7,8-HxCDF	1.7	1.2	-	0.5 (≤5.3)	-	-
1,2,3,6,7,8-HxCDF	2.2	0.76	-	1.44 (≤5.3)	-	
2,3,4,6,7,8-HxCDF	1.5	0.16	-	1.34 (≤5.3)	-	-
1,2,3,7,8,9-HxCDF	1.4	0.18	<u>.</u>	1.22 (≤5.3)	-	-
1,2,3,4,6,7,8-HpCDF	3.2	2.7	-	0.5 (≤5.3)	-	-
1,2,3,4,7,8,9-HpCDF	2.1	0.99	-	1.11 (≤5.3)	-	-
OCDF	9.1	7.8	-	1.3 (≤11)	-	-

	Concentra	ation (pg/g)	RPD	DDD D:4		
Compound	SSAN7-02-1BPC	SSAN7-02-1BPC_FD	(Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	2.4	2.4	-	0 (≤1.1)	-	-
1,2,3,7,8-PeCDD	11	10	-	1 (≤5.3)	-	-
1,2,3,4,7,8-HxCDD	6.6	5.3	-	1.3 (≤5.3)	-	-
1,2,3,6,7,8-HxCDD	15	12	-	3 (≤5.3)	-	-
1,2,3,7,8,9-HxCDD	11	8.6	-	2.4 (≤5.3)	-	-
1,2,3,4,6,7,8-HpCDD	39	35	11 (≤50)	-	-	-
OCDD	40	35	-	5 (≤11)	-	-
2,3,7,8-TCDF	130	170	27 (≤50)	-	-	-
1,2,3,7,8-PeCDF	160	210	27 (≤50)	-	-	-

	Concentra	ation (pg/g)	222	B.44		
Compound	SSAN7-02-1BPC	SSAN7-02-1BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,4,7,8-PeCDF	77	82	6 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	230	250	8 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	200	160	22 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	44	35	23 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	42	21	-	21 (≤5.3)	J (all detects)	А
1,2,3,4,6,7,8-HpCDF	500	440	13 (≤50)	-	•	-
1,2,3,4,7,8,9-HpCDF	270	220	20 (≤50)	-	-	-
OCDF	1300	1200	8 (≤50)	-	-	•

	Concentra	ition (pg/g)	220	D.W.		
Compound	SA156-3BPC	SA156-3BPC_FD**	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.15	0.54U		0.39 (≤0.54)	<del>-</del>	-
1,2,3,7,8-PeCDD	0.56	2.7U	•	2.14 (≤2.7)	-	-
1,2,3,4,7,8-HxCDD	0.84	2.7U	-	1.86 (≤2.7)	-	-
1,2,3,6,7,8-HxCDD	0.68	2.7U	-	2.02 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	0.81	2.7U	-	1.89 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	1.4	2.7U	-	1.3 (≤2.7)	-	-
OCDD	5.6	5.4U		0.2 (≤5.4)	-	-
2,3,7,8-TCDF	0.51	0.16	-	0.35 (≤0.54)	-	-
1,2,3,7,8-PeCDF	1.7	2.7U	-	1 (≤2.7)	-	-
1,2,3,4,7,8-HxCDF	4.1	2.7U	-	1.4 (≤2.7)	-	<u>-</u>

	Concentra	centration (pg/g)				
Compound	SA156-3BPC	SA156-3BPC_FD**	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,6,7,8-HxCDF	3.3	2.7U	-	0.6 (≤2.7)	-	-
2,3,4,6,7,8-HxCDF	1.4	2.7U	-	1.3 (≤2.7)	-	-
1,2,3,7,8,9-HxCDF	1.7	2.7U	-	1 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDF	10	1.2	-	8.8 (≤2.7)	J (all detects)	Α
1,2,3,4,7,8,9-HpCDF	5.1	2.7U	_	2.4 (≤2.7)	-	-
OCDF	25	5.4	-	19.6 (≤5.4)	J (all detects)	А

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D140543

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D140543	SSAN6-02-3BPC SA156-3BPC_FD**	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0D140543	SSAN6-02-3BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D140543	SSAN7-02-1BPC_FD	2,3,7,8-TCDF	J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D140543	SSAN6-02-3BPC SSAN7-02-1BPC_FD SSAJ3-02-1BPC_FD SA156-3BPC_FD**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D140543	SSAN6-02-3BPC SSAN7-02-1BPC_FD SSAJ3-02-1BPC_FD SA156-3BPC_FD**	All compounds reported as EMPC	JK (all detects)	А	Project Quantitation Limit (k)
G0D140543	SSAN7-02-1BPC_FD	1,2,3,7,8,9-HxCDF	J (all detects)	A	Field duplicates (Difference) (fd)
G0D140543	SA156-3BPC_FD**	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Α	Field duplicates (Difference) (fd)

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0D140543

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D140543	SSAJ3-02-1BPC_FD	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 1,2,3,4,7,8-HxCDF	0.41U pg/g 1.4U pg/g 0.60U pg/g 0.69U pg/g 1.2U pg/g 0.18U pg/g	A	bl
G0D140543	SA156-3BPC_FD**	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF	0.16U pg/g 1.2U pg/g	А	bl

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0D140543

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D140543

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23188F21 Stage 2B/4 SDG #: G0D140543

Date: 51/10
Page: //of/
Reviewer:
2nd Reviewer:

Laboratory: Test America

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
l.	Technical holding times	$\phi$	Sampling dates: 4/12/10
II.	HRGC/HRMS Instrument performance check	A	/ /
III.	Initial calibration	Ã	
IV.	Routine calibration/I	A.	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	W	
VII.	Laboratory control samples	A	109
VIII.	Regional quality assurance and quality control	N,	
IX.	Internal standards	w	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	$ \mathcal{M} $	Not reviewed for Stage 2B validation.
XII.	System performance	\$	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	1	
XIV.	Field duplicates	an	D=2+SSAN7-02-1BPC, 3+SSAJ3-02-1BPC(G0D140422), D=4+SA156-3BPC(G0D140435)
XV.	Field blanks	W	FB04062010-RZB(G0D120488)FB-04072010-RZD(G0D090441) FB-040710-RZC(G0D130519), EB-04122010-RIG3-RZD (G0D140534) EB-04122010-RIG2-RZB, EB-04122010-RIG1-RZB (G0D140534)

A = Acceptable Note:

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

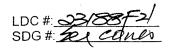
D = Duplicate TB = Trip blank

EB = Equipment blank

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

					<u> </u>		
10	SSAN6-02-3BPC 5	11	0/104#5MB	21		31	
2 c	SSAN7-02-1BPC_FD	12	0/09260MB	22		32	
3 Ŋ	SSAJ3-02-1BPC_FD	13	<u> </u>	23		33	
4 6	SA156-3BPC_FD**	14		24		34	
5	SSAN6-02-3BPCMS	15		25		35	
6	SSAN6-02-3BPCMSD	16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:			
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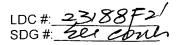


## **VALIDATION FINDINGS CHECKLIST**

	Page:	
	Reviewer:	9
2nd	Reviewer:	

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times			,	
All technical holding times were met.				-
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check		······		
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% ?		<del>-</del>		
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?				
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?		(		
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?				
IV. Continuing calibration	VII.			
Was a routine calibration performed at the beginning and end of each 12 hour period?				
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?		,		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				2000년 대한 마음 마음 발표 18 발표 19 발표 19 등 19
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?				
VI. Matrix spike/Matrix spike duplicates			Trive ve Street	
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?				
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



## **VALIDATION FINDINGS CHECKLIST**

Page: of a Reviewer: 2nd Reviewer:

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# 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. ocpp	L. 1,2,3,6,7,8-HxCDF	a. 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	1. 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 #: 23/88 SDG #: Xe

## VALIDATION FINDINGS WORKSHEET Blanks

Reviewer: 2nd Reviewer:

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".

Were all samples associated with a method blank? V N/A Y/N N/A

Was a method blank analyzed for each matrix?

Associated Samples: Was the blank contaminated? If yes, please see qualification below. n date: 420/0 Blank analysis date: 426/10 /@Blank analysis date: 子色 Blank extraction date: 489 YN Z

ication									
Sample Identifi									
	a								
	<b>C</b>								
Blank ID	DUSSHI	150	180	330					
pt									
Compour		7	7	0					
	Blank ID	Blank ID D D D D D D D D D D D D D D D D D D	Blank ID   D   D   D   D   D   D   D   D   D	Blank ID 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	Blank ID	Blank ID  0 3/ 0 3/ 0 3/ 0 3/	Blank ID	Blank ID	Blank ID  0 3/ 0 3/ 0 3/ 0 38

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

Compound	Blank ID	Sample Identification

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

LDC #: 23188F21

SDG #: See Cover

## VALIDATION FINDINGS WORKSHEET Blanks

Page: 1 of 1 Reviewer: 2nd Reviewer:

METHOD: HRGC/HRMS Dioxins (EPA Method 8290)

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? If yes, please see qualification below.

Associated samples:

Blank analysis date: 4/25/10 Blank extraction date: 4/19/10

Sonc. units: pg/L Cx

Sample Identification 0.16/U 1.2/∪ 0.41/U 0.60/U 0.69/∪ 0.18/U 1.4/U 1.2/U 2.95 0.43 0.65 3.15 1.5 0.7 1.2 0.7 <u>4</u>. 0.7 ধ 0109260MB Blank ID 0.086 0.14 0.590.28 0.14 0.14 0.24 0.30 0.13 0.63 Compound G

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".

F: ();

LDC #: 23188EF21 SDG #: See Cover

# VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: \_\_lof\_ Reviewer:\_ 2nd Reviewer:\_

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

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

Associated sample units: Blank units: pg/L

b/bd

Sampling date: 4/12/10

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

4

Sample Identification 0.00295 0.00285 0.00475 0.0065 0.0055 0.0041 0.0085 0.014 0.006 900.0 0.018 0.05 0.01 0.01 ž EB-04122010-RIG1-RZB Blank ID 0.59 0.57 0.82 0.95 2.8 2.0 2.0 3.6 1.2 1.3 10 1.7 1.2 7: Compound CRQL G Ω

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

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SDG #:See Cover LDC #: 23188F21

# VALIDATION FINDINGS WORKSHEET

Field Blanks

Reviewer: 2nd Reviewer:

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

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

Associated sample units: Blank units: pg/L / Sampling date: 4/12/10

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

Compound	Blank ID			S	Sample Identification	tion		
	FB-04122010-RIG2-RZB	5X						
0	0.31	0.00155						
D	0.35	0.00175						
Ш	0.60	0.003						
Щ	1.8	0.009						
9	4.0	0.02						
I	4.8	0.024						
	4.3	0.0215						
J	2.2	0.011						
X	7.3	0.0365						
Γ	4.6	0.023						
Σ	1.3	0.0065						
Z	0.85	0.00425						
0	13	0.065						
۵.	5.6	0.028						
۵	26	0.13						
			-					
CROL								

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

SDG #:See Cover LDC #: 23188F21

# VALIDATION FINDINGS WORKSHEET

Field Blanks

Page: 2nd Reviewer: Reviewer:

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

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

Blank units: pg/L Associated sample units

b/bd Associated sample units:\_

Sampling date: 4/12/10

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

က

Sample Identification 0.00235 0.0032 0.0095 0.0385 0.00295 0.019 0.006 0.025 0.023 0.025 0.135 0.011 0.024 0.07 X EB-04122010-RIG3-RZD Blank ID 0.64 0.47 1.9 0.59 5.0 4.6 3.8 2.2 4.8 1.2 5.0 7.7 4 27 Compound ပ Ω I Σ ш 0

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

THE OTTO CITY CITY CITY CALL CALL

LDC#: 23/88 SDG #:See Cover

## VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

Y N N/A Were field blanks identified in this SDG? Blank units: <u>pg/L</u> Associated sample units

b/bd Associated sample units:

Sampling date: 4/8/10

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

1-2(>5x Sample Identification Associated Samples: 0.00385 0.00285 0.00335 0.0037 0.0041 0.185 0.0048 0.0055 0.0048 0.0105 0.0075 0.0335 0.021 0.005 0.005 X EB-04072010-RZC Blank ID 0.77 0.74 0.82 0.57 96.0 0.67 96.0 4.2 1.0 7 0. 1.5 37 2.1 6.7 Compound G

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

SDG #: See Cover LDC #: 23188F21

# **VALIDATION FINDINGS WORKSHEET**

Field Blanks

Reviewer: 2nd Reviewer:

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

b/bd Associated sample units:\_ (Y N/A Were field blanks identified in this SDG?

Blank units: pg/L Sampling date: 4/7/10

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

က

Sample Identification 0.00445 0.0075 0.0415 0.0075 0.0065 0.0205 0.008 0.008 0.011 0.007 0.007 FR-04072010-RZD Blank ID 0.89 1.5 2.2 8.3 4. 1.6 1.6 <del>1.</del>3 4. 4.1 Compound CROL တ ≥ z

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

SDG #:See Cover LDC #: 23188F21

# VALIDATION FINDINGS WORKSHEET

Field Blanks

\_ | | | | 2nd Reviewer:\_ Page:\_\_ Reviewer:

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

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

b/bd Associated sample units: Blank units: pg/L Sampling date: 4/6/10

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

Sample Identification Associated Samples: 0.0034 0.0125 0.0135 0.0041 0.0047 0.009 0.006 0.031 0.007 0.022 젉 FB04062010-RZB Blank ID 0.68 0.82 0.94 2.5 6.2 2.7 4. 1.2 4.4 Compound G I 0 Z

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

## Watrix Spike/Matrix Spike Duplicates

Page: /of/ Reviewer: Q

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

SDG #: 50ck

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

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

associated MS/MSD. Soil / Water.

Was a MS/MSD analyzed every 20 samples of each matrix?

Y(N) N/A Were the MS/MSD percent recoveries (%R) and the relative perce

Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

# Data					Me	COM			
( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( ) ( )	*	Date	MS/MSD ID	Compound	%R (Limits)	%R (Limits)	RPD (Limits)	Associated Samples	Qualifications
					M F	1 )	1 ) 5/2		No and
			/				( )		J (
					( )	)	)		
					( )	)			
					( )	( )	( )		
					( )	( )	( )		
					( )		( )		
	┪				( )	)	(		
					( )	( )	( )		
	1				( )				
	7				( )	( )			
	1				( )	( )	(		
					( )	( )	( )		
					( )	( )			
	1				( )	( )	(		
					( )	( )	( )		
	+				( )	( )	( )		
	1				)	( )	( )		
	$\dashv$				( )	( )	( )		
					( )	( )	( )		
	1				( )	( )	( )		
( ) ( )	7				( )	( )	( )		
	$\dashv$				( )	( )	( )		

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**	#200
LDC	SDG

## **VALIDATION FINDINGS WORKSHEET** Internal Standards

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

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 were within the 40-135% criteria? Was the S/N ratio all internal standard peaks > 10?

Y N N/A

	-		-			
#	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135%)	Qualifica
		/	7	•	34 (40-135	) JANG (A. B)
Г					•	
		7	4		30	
					)	
					)	
					)	)
					)	
					)	
					)	(
					)	
					)	
					)	
					)	
					)	
					)	
					)	
					)	
					)	
					)	
					)	
		Internal Standards	Check Standard Used		Internal Standards	Check Standard Used
Ą	<sup>13</sup> C-2,3,7,8-TCDF	ODF		σ	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	
B.	13C-2,3,7,8-TCDD	CDD		Ξ	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	
ن ن	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF			13C-OCDD	
D.	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		Ϋ́.	<sup>13</sup> C-1,2,3,4-TCDD	
щ	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	8-HxCDF		نـ	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
ш	F 3C-123678-HxCDD	8-HxCDD				



## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

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? Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

Qualifications	( = ) \$ ( = )			ーリンメフ						
Associated Samples										
god > call Laws	80	74		# EMPC 1881/15	(8 +69)	/ /				
Sample ID		B		4//						
Date							0			
*										

Comments: See sample calculation verification worksheet for recalculations

LDC#:<u>23188F21</u> SDG#: See Cover

## VALIDATION FINDINGS WORKSHEET Field Duplicates

Page:\_lof 3
Reviewer:\_\_\_\_\_
2nd Reviewer:\_\_\_\_\_

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

YN NA YN NA Were field duplicate pairs identified in this SDG?

Were target analytes detected in the field duplicate pairs?

	Concentra	ition (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	SSAJ3-02-1BPC	3	RPD	Difference	Limits	(Parent Only)
А	0.22	1.1U		0.88	( <u>&lt;</u> 1.1)	
В	0.90	5.3U		4.4	( <u>&lt;</u> 5.3)	
С	0.99	5.3U		4.31	( <u>&lt;</u> 5.3)	
D	1.2	0.13		1.07	( <u>&lt;</u> 5.3)	
E	1.1	0.12		0.98	( <u>&lt;</u> 5.3)	
F	1.6	0.41		1.19	( <u>≤</u> 5.3)	
G	5.9	1.4		4.5	( <u>≤</u> 11)	
Н	0.90	0.60		0.3	( <u>&lt;</u> 1.1)	
1	2.1	0.69		1.41	( ≤5.3)	
J	1.5	0.39		1.11	( <u>&lt;</u> 5.3)	
к	1.7	1.2		0.5	( ≤5.3)	,
L	2.2	0.76		1.44	( <u>≤</u> 5.3)	
М	1.5	0.16		1.34	( <u>&lt;</u> 5.3)	
N	1.4	0.18		1.22	( <u>≤</u> 5.3)	
0	3.2	2.7		0.5	( <u>≤</u> 5.3)	
Р .	2.1	0.99		1.11	( <u>≤</u> 5.3)	
Q	9.1	7.8		1.3	(<11)	

	Concentration	n (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	SSAN7-02-1BPC	2	RPD	Difference	Limits	(Parent Only)
Α	2.4	2.4		0	( <u>&lt;</u> 1.1)	
В	11	10		1	( ≤5.3)	
С	6.6	5.3		1.3	( ≤5.3)	
D	15	12		3	( <u>&lt;</u> 5.3)	
E	11	8.6		2.4	( ≤5.3)	
F	39	35	11			
G	40	35		5	( <u>&lt;</u> 11)	

LDC#: 23188F21 SDG#: See Cover

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

	Page:	<u> </u>	<u> </u>
	Reviewer:	4	
nd	Reviewer:		

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

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

	Concentra	ition (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	SSAN7-02-1BPC	2	RPD	Difference	Limits	(Parent Only)
Н	130	170	27			
-	160	210	27			
J	77	82	6			
К	230	250	8			
L	200	160	22			
М	44	35	23			
N	42	21		21	( ≤5.3)	Idet & A
0	500	440	13			/
Р	270	220	20			
Q	1300	1200	8			

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	SA156-3BPC	4	RPD	Difference	Limits	(Parent Only)
А	0.15	0.54U		0.39	( ≤0.54)	
В	0.56	2.7U		2.14	( <u>&lt;</u> 2.7)	
С	0.84	2.7U		1.86	( <u>&lt;</u> 2.7)	
D	0.68	2.7U		2.02	( ≤2.7)	
E	0.81	2.7U		1.89	( <u>≤</u> 2.7)	
F	1.4	2.7U		1.3	( <u>&lt;</u> 2.7)	
G	5.6	5.4U		0.2	( <u>&lt;</u> 5.4)	
Н	0.51	0.16		0.35	( <u>&lt;</u> 0.54)	
ı	1.7	2.7U		1	( <u>&lt;</u> 2.7)	
κ	4.1	2.7U		1.4	( <u>&lt;</u> 2.7)	
L	3.3	2.7U		0.6	( ≤2.7)	,
М	1.4	2.7U		1.3	( ≤2.7)	
N	1.7	2.7U		1	( <u>&lt;</u> 2.7)	
0	10	1.2		8.8	( ≤2.7)	Stote A

LDC#:_	23188F21
SDG#:	See Cover

## **VALIDATION FINDINGS WORKSHEET** Field Duplicates

Page:_=	≥of_ <u>&gt;</u> >
Reviewer:	9-
2nd Reviewer:	<u> </u>

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

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

	Concentrat	ion (pg/g)	(≤50)	(pg/g)	(pg/g)	Qualifications
Compound	SA156-3BPC	4	RPD	Difference	Limits	(Parent Only)
Р	5.1	2.7U		2.4	( <u>&lt;</u> 2.7)	
Q	25	5.4		19.6	( ≤5.4)	dats/A

V:\FIELD DUPLICATES\23188F21.wpd

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORNSHEE

107 rage: Reviewer: 2nd Reviewer:

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

 $\begin{array}{l} A_x = \text{Area of compound,} \\ C_x = \text{Concentration of compound,} \\ S = \text{Standard deviation of the RRFs,} \\ \end{array}$ 

 $A_{\bf k}=A {\bf rea}$  of associated internal standard  $C_{\bf k}=Concentration$  of internal standard  $X=M {\bf ean}$  of the RRFs

L_				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF ( 🍂 ∋std)	RRF ( &SStd)	%RSD	%RSD
-	10/2	7/1/-	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	2.889.51886.0	218891	101812	1.0/872	4.65.936	4.659
	(305)	24/12	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	20/501	DO120.1	1.11325-	255/11	7.43940	7.440
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.08449	64480.1	1.09101	101601	760295	5.63/
			1,2,3,4,6,7,8-HpCDD (¹3C-1,2,4,6,7,8,-HpCDD)	1.02088	880801	1.08662	1.08562	4.4945	4.495
			OCDF (4c-OCDD)	1.4.2582	1.4258>	1.57900	1.57900	8.93881	6868
2	ate	(1) L	(12) 2,3,7,8-TCDF (18C-2,3,7,8-TCDF)	346.0	0.45	0.0X	0.9X	4.44	433
	(405)	11/1/	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1001	1501	40-1	40:	X03	10.4
			1,2,3,6,7,8-HxCDD (¹3C-1,2,3,6,7,8-HxCDD)	711.1	1:14	0:	0.	573	(SK.72)
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	1072	1 - 1	1 . 1	3.60	3.75
			OCDF (13C-OCDD)	145	1445	.5	1.51	585	5.89
တ	1041	7.7.	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.088	1.088	3.	1.10	601	1. x
		01/10	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 (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF ("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

Page: of And 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 \* (ave, RRF - RRF)/ave, RRF RRF =  $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$ 

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

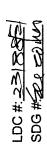
 $A_{\kappa}$  = Area of compound,  $A_{\kappa}$  = Area of associated internal standard  $C_{\kappa}$  = Concentration of compound,  $C_{\kappa}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
1		Calibration		Average RRF	RRF	RRF		
*	Standard ID	Date	Compound (Reference Internal Standard)	(initial)	(၁၁)	(၁၁)	%D	Q%
	28401043bs		2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.98315	THICK'S	1.92144	[E9	E. I
		4/00/10	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	201201	1800/1	1.10087	4.7	47
		`	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.0 Salt 9	1.13455	1.134SK	4.8	4.8
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	& 90E01	028001	1.00820	4.0	3.
			OCDF ("c-OCDD)	1.42582	1.38551	1.3855/	2.00	8.0
2	Saparathic	/ ' / '	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.945.	060	0.90	4.3	43
	,	a1/20/4	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.001	960	0.96	6.3	6.2
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.114	1.08	1.00	7 8	1.4
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	1.03	60.1	0 N	3.9
		,	OCDF (*3C-OCDD)	1.445	1.36	1.36	59	58
က	63HY185D	5/2/10	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	880.1	1.00	1:00	188	8
		11/	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)					

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.

1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)

OCDF ("C-OCDD)



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

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

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

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

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

MS/MSD samples: \_

RPD = I MSR - MSDR I \* 2/(MSR + MSDR)

	S	ike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	Reported	Recalculated
Compound	¥¥.	Added (S)	Concentration	Concentration ( ( ( )	centration	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	RPD
	SW	MSD		MS	MSD	Renorted	Recalc	Reported	Recalc		•
2,3,7,8-TCDD	7	0.0	1.8	1.60	3/8	817	[3	921	126		d .
1,2,3,7,8-PeCDD	106	1001	1	139	(53	911	1.0	130	130	9	Ø,
1,2,3,4,7,8-HxCDD			9.6	1.6	4	101	001	126	951	100	2
1,2,3,4,7,8,9-HpCDF	<b> </b>		490	715	733	800	7 7	655	122	90	2.5
OCDF	7 7	210	3900	4340	500	0/5	20 8	155	248	5)	15,
				-							

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



## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy 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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0/09/36

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

	S	ike	Spiked	ample	SOI	S	I CSD	d.	I/SO I	CS/I CSD
Compound	**************************************	Added (1)	Concentration (F3)	ration	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	٥٥
	1 08	1 CSD	SUI	usoı	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	io=	N.A	19.8	₩ ₩	99	66				
1,2,3,7,8-PeCDD	201		C01		102	102				
1,2,3,4,7,8-HxCDD	_		108		201	18				
1,2,3,4,7,8,9-HpCDF	<i>^</i>		701		104	701				
OCDF	200	<b>~</b>	202	<b>~</b>	101	101				

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

-	HPCDF HPCDF HPCDD HPCDD HPCDD (S) NCDPE PFK	HPCDF (\$) HPCDD (\$) HPCDD (\$) HPCDD (\$) HPCDD (\$) OCDF OCDF OCDD (\$) OCDD (\$) OCDP OCDD (\$) OCDP OCDD (\$)
C, H*C , 7C O	13C <sub>12</sub> H <sup>3C</sup> G), O <sup>c</sup> 13C <sub>12</sub> H <sup>3C</sup> G), O <sup>c</sup> 13C <sub>12</sub> H <sup>3C</sup> G1, 37ClO <sub>2</sub> 13C <sub>12</sub> H <sup>3C</sup> G1, 37ClO <sub>2</sub> 13C <sub>12</sub> H <sup>3C</sup> G1, 37ClO <sub>2</sub> 13C <sub>12</sub> H <sup>3C</sup> G1, 37Cl <sub>2</sub> O C <sub>12</sub> H <sup>3C</sup> G1, 37Cl <sub>2</sub> O C <sub>3</sub> F1,	13 <sup>C</sup> <sub>12</sub> H <sup>3</sup> <sup>C</sup> <sub>13</sub> O <sup>1</sup> 13 <sup>C</sup> <sub>12</sub> H <sup>3</sup> <sup>2</sup> Cl <sup>3</sup> O <sup>1</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O 13 <sup>C</sup> <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O 13 <sup>C</sup> <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> <sup>3</sup> Cl <sup>2</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> O C <sub>12</sub> H <sup>3</sup> <sup>3</sup> Cl <sup>3</sup> O C <sub>12</sub> H <sup>3</sup> Cl <sup>3</sup> O C <sub>12</sub> H <sup>3</sup> Cl <sup>2</sup> O C <sub>13</sub> H <sup>3</sup> Cl <sup>2</sup> O C <sub>14</sub> H <sup>3</sup> Cl <sup>2</sup> O C <sub>15</sub> H <sup>3</sup> Cl <sup>2</sup> O C C <sub>15</sub> H <sup>3</sup> Cl <sup>2</sup> O C C C <sub>15</sub> H <sup>3</sup> Cl <sup>2</sup> O C C C C C C C C C C C C C C C C C C C
A + A A A A A A A A A A A A A A A A A A	0101 + 01 ×	
409.7788 417.8250 419.8220	423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728] 441.7428 443.7399 457.7377 459.7380 469.7780 471.7750 513.6775
4		ıo
1	TCDD TCDD (S) TCDD (S) HXCDPE PFK	TCDD TCDD (S) TCDD (S) HXCDPE PFK PCDF PCDF PCDF PCDD (S)
C <sub>12</sub> H, <sup>45</sup> Cl <sub>4</sub> O C <sub>12</sub> H, <sup>45</sup> Cl <sub>4</sub> O <sup>13</sup> C <sub>12</sub> H, <sup>45</sup> Cl <sub>4</sub> O <sup>13</sup> C <sub>12</sub> H, <sup>45</sup> Cl <sub>4</sub> O	C,2H,3Cl,02 C,2H,3Cl,3Cl02 13C,2H,3Cl,02 13C,2H,3Cl,37ClO C,2H,34Cl,37ClO C,F13	C12 H 30 C1 D2 C12 H 30 C1 D2 C12 H 30 C1 D2 C12 H 30 C1 J D2 C12 H 30 C1
M M M M	0 00X	
303.9016 305.8987 315.9419 317.9389 319.8965	321.8936 331.9368 333.9338 375.8364 [354.9792]	321.8836 331.9368 333.9338 375.8364 [354.9792] 339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 357.8516 367.8949 369.8919 409.7974
-	7000	9000T 000000000000000000000000000000000

(a) The following nuclidic masses were used:

 H = 1.007825 O = 15.994915 

 C = 12.000000  $^{36}CI = 34.968853$ 
 $^{19}C = 13.003355$   $^{7}CI = 36.965903$  

 F = 18.9984 

S = internal/recovery standard

LDC #: 23/88 x/ SDG #: 600 CONCA

only.

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:	/of /
Reviewer:	9
2nd reviewer:	

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

′.	[Y]	N	N/A
į	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?

Concent	ration	$= \frac{(A_{\circ})(I_{\circ})(DF)}{(A_{\circ})(RRF)(V_{\circ})(\%S)}$
$A_{x}$	=	Area of the characteristic ion (EICP) for the compound to be measured
$A_{is}$	=	Area of the characteristic ion (EICP) for the specific internal standard
l <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	<b>=</b>	Dilution Factor.
%S	=	Percent solids, applicable to soil and solid matrices

Conc. = (145)2.1 (4000) (10)(0.918)
(818871.38)(1.42582)(10)(0.918)

= 5.4 p-s/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	<del></del>	H H			
		<u> </u>			

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 14, 2010

LDC Report Date:

May 24, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D160437

Sample Identification

SSAO4-01-1BPC SA106-3BPC

## 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.

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

The exact mass of 380.9760 of PFK was verified. The static resolving power was at least 10,000 (10% valley definition).

## III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound for samples.

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

### V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	All samples in SDG G0D160437

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
SA106-3BPC	2,3,7,8-TCDF	1.4 pg/g	1.4U pg/g

Sample FB-04072010-RZC (from SDG G0D130519) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZC	4/8/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 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-HxCDF 1,2,3,4,6,7,8-HyCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF 0CDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.0 pg/L 1.5 pg/L 6.7 pg/L	All samples in SDG G0D160437

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
SSAO4-01-1BPC	<sup>13</sup> C-OCDD	34 (40-135)	OCDD	J (all detects) UJ (all non-detects)	Р
		:	OCDF	J (all detects) UJ (all non-detects)	

## X. Target Compound Identifications

All target compound identifications were within validation criteria.

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAO4-01-1BPC	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 G0D160437	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D160437	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

## XII. System Performance

The system performance was acceptable.

## XIII. Overall Assessment of Data

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

## XIV. Field Duplicates

No field duplicates were identified in this SDG.

## Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0D160437

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D160437	SSAO4-01-1BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0D160437	SSAO4-01-1BPC	OCDF	J (all detects)	Р	Compound quantitation and CRQLs (e)
G0D160437	SSAO4-01-1BPC SA106-3BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0D160437	SSAO4-01-1BPC SA106-3BPC	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 G0D160437

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D160437	SA106-3BPC	2,3,7,8-TCDF	1.4U pg/g	Α	bl

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D160437

No Sample Data Qualified in this SDG

### **Tronox Northqate Henderson**

_DC #: <u>23188G21</u>	_ VALIDATION COMPLETENESS WORKSHEET
SDG #: G0D160437	Stage 4
_aboratory: <u>Test America</u>	<del></del>

Date: <u>&lt;</u>	9
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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
1.	Technical holding times	A	Sampling dates: 4/14/10
II.	HRGC/HRMS Instrument performance check	A	
111.	Initial calibration	4	
IV.	Routine calibration/io	A	
V.	Blanks	âN	
VI.	Matrix spike/Matrix spike duplicates	M	No aplassid - No Qual
VII.	Laboratory control samples	A	109
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X	Target compound identifications	4	
XI.	Compound quantitation and CRQLs	in	
XII.	System performance	A	
XIII.	Overall assessment of data		
XIV.	Field duplicates	N.	
XV.	Field blanks	KW	FB-04070-RZC (40D130519)

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

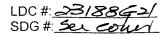
TB = Trip blank

EB = Equipment blank

### Validated Samples:

·			r	i		<u> </u>
1	SSAO4-01-1BPC <i>S</i>	11	0110455MB	21	31	
2	SA106-3BPC	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:	 			
			-	
				<u>.</u>

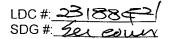


### **VALIDATION FINDINGS CHECKLIST**

	Page:_	/of <u>~</u>
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2nd	Reviewer:	

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

Validation Area	Yes	No	NA	Findings/Comments ´
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?		-		
Is the static resolving power at least 10,000 (10% valley definition)?	/			10 May 10
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?				
III. Initial calibration	,		·····	
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) $\leq$ 20% for unlabeled standards and $\leq$ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?				
IV. Continuing calibration				
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?				
V. Blanks				
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?				
VI. Matrix spike/Matrix spike duplicates		Accident Logical		
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?				
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				



### **VALIDATION FINDINGS CHECKLIST**

Page:_	<u> ユ</u> of <u>ユ</u>
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2nd Reviewer:	

VIII. Regional Quality Assurance and Quality Control		· · · · ·	<del>,</del> .	
Were performance evaluation (PE) samples performed?		/		
Were the performance evaluation (PE) samples within the acceptance limits?				
IX. Internal standards	T		<del></del>	
Were internal standard recoveries within the 40-135% criteria?	ļ,	<u>                                     </u>	-	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?		<u>.</u>		
X. Target compound identification		· T	Т	
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?	/	<u> </u>		
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?			ļ	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	/		<u> </u>	
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	/			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?		-	/	
Was an acceptable lock mass recorded and monitored?				
XI. Compound quantitation/CRQLs			Areas <u>Bress</u>	
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.			<u>                                     </u>	
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	a.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	1. 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:

1288820	see anno
C#: 23/2	G #:222
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### VALIDATION FINDINGS WORKSHEET

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Page:	Reviewer:	2nd Reviewer:

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".

Were all samples associated with a method blank?

Was a method blank analyzed for each matrix?

Was the blank contaminated? If yes, please see qualification below. n date: 4/2/10 Blank analysis date: 4/2/10 Blank extraction date: 4/2/10 N N N N N N N N N N N N

Conc. units: 23/9

Associated Samples:

Sample Identification 3 M 4 Blank ID 4552 M Ŋ W. Compound

	Associated Samples:
Blank analysis date:	
	S:
Blank extraction date:	Conc. units:

Compound	Blank ID	Sample Identification	

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

LDC #->3/88 f-2 SDG #:See Cover

## VALIDATION FINDINGS WORKSHEET FIELD Blanks

Reviewer: 2nd Reviewer: C

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:

Compound Blank ID	Blank ID	order:	Associated Samples:	ん( / ラSメ / Sample Identification	
	FB-04072010-RZC	5X			
v	0.77	0.00385			
Q	0.74	0.0037			
ш	0.82	0.0041			
4	4.2	0.021			
9	37	0.185			
I	0.57	0.00285			
	96.0	0.0048			
٦	0.67	0.00335			
<b>Y</b>	1.1	0.0055			
-1	0.96	0.0048			
M	1.0	0.005			
Z	1.0	0.005			
0	2.1	0.0105			
a	1.5	0.0075			
O	6.7	0.0335			
CRQL					

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

	Z
1	Cen
33/	26
#	#
LDC	SDG

## **VALIDATION FINDINGS WORKSHEET**

Page: Reviewer:\_ 2nd Reviewer:

Internal Standards

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

Are all internal standard recoveries were within the 40-135% criteria? Was the S/N ratio all internal standard peaks > 10?

Y N N/A

*	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135%)	-135%)	Qualifications
		/	I	<i>18</i> 0	4) H	40-135)	1/14 f (f. 8)
					)	(	
					)	) (	
					)	(	
					)		
					)	)	
					)	(	
					)	(	
					)	(	
					J	)	
					)	) (	
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					)	^	
						^	
					)	^	
					)		
					)	(	
						- (	
						^	
				Andrew Company	)	) [	
		Internal Standards	Check Standard Used		Intern	Internal Standards	Check Standard Used
Ą	<sup>13</sup> C-2,3,7,8-TCDF	:DF		Ġ.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	ЭF	
B.	<sup>13</sup> C-2,3,7,8-TCDD	00;		Ŧ.	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	QC	
Ö	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF		-	13C-OCDD		
Ö	<sup>13</sup> C-1,2,3,7,8-PeCDD	•eCDD		ᅶ	13C-1,2,3,4-TCDD		
ш	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	-HxCDF		اد	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
Ш	13C-12.3.6.7.8-HxCDD	-HxCDD					

LDC #:03/5047

### Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

\ \ Reviewer: Page:

2nd Reviewer:

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).

Qualifications	1 Jehn (e)	UK(K)								
Associated Samples	/	m								
gods > calib lange.	A	EMPC MOUBLES	(& Hac)							
Sample ID	/	$\mathcal{M}($				-				
Date							0			
#										

Comments: See sample calculation verification worksheet for recalculations

SDG #: 2010 4

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

/of/ Page: Reviewer: 2nd Reviewer:

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:

 $A_{\rm s}$  = Area of associated internal standard  $C_{\rm k}$  = Concentration of internal standard X = Mean of the RRFs

RRF =  $(A_{\nu}/(C_{\nu})/(A_{\nu})(C_{\nu})$ 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,

L									
				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Average RRF (initial)	RRF ( SStd)	RRF (CS-3 std)	%RSD	%RSD
-	19th	0/0/1	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.860	0.860	787	7.8.0	4.01	801
	(105)	01/01/	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	4560	0.934	0.95	0.95	00/	8 61
			1,2,3,6,7,8-HxCDD (19C-1,2,3,6,7,8-HxCDD)	1.058	1.058	00.	601	17.2	011
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	0.998	866.0	1.05	501	٤-5/	127
			OCDF ("C-OCDD)	1.437	1.437	1.52	1.52	141	140
2	10/2	11.1.	2,3,7,8-TCDF ( <sup>15</sup> C-2,3,7,8-TCDF)	1.088	1.088	1.10	0/:/	e si	24.
		1/1/1	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)						
			ocpf (4c-ocpb)						
က	10/2	1 //	2,3,7,8-TCDF (*G-2,3,7,8-TCDF)	3460	0.945	86.0	86.0	4.44	4 33
	(405)	01/2/14	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.001	1.02	1.04	104	202	1000
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.114	1.114	6/:/	0 !!	533	11.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1.072	1.072	//:/	///	2,60	3.75
			OCDF ("C-OCDD)	1.445	1.445	1/2/	1.01	dov	289

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.

· SDG # Lee COUL LDC #:23/8893

### Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$ 

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

 $A_x$  = Area of compound,  $C_x$  = Concentration of compound,

 $A_{\mathtt{k}} = \mathsf{Area}$  of associated internal standard  $C_{\mathtt{k}} = \mathsf{Concentration}$  of internal standard

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>0%</b>	۵%
-	Mayor	· ·	2,3,7,8-TCDF (*3C-2,3,7,8-TCDF)	0.860	0.93	0.93	7.0	01
	, ,	4/21/10	4/>//v 2.3,7,8-TCDD (°3C-2,3,7,8-TCDD)	7786.0	0.93	0.93	0.	0.1
		`	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	850.1	1.15	1.15	00 00	00.00
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	866.0	1.06	1.06	6 2	1
			OCDF (3C-OCDD)	1.437	7.5.1	1.54	1.7	7.7
8	BUTUALISE	/ /	2,3,7,8-TCDF (¹°C-2,3,7,8-TCDF)	2460	0.938	86.0	1.3,	/. W
		2/3/10	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.021	560	26.0	6.8	00.00
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.114	1.15	1.15	7.4	N.A
			1,2,3,4,6,7,8-HpCDD ( <sup>3</sup> C-1,2,4,6,7,8,-HpCDD)	1.072	1.01	1.0.1	4.4	7.4
			OCDF (3c-OCDD)	1.445	041/40	1.40	2.4	3.4
ဗ	EdAN-/10562	1/10502 4/1/13	2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	1.088	1.15	1.16	5.8	5.8
		silet.	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (°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 ("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 #: 23/8/84 = 3 SDG #: 28 COULT

### Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

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

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratoy 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 = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0/10455

d		Recalculated						-			•
I CS/I CSD	RPD	Reported									
ים. קי	ecovery	Recalc									
I CSD	Percent Recovery	Reported									
SS	Recovery	Recalc	96	103	95	8	99	,			
9	Percent Recovery	Reported	96	601	95	18	0				
Sample	tration	LCSD	NA								
Spiked	Concentration	SJI	19.1	103	952	4.8	861				
ike	Added (PS/S)	I CSD	NX								
os	**************************************	S	20.0	981	_	->	200				
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF				

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

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ol nol	Elemental Composition	Analyte
-	303.9016	Σ	o'h"go"o	TCDF	4	407.7818	M+2	C.,H <sup>25</sup> Cl, <sup>37</sup> ClO	HDCDF
	305.8987	M+2	C <sub>12</sub> H <sub>3</sub> **C1 <sub>3</sub> **C10	TCDF		409.7788	M +	C,H*Cl,*7Cl,O	HPCDF
	315,9419	Σ	13C12H, 35C1,O	TCDF (S)		417.8250	Σ	13C,2H3CI,O	HpCDF (S)
	317.9389	M+2	13C12H, 45C137C1O	TCDF (S)		419.8220	M+2	13C12H35CI637CIO	HPCDF
	319.8965	Σ	C;_H,**Ci_O,	TCDD		423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>8</sub> 37ClO <sub>2</sub>	Ньсрр
	321.8936	M+2	C12H,**C13**C102	TCDD		425.7737	M+4	Ci,Hach, 37Cl,O,	HPCDD
	331.9368	Σ	13C1,H, #C1,O3	TCDD (S)		435.8169	M+2	ISC, HSCI 37CIO,	HPCDD (S)
	333.9338	M+2	13C12H, 35C1, 37C1O,	TCDD (S)		437.8140	M+4	13C, H*C  37C ,O,	HpCDD (S)
	375.8364	M+2	C <sub>1</sub> ,H <sub>4</sub> *Cl <sub>5</sub> *7ClO	HXCDPE		479.7165	M+4	C,H <sup>35</sup> Cl, <sup>37</sup> Cl,O	
	[354.9792]	LOCK		PFK		[430.9728]	LOCK	, , , , , , , , , , , , , , , , , , ,	PFK
2	339,8597	M+2	C, H, **CI, *7CIO	PeCDF	5	441,7428	0+W	C.36Cl.37ClO	OCOF
	341.8567	M+4	0,1,1,30,1,0	PecDF		443.7399	Σ + +	C. &CI.37CI.O	OCDF
	351.9000	M+2	13C, H, 35C  37C10	PeCDF (S)		457,7377	M+2	C. 301 300	oce
•	353.8970	M+4	13C; H, 35Cl, O	PeCDF (S)		459.7348	- M	0,0%(0%,0)	ocpp
	355.8546	M+2	C,H,*CI,*OO,	PeCDD		469.7780	M+2	OD2-12-21-21-21-21-21-21-21-21-21-21-21-21	OCDD (S)
	357.8516	M+4	C;H,36C1,37C1,O;	PeCDD		471.7750	M+4	10, 30, 30, 0, 10, 10, 10, 10, 10, 10, 10, 10, 10	OCDD (S)
	367.8949	M+2	13C12H3**C14**CIO2	PeCDD (S)		513.6775	<b>⊼</b> 4+	C;3*C 3*C ;O	DCDPE
	369.8919	M+4	13C <sub>12</sub> H <sub>3</sub> 3Cl <sub>3</sub> 3′Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)		[422.9278]	LOCK	, , <u>, , , , , , , , , , , , , , , , , </u>	PFK
	409.7974	M+2	C <sub>12</sub> H <sub>3</sub> 33Cl <sub>8</sub> 37ClO	HPCDPE	-			-	
	[354.9792]	LOCK	, F.,	PFK					
8	373.8208	M+2	C,H,*C ,*7C O	HXCDF					
	375.8178	M+4	C,'H, **Cl, **Cl, O	HXCDF					
	383.8639	Σ	10,0%,H,00,0	HXCDF (S)					
	385.8610	CI.	13C1,H,35CI,37CIO	HXCDF (S)					
	389.8156	M+2	C, H, scl, sclo,	HXCDD					
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>30</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	НХСД					
	401.8559		13C12H2 35CIG2	HXCDD (S)					
	403.8529		13C12H23CI43CI2O2	HXCDD (S)					
	445.7555	M+4	C12H2*C18*3C12O	OCDPE					
	[430.9728]	X O	G,F <sub>1</sub> ,	PFK					

### (a) The following nuclidic masses were used:

H = 1.007825 O = 15.994915 C = 12.000000 %CI = 34.968853 <sup>37</sup>CI = 36.965903 F = 18.9984

S = internal/recovery standard

### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Were all reported results recalculated and verified for all level IV samples?

Page:	/of_/
Reviewer:	Q
2nd reviewer:_	

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

V N	N/A	Were all recalculated results for detected ta	rget compounds agree within 10.0% of the reported results?
Conce	entration :	= (A_)(L_)(DF) (A_)(RRF)(V_)(%S)	Example:
$A_{x}$		Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D:
$A_{is}$		Area of the characteristic ion (EICP) for the specific internal standard	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	Conc. = (19461360) (2000) ( (8279200) (1072) (10.08) (927)
$V_{o}$	=	Volume or weight of sample extract in milliliters (ml)	

RRF Relative Response Factor (average) from the initial calibration Dilution Factor. Df %S Percent solids, applicable to soil and solid matrices

Exε	ample:		
		,	T

Conc. = (19461360) (200 (82792200) (1.072	0)(/0.08	159271
=46.9 P3/g		

4	Samula ID	Compound	Reported Concentration	Calculated Concentration	Qualification
#	Sample ID	Compound	( )	( )	Quantication
					ļ
				·	
<u> </u>	<u> </u>				

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

Project/Site Name:

Tronox LLC Facility, PCS, Henderson, Nevada

**Collection Date:** 

April 15, 2010

LDC Report Date:

May 21, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0D170488

Sample Identification

SSAN6-03-1BPC SA58-3BPC

### 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.
- 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
0110455MB	4/20/10	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.31 pg/g 0.31 pg/g 0.30 pg/g	All samples in SDG G0D170488

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
SA58-3BPC	1,2,3,7,8-PeCDF	0.49 pg/g	0.49U pg/g
	1,2,3,4,6,7,8-HpCDF	1.1 pg/g	1.1U pg/g

Sample FB-04072010-RZC (from SDG G0D130519) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB-04072010-RZC	4/8/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	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 1.1 pg/L 0.96 pg/L 1.0 pg/L 1.0 pg/L 1.0 pg/L 2.1 pg/L 6.7 pg/L	All samples in SDG G0D170488

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

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-03-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	38 (40-135) 28 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	P
SA58-3BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF <sup>13</sup> C-OCDD	38 (40-135) 20 (40-135)	OCDD 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) UJ (all non-detects)	Р

### 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 G0D170488	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0D170488	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	Α

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 G0D170488

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0D170488	SSAN6-03-1BPC SA58-3BPC	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)	Р	Internal standards (%R) (i)
G0D170488	SSAN6-03-1BPC SA58-3BPC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0D170488	SSAN6-03-1BPC SA58-3BPC	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 G0D170488

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0D170488	SA58-3BPC	1,2,3,7,8-PeCDF 1,2,3,4,6,7,8-HpCDF	0.49U pg/g 1.1U pg/g	Α	bl

Tronox LLC Facility, PCS, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0D170488

No Sample Data Qualified in this SDG

### Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 23188H21 VALIDATION COMPLETENESS WORKSF
SDG #: G0D170488 Stage 2B
Laboratory: Test America

/ /
<i>0 ب 0حول</i> Date:
Page:
Reviewer: Q'
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
1.	Technical holding times	A	Sampling dates: 4/,5/10
11.	HRGC/HRMS Instrument performance check	A	/ /
III.	Initial calibration	$\downarrow$	
IV.	Routine calibration/I	À	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	w	No splassid - No Conal
VII.	Laboratory control samples	A	105
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
Χ.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	ØN.	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	M	FB-040TiD RZC (40D130519)

Note: A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank Is detected D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples:

1	SSAN6-03-1BPC	S 11	0110455173	21	31	
2	SA58-3BPC	/ 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.Hp.CDE	1-
A 102.00				O. Iotal npc00
0, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,	6. Octob	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V Total TODE
1				V. IOGAL LCOP
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. Jotal Pecor
D. 1,2,3,6,7,8-HxCDD	1. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	A Total Boods	
			O. Iolai racoo	A. Lotal 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 H×COO	
				Y. DOM TOCOT

Notes:

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### VALIDATION FINDINGS WORKSHEET Blanks

Page: / of /	Reviewer:	2nd Reviewer:
		ŭ

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".

Were all samples associated with a method blank?

Was a method blank analyzed for each matrix?

Was the blank contaminated? If yes, please see qualification below. 

Associated Samples: Blank extraction date: 4/20/10 Blank analysis date: 4/26/10 Conc. units: 79/

ıtion							
Sample Identification							
	×						
	1>5×						
			-				
	N		N.840	17/1-1			
Blank ID	SERVED 10	0.31	150	030			
Compound			£	U	7		

Blank analysis date:		
Blank extraction date:	Conc. units:	

	Blank ID				
conc. ames.	Compound				

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#:23/88/4> SDG #: See Cover

## VALIDATION FINDINGS WORKSHEET

Field Blanks

2nd Reviewer: Page: Reviewer:

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

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

Associated sample units: Blank units: pg/L Associated Sampling date: 4/8/10 Field blank type: (circle-fond) Eight Bl

Compound Blank ID	E) Field Blank's Rinsate Blank ID	/ Other:	Associated Samples:	- 11	M (>5x		
	G vibio			San	Sample Identification		
	FB-04072010-RZC	2X					
	0.77	0.00385					
	0.74	0.0037					
	0.82	0.0041					
	4.2	0.021					
	37	0.185					
	0.57	0.00285					
	0.96	0.0048					
	0.67	0.00335					
	1.1	0.0055					
	96.0	0.0048					
	1.0	0.005					
	1.0	0.005					
	2.1	0.0105					
	1.5	0.0075					
	6.7	0.0335					

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

SDG #: Les cours LDC #: 23/88/4/

## **VALIDATION FINDINGS WORKSHEET**

Page: of

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".

XAN N/A

Are all internal standard peaks > 10?

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

#	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135%)	.: 40-135%)	Qualifications ( )
			4	(1)	38	(40-135	JUNY (4.0-8)
			Н	7	28	)	
						)	
		d	Þ	38	()O	)	
			Н	9	0	/\hat{\alpha}	۲
						)	
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						)	
						<b>)</b>	
						)	
П						)	
						)	
						)	
						)	
		Internal Standards	Check Standard Used		и	Internal Standards	Check Standard Used
Ą.	<sup>13</sup> C-2,3,7,8-TCDF	CDF		.S	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	1pCDF	
B.	13C-2,3,7,8-TCDD	cob		Ï	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	ЧрСDD	
Ċ	<sup>13</sup> C-1,2,3,7,8-PeCDF	PeCDF		<b></b> :	13C-OCDD		
Ο.	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		ᅶ	13C-1,2,3,4-TCDD		
Ē.	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	8-HxCDF		نـ	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	CDD	
F	<sup>13</sup> C-1 2 3 6 7 8-HxCDD	8-НхСОО					

LDC #: <u>23/884</u>2/ SDG #: <u>36/ GOV</u>EN

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of Reviewer:

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/N

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).

4 cults (k)	Sample ID
	41) 24
	(8 + lag

Comments: See sample calculation verification worksheet for recalculations