

#### LABORATORY DATA CONSULTANTS, INC.

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

Northgate Environmental Management, Inc.

October 27, 2010

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

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

**Data Validation** 

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on September 30, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

#### **LDC Project # 24089:**

#### SDG # Fraction

G0G290426, G0H110567, G0H110575 G0H120594, G0H180547, G0H190584

G0H190600, G0H250498

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

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Attachment 1

DL 09/30/10

LDC #: 24089

SDG #: <u>G0G290426</u>, <u>G0H110567</u>, <u>G0H110575</u>, <u>G0H120594</u> <u>G0H180547</u>, <u>G0H190584</u>, <u>G0H190600</u>, <u>G0H250498</u> Page: 1 of 1 Reviewer: TC 2nd Reviewer: JE

#### Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	Х			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	Х			
III. EDD Lab Anomalies				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			х	See EDD_discrepancy_ form_LDC24089_102710.doc
IV. EDD Delivery				101m_25 C2 1005_102/10.duc
Was the final EDD sent to the client?	X			

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

June 29, 2010

**LDC Report Date:** 

October 15, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0G290426

Sample Identification

SSAK6-06-2BPC SSAK6-06-2BPCMS SSAK6-06-2BPCMSD

#### 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.

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
021094	7/29/10	OCDD	1.7 pg/g	All samples in SDG G0G290426

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:

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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

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Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
021094	7/29/10	OCDD	1.7 pg/g	All samples in SDG G0G290426

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
SSAK6-06-2BPC	OCDD	7.2 pg/g	7.2U pg/g

No field blanks were identified in this SDG.

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

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

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

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

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

Not applicable.

#### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK6-06-2BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 26 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

#### X. Target Compound Identifications

Raw data were not reviewed for this SDG.

#### XI. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0G290426	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0G290426	All compounds reported by the lab 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 Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0G290426

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0G290426	SSAK6-06-2BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0G290426	SSAK6-06-2BPC	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0G290426	SSAK6-06-2BPC	All compounds reported as EMPC.	JK (all detects)	Α	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0G290426

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0G290426	SSAK6-06-2BPC	OCDD	7.2U pg/g	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0G290426

No Sample Data Qualified in this SDG

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METL	AOD: UDCC/UDMC Dia	vino/F	\ibanzafı ıra	(EDA CI	M 040 M-	4h = 4 0000	`		2nd Reviewer:
VIC I F	HOD: HRGC/HRMS Did	XIIIS/L	nbenzolurar	IS (EPA S	VV 846 IVIE	tnoa 8290	)		
The s	amples listed below we	re revi	ewed for ea	ach of the f	ollowing v	alidation a	reas. Validatio	on findir	ngs are noted in attached
/allua	tion findings worksheet	S.							
	Validatio	n Area					Comm	ents	
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II.	HRGC/HRMS Instrument	perform	ance check	4	Journal				
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IV.	Routine calibration/ICV			4					
V.	Blanks			51A					
VI.	Matrix spike/Matrix spike d	luplicate	es	4					
VII.	Laboratory control sample			A	ic	2			·
VIII.	Regional quality assurance		uality control	N					
IX.	Internal standards			SW					
X.	Target compound identification	ations		N					
XI.	Compound quantitation an	d CRQI	_S	510					
XII.	System performance			N					
XIII.	Overall assessment of data	а		A					
XIV.	Field duplicates			N					
XV.	Field blanks	···		N					
Note:	A = Acceptable N = Not provided/applicab SW = See worksheet	le	R = Rin	o compound: sate eld blank	s detected	TB =	Duplicate : Trip blank : Equipment blan	k	
/alidate	ed Samples: SO/L								
1	SSAK6-06-2BPC	11	021094	<u> </u>	21			31	
	SSAK6-06-2BPCMS	12			22			32	
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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. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

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# VALIDATION FINDINGS WORKSHEET

Blanks

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".

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? Blank analysis date: 8 13/10 Blank extraction date: 729 10

Associated samples:

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Sample Identification 7,2/ ppea120 Blank ID Compound J Conc. units:\_

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 # 24089A 2/

# VALIDATION FINDINGS WORKSHEET

Internal Standards

Page:

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

he 40-135% criteria?	
Are all internal standard recoveries were within the 4	Was the S/N ratio all internal standard peaks > 10?
N/A/A	∀N N

#	Date	Lab ID/Reference	Internal Standard	% Rec	% Recovery (Limit: 40-135%)		Qualifications	
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П		Internal Standards	Check Standard Used		Recovery Standards	and a second sec	Check Standard Used	ard Used
A	<sup>13</sup> C-2.3.7.8-TCDF	DF		K. 13C-1.	<sup>13</sup> C-1.2.3.4-TCDD			
a	13C-2,3,7,8-TCDD	DD			<sup>13</sup> C-1 2 3 7 8 9-HxCDD			
d	<sup>13</sup> C-1 2 3 7 8-PeCDE	PACDE		M				
4	<sup>13</sup> C-1,2,3,7,8-PeCDD	чеспп		Z				
Щ	<sup>13</sup> C-1,2,3,6,7,8-HxCDE	-HxCDE		q				
щ	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	-HxCDD		Д				
व	13C-1,2,3,4,6,7,8-HpCDE	, 8-НрСDF		С				
크	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	, 8-НрСDD		В				
7	്പോഗവ							

LDC# 24089A2/ SDG#: Accomp

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

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

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

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

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Qualifications	1/A dut (4p)											
Associated Samples	) IA									THE PROPERTY OF THE PROPERTY O		
Finding	Al compound reported	beton 1 pal "										
Sample ID												
Date												
#												

Comments: See sample calculation verification worksheet for recalculations

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7 through August 9, 2010

**LDC Report Date:** 

October 15, 2010

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H110567

#### Sample Identification

SSAJ3-02-12BPC

SSAJ3-02-15BPC

SSAJ3-02-16BPC

SSAJ3-02-19BPC\*\*

SSAJ3-02-8BPC FD

SSAJ3-02-8BPC

SSAJ3-02-5BPC

EB-08072010

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

#### Introduction

This data review covers 7 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

#### I. Technical Holding Times

All technical holding time requirements were met.

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

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

Instrument performance was checked at the required daily frequency.

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

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

#### III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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
0228363	8/16/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.096 pg/g 0.80 pg/g 0.064 pg/g 0.26 pg/g 0.087 pg/g 0.16 pg/g	All soil samples in SDG G0H110567
0225283	8/13/10	OCDD	3.9 pg/L	All water samples in SDG G0H110567

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
SSAJ3-02-12BPC	1,2,3,7,8,9-HxCDD	0.20 pg/g	0.20U pg/g
	OCDD	1.2 pg/g	1.2U pg/g
SSAJ3-02-15BPC	1,2,3,7,8,9-HxCDD	0.15 pg/g	0.15U pg/g
	OCDD	1.4 pg/g	1.4U pg/g
SSAJ3-02-16BPC	1,2,3,7,8,9-HxCDD	0.28 pg/g	0.28U pg/g
	1,2,3,4,6,7,8-HpCDD	0.27 pg/g	0.27U pg/g
	OCDD	1.3 pg/g	1.3U pg/g
SSAJ3-02-19BPC**	1,2,3,4,6,7,8-HpCDD	0.16 pg/g	0.16U pg/g
	OCDD	0.83 pg/g	0.83U pg/g
	1,2,3,4,7,8-HxCDF	0.24 pg/g	0.24U pg/g
	1,2,3,4,6,7,8-HpCDF	0.78 pg/g	0.78U pg/g
	1,2,3,4,7,8,9-HpCDF	0.29 pg/g	0.29U pg/g
SSAJ3-02-8BPC_FD	1,2,3,7,8,9-HxCDD	0.15 pg/g	0.15U pg/g
	1,2,3,4,6,7,8-HpCDD	0.36 pg/g	0.36U pg/g
	OCDD	0.93 pg/g	0.93U pg/g
SSAJ3-02-8BPC	1,2,3,7,8,9-HxCDD	0.16 pg/g	0.16U pg/g
	1,2,3,4,6,7,8-HpCDD	0.24 pg/g	0.24U pg/g
	OCDD	1.5 pg/g	1.5U pg/g
SSAJ3-02-5BPC	1,2,3,4,6,7,8-HpCDD	0.21 pg/g	0.21U pg/g
	OCDD	1.5 pg/g	1.5U pg/g
	1,2,3,4,7,8-HxCDF	0.23 pg/g	0.23U pg/g
	1,2,3,4,6,7,8-HpCDF	1.0 pg/g	1.0U pg/g
EB-08072010	OCDD	7.6 pg/L	7.6U pg/L

Sample EB-08072010 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-08072010	8/13/10	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	7.6 pg/L 1.5 pg/L 2.0 pg/L 1.0 pg/L 3.6 pg/L 9.5 pg/L	No associated samples in this SDG

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

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

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

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

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

Not applicable.

#### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAJ3-02-16BPC	13C-OCDD	32 (40-35)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ3-02-19BPC**	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-35) 30 (40-35) 36 (40-35)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-02-8BPC_FD	<sup>13</sup> C-OCDD	29 (40-35)	OCDD	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
SSAJ3-02-5BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-35) 19 (40-35) 37 (40-35)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

#### X. Target Compound Identifications

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

#### XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAJ3-02-12BPC SSAJ3-02-16BPC SSAJ3-02-19BPC** SSAJ3-02-8BPC SSAJ3-02-5BPC EB-08072010	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H110567	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H110567	All compounds reported by the lab 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 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 SSAJ3-02-8BPC\_FD and SSAJ3-02-8BPC were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentra	tion (pg/g)	RPD	Difference		
Compound	SSAJ3-02-8BPC_FD	SSAJ3-02-8BPC	(Limits)	(Limits)	Flags	A or P
1,2,3,6,7,8-HxCDD	0.11	0.15	-	0.04 (≤2.7)	-	<u>-</u>
1,2,3,7,8,9-HxCDD	0.15	0.16	-	0.01 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	0.36	0.24	-	0.12 (≤2.7)	-	-
OCDD	0.93	1.5	-	0.57 (≤5.3)	-	-
2,3,7,8-TCDF	0.51U	0.37	-	0.14 (≤0.51)	-	-
1,2,3,7,8-PeCDF	0.52	0.48	-	0.04 (≤2.7)	-	-
2,3,4,7,8-PeCDF	0.30	0.24	-	0.06 (≤2.7)	<del>-</del> .	-
1,2,3,4,7,8-HxCDF	1.0	0.67	-	0.33 (≤2.7)	-	-
1,2,3,6,7,8-HxCDF	0.73	0.68	-	0.05 (≤2.7)	•	-
2,3,4,6,7,8-HxCDF	0.23	0.16	-	0.07 (≤2.7)	-	-
1,2,3,7,8,9-HxCDF	0.29	0.26	-	0.03 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDF	2.6	1.9	-	0.7 (≤2.7)	-	-
1,2,3,4,7,8,9-HpCDF	0.98	0.75	_	0.23 (≤2.7)	•	-

	Concentral	tion (pg/g)	222	Diff			
Compound	SSAJ3-02-8BPC_FD	SSAJ3-02-8BPC	RPD (Limits)	Difference (Limits)	Flags	A or P	
OCDF	5.3	4.4	-	0.9 (≤5.3)	-		

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H110567

SDG	Sample	Compound	Flag	A or P	Reason
G0H110567	SSAJ3-02-16BPC SSAJ3-02-8BPC_FD	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H110567	SSAJ3-02-19BPC** SSAJ3-02-5BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H110567	SSAJ3-02-12BPC SSAJ3-02-16BPC SSAJ3-02-19BPC** SSAJ3-02-8BPC SSAJ3-02-5BPC EB-08072010	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (2nd column confirmation) (o)
G0H110567	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-16BPC SSAJ3-02-19BPC** SSAJ3-02-8BPC_FD SSAJ3-02-8BPC SSAJ3-02-5BPC EB-08072010	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0H110567	SSAJ3-02-12BPC SSAJ3-02-15BPC SSAJ3-02-16BPC SSAJ3-02-19BPC** SSAJ3-02-8BPC_FD SSAJ3-02-8BPC SSAJ3-02-5BPC EB-08072010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

#### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H110567

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H110567	SSAJ3-02-12BPC	1,2,3,7,8,9-HxCDD OCDD	0.20U pg/g 1.2U pg/g	Α .	bl
G0H110567	SSAJ3-02-15BPC	1,2,3,7,8,9-HxCDD OCDD	0.15U pg/g 1.4U pg/g	Α	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H110567	SSAJ3-02-16BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.28U pg/g 0.27U pg/g 1.3U pg/g	А	bl
G0H110567	SSAJ3-02-19BPC**	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	0.16U pg/g 0.83U pg/g 0.24U pg/g 0.78U pg/g 0.29U pg/g	А	ы
G0H110567	SSAJ3-02-8BPC_FD	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.15U pg/g 0.36U pg/g 0.93U pg/g	А	bl
G0H110567	SSAJ3-02-8BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	0.16U pg/g 0.24U pg/g 1.5U pg/g	А	bi
G0H110567	SSAJ3-02-5BPC	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	0.21U pg/g 1.5U pg/g 0.23U pg/g 1.0U pg/g	A	bl
G0H110567	EB-08072010	OCDD	7.6U pg/L	А	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0H110567

No Sample Data Qualified in this SDG

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_DC #:	24089B21	VALIDATION COMPLETENESS WORKSHEET	Date:/0//2///
SDG #:	G0H110567	_ Stage 2B/4	Page: / of/
_aboratoı	ry: Test America	<del>-</del>	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
<u> </u>	Technical holding times	A	Sampling dates: 8/7 — 8/9/10
11.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	A	
IV.	Routine calibration/ <del>ICV</del>	A	
V.	Blanks	یس	
VI.	Matrix spike/Matrix spike duplicates	А	SS:AI3-03-5BPC US ID
VII.	Laboratory control samples	Α	ريا
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	رىبى	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	7=5+6
XV.	Field blanks	س ی	EB=8

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

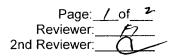
Validated Samples: \*\* Indicates sample\_underwent Stage 4 validation

	5011 + 1	nater				
1	SSAJ3-02-12BPC	11	0728363	21	31	
2	SSAJ3-02-15BPC	12	0225283	22	32	
3	SSAJ3-02-16BPC	13		23	33	,
4	SSAJ3-02-19BPC**	14		24	34	
5	SSAJ3-02-8BPC_FD	15		25	35	
6	SSAJ3-02-8BPC	16		26	36	
7	SSAJ3-02-5BPC	17		27	37	
8	EB-08072010	18		28	38	
9		19		29	39	
10		20		30	40	

Notes:		

### LDC #: 24089 B2 SDG #: LONE

#### **VALIDATION FINDINGS CHECKLIST**



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?		<u>-</u> هر در		
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?		Patricular in a second		
V. Blanks				n Su
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?	_			ONE POR AN AN AN AND AND AN AND AN AND AN AND AN AND AN AND AN AND AND
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?				Degression and the control of schools in the control of the contro
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?				

LDC #: 24089 B2 SDG #: 100 comm

#### **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 7
2nd Reviewer: 7

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

# **VALIDATION FINDINGS WORKSHEET**

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

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

Notes:

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2
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#
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# VALIDATION FINDINGS WORKSHEET

Blanks

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

> 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)

Were all samples associated with a method blank?

N N N

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

Blank extraction date: 위 네이

Blank analysis date:  $8|\infty|$ 

All soils (b) Associated samples:

\* FMR

6.21#/N 0.23\*/u 2 0.7 1.5 ( 0.24 /4 h/2)·0 ها 0.92/4 0.36/4 0,15/4 Ş L Sample Identification 0.10\*/4 0.83/4 0.24\*/4 n/\*8L.0 0.29/4 2 0.28/11 1/12.a 1.3/ 0.15\*/4 Z 1. T I 1 Ĺ 1.2\*/4 0.20/4 ١ 0.32 0%0 ٥ 0.48 0.435 ふえ 5 02283 D.064 \* \$180.a 0.096 Blank ID \* \* 98.0 0.12 0.26 0.16 Compound Ø Conc. units: 🙉 D ٩ 77 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".

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## VALIDATION FINDINGS WORKSHEET Blanks

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 all samples associated with a method blank? 

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Blank analysis date: 왕 | 2 | Was the method blank contaminated? Blank extraction date: 8/13/10

Conc. units:

Associated samples:\_

al waln (bl) \* FMPC

Sample Identification \* 875770 Blank ID w 2 Compound 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".

## LDC# 24089/82/

## VALIDATION FINDINGS WORKSHEET Field Blanks

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

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

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

Brank units: 20 Associated sample units: 29

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

Associated Samples:

\* FMPC

Sample Identification \* 0% \* \* \* Blank ID 1/2 0: 60 97 į Compound Ф 3 A #

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 # 24089/32/

# VALIDATION FINDINGS WORKSHEET

Page: \_of\_

2nd Reviewer: 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) N N/A

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

*	Date	Lab ID/Reference	Internal Standard	% Re	% Recovery (Limit: 40-135%)	40-135%)	Qua	Qualifications	
1		3	Н		32 (	(58-01	) 9/W/L	(۲) و	Out 6,0
			The state of the s		)			,	-
					)	(		~	
		4	Н	8	35	(		land	A P
			L	('')	3 O	)			<i>д</i>
			$\mathcal{P}$		36	· ·			B, P
					)	(			
					)	(			
		Ŋ			29 (	^			ଉ, ଚ
					<b>)</b>	(			•
					)	(			
		7	H		35 (	_			ഥ
			T		) 61				9,0
			6		37 (	^ <del>}</del>	<b>→</b>	<b>→</b>	9
					)	(			
					)	(			
$\dashv$					)	(			
寸									
					)	)		-	
		Internal Standards	Check Standard Used		Rec	Recovery Standards	ch Ch	Check Standard Used	Used
┛	<sup>13</sup> C-2.3.7.8-TCDF	DF		13C-	<sup>13</sup> C-1.2.3.4-TCDD				
В	13C-2 3 7 8-TCDD	DD		130	<sup>13</sup> C-1 2 3 7 8 9-HxCDD	סם			
d	<sup>13</sup> C-1 2 3 7 8-PeCDE	PECDE		M					
Д	<sup>13</sup> C-1 2 3 7 8-PeCDD	эеспр		Z					
Ц	13C-1,2,3,6,7,8-HxCDE	-HxCDF		q					
Щ	<sup>13</sup> C-1,2,3,6,7,8-HxCDD	-HxCDD		а					
d	13C-1,2,3,4,6,7,8-HpCDE	, 8-НрСDF		d					
Ħ	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	, 8-НрСDD		22					
7	130 000			<u>+</u>					

LDC#: 2/08782/

## Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page:

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".

X N N/A

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

*	oteC	Polymon	Fibelia	Solumno S bodo i coco A	Cucitostille
			All compounds reported below PQL	Washington	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		Н	no and column	1,3,4 b,7 x	none/p (6)
			/ ~		)

Comments: See sample calculation verification worksheet for recalculations

LDC#:\_2408982)

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

Page:	<u>/</u> of/
Reviewer:	17
2nd Reviewer:	W

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?

\* EMPC

	Concentrat	tion pg/g)	(pg/g)	(pg/g)	Qualifications
Compound	5	6	Difference	Limits	(Parent Only)
D	0,11 🛠	0.15 🛪	0.04	≤2.7	
E	0.15	0.16	0.01	≤2.7	
F	0.36	0.24 ⊁	0.12	≤2.7	
G	0.93	1.5	0.57	≤5.3	
Н	0.51U	0.37 💥	0.14	≤0.51	
1	0.52 🛠	0.48	0.04	≤2.7	:
J	0.30	0.24	0.06	≤2.7	
к	1.0	0.67	0.33	≤2.7	
L	0.73 拌	0.68	0.05	≤2.7	
М	0.23 🔻	0.16	0.07	≤2.7	
N	0.29	0.26	0.03	≤2.7	
0	2.6	1.9	0.7	≤2.7	
Р	0.98	0.75	0.23	≤2.7	
Q	5.3 *	4.4	0.9	≤5.3	

V:\FIELD DUPLICATES\24089B21.wpd

LDC# 24089.82, ere comb SDG#:\_

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

2nd Reviewer:\_ Reviewer.

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

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

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

 $A_x$  = Area of compound,  $C_x$  = Concentration of compound, S = Standard deviation of the RRFs,

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = 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 ( C&う std)	RRF (シろ std)	%RSD	%RSD
-	1041	01/02/1	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	700.1	1.00%	7.081.1		4.19165	5961.4
		<u>.</u>	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	30	١:١٥٥	1.0190	1.0790	8.10393	
		ī	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.146	0-1-1	1.0831	1.083	L5906.8	
		ı	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1.086	1.086	2110.1	6110.1	ZzL10.9	
			OCDE (130-OCDD)	1.54 9	1.89	++4607	1.4726	Q21 H.25	Ц
2			2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)			1.47%			_
			2,3,7,8-TCDD ('3C-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)						
8			2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD ('3C-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)						
			OCDE (13C_OCDE)						

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.

12868 Ope 103 LDC #: SDG#:

### Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer:\_ 2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_b)/(A_x)(C_x)$ 

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

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

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

C<sub>s</sub> Where:

 $A_{\rm is}$  = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	₩	Q%
-	eed 21:35	(,, 05 2	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.00.1	1.002	P400P.0	10.	10-
		<u>}</u>	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.1166	1.166	1-166171.0063		6.0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.146	1.146	1.16495	١-	٩
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.086	1891	1.08079	0.0	5.0
			OCDE (13C-OCDD)	1.549	1-549	1.44167	6,9	200
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
		, , , ,	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)					
			OCDE (¹3C-OCDD)					
3			2,3,7,8-TCDF ('³C-2,3,7,8-TCDF)					
			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.

LDC # 240 87 B2, 16 50 21 SDG#:

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

2nd Reviewer. 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:

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

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

554IB-03-MS/MSD samples:

5BPC MS 10

	Š	ike	Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spike Duplicate	e Duplicate	Reported	Recalculated
Compound	Ad ( pg	Added	Concentration (あつ)	Concentration ( わタ 9	oncentration ( わタ 9	Percent Recovery	Recovery	Percent Recovery	Recovery	RPD	RPD
	O 1 SM	J'Û	0101	) f	MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	21.7	21.0	ΩN	7.81	18.7	36	A.	89	89	1.7	2.7
1,2,3,7,8-PeCDD	100	105	an an	1/2	96%	30146	sal	46	þЬ	7	7
1,2,3,4,7,8-HxCDD	7	^	<b>~</b>	201	ss. 1	47	179	8	8	<u>ş</u>	<u>σ</u>
1,2,3,4,7,8,9-HpCDF	1	1	7	71100+	pal	901	901	711	811	Le	1.
OCDF	212	012	29.0	83	<b>५</b> %	119	119	123	[123	23	23

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

SDG #: 24089,82/ SDG #: 44, Lond

# 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:

M Recovery = 100 \* SSC/SA Where: SSC =

Where: SSC = Spiked sample concentration SA = Spike added

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

LCS = Laboraotry control sample percent recovery

LCS ID: 022 8363 Le

LCSD = Laboratory control sample duplicate percent recovery

	ű	11.0	Spiked	amole	SOI	S.	ICSD	0	1 CS/1 CSD	csn
	À À	Added	Concentration	ration	Percent Recovery	ACOVB/V	Percent Recovery	scovery	RPD	٥
punaduna	83.	(S)	103 108	d I CSD	Renorted	Recalc	Reported	Racalc	Reported	Recalculated
2.3.7.8-TCDD	26.02	40	1.77	47	128	S <sub>X</sub>				
1.2.3.7.8-PeCDD	100.0		36.6		27	87				
1.2.3.4.7.8-HxCDD	100.0		93.6		plo	वैत				
1,2,3,4,7,8,9-HpCDF	100		78.0		19	79				
OCDF	agr	1	169	<del>- &gt;</del>	₽8	8₁	£2	\		
							\			
								·		
						٠		,		

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.

# lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

	$\Gamma$			
	Analyte	HPCDF HPCDF (S) HPCDD HPCDD HPCDD HPCDD HPCDD (S) NCDPE PFK	OCDF OCDF OCDD OCDD (S) OCDD (S) DCDPE PFK	
	Elemental Composition	C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO 13C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7ClO 13C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O 13C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O 13C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O 13C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>36</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O	C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> ClO C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> ClO C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> ClO <sub>2</sub> C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> ClO <sub>2</sub> (10C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> ClO <sub>2</sub> (10C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> <sup>26</sup> Cl, <sup>37</sup> Cl <sub>2</sub> O	
	Ol uol	M M M M M M M M M M M M M M M M M M M	M M H 2 A 4 A 4 A 4 A 4 A 4 A 4 A 4 A 4 A 4 A	
	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 471.7750 513.6775	
	Descriptor	4	ro 	-
	Analyte	TCDF TCDF (8) TCDF (8) TCDD TCDD TCDD (8) TCDD (8) HXCDPE PFK	Pecde Pecde Pecde (S) Pecdd Pecdd Pecdd (S) Pecdd (S) Pecdd (S)	HXCDF HXCDF (S) HXCDF (S) HXCDD HXCDD HXCDD HXCDD (S) HXCDD (S) OCDPE
Flamental Composition		C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'C10 'C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'C10 'C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'C C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'C 'C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'CO'C 'C <sub>12</sub> H <sub>4</sub> **Cl <sub>4</sub> O'CO'C	C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO <sup>10</sup> C <sub>1</sub> 2H <sub>3</sub> 3C <sub>1</sub> 3 <sup>2</sup> ClO C <sub>2</sub> F <sub>13</sub>	C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> 13C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> 13C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> 13C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> 3C <sub>13</sub> C <sub>10</sub>
Gl nol		W W W W W W W W W W W W W W W W W W W	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 COCK
Accurate mass <sup>(a)</sup>	303 8016	305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	333.8597 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 403.8529 445.7555 [430.9728]
Descriptor	-	-	N	Ø

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000  $^{19}C = 13.003355$ F = 18.9984

O = 15.994915  $^{3}CI = 34.968853$   $^{37}CI = 36.965903$ 

S = internal/recovery standard

LDC #:_	2	4089B	2/
		cover	

Sample Calculation Verification

Page:	/_of	/
Reviewer:	F	7
2nd reviewer:_	W	/

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

N N/A

Df

%S

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

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

Concentration = (A)(I,)(DF)
(A<sub>k</sub>)(RRF)(V<sub>o</sub>)(%S)

A<sub>k</sub> = Area of the characteristic ion (EICP) for the compound to be measured

A<sub>k</sub> = Area of the characteristic ion (EICP) for the specific internal standard

I<sub>s</sub> = Amount of internal standard added in nanograms (ng)

V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).

RRF = Relative Response Factor (average) from the initial calibration

Percent solids, applicable to soil and solid matrices

Dilution Factor.

Example:
Sample I.D. 4 . OCPD

 $\begin{array}{lll} \text{Conc.} = & (596.79) & (4000) & (\\ 265175.8) & (1.197) & (10.0) & (0.916) \\ = & 0.83 & pq | q \end{array}$ 

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

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 7, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H110575

### Sample Identification

SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-15BPC SSAI3-02-19BPC\*\* SSAI3-02-25BPC SSAI3-02-5BPC FD SSAI3-03-11BPC SSAI3-03-14BPC SSAI3-03-23BPC SSAI3-03-25BPC\*\* SSAI3-04-11BPC SSAI3-04-14BPC

SSAI3-04-14BPC FD SSAI3-04-15BPC SSAI3-04-23BPC

SSAI3-03-5BPC

SSAI3-04-8BPC

SSAI3-03-8BPCMS

SSAI3-03-8BPCMSD

SSAI3-03-5BPCMS

SSAI3-03-5BPCMSD

SSAI3-02-8BPC

SSAI3-02-5BPC

SSAI3-03-8BPC

SSAI3-04-25BPC SSAI3-04-5BPC

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

### Introduction

This data review covers 26 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

### I. Technical Holding Times

All technical holding time requirements were met.

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

### II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

### III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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
0225295-MB	8/13/10	1,2,3,4,6,7,8-HpCDD OCDD	0.17 pg/g 0.66 pg/g	SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-15BPC SSAI3-02-15BPC SSAI3-02-25BPC SSAI3-03-25BPC_FD SSAI3-03-11BPC SSAI3-03-14BPC SSAI3-03-23BPC SSAI3-03-25BPC** SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-14BPC SSAI3-04-15BPC SSAI3-04-25BPC SSAI3-04-25BPC SSAI3-04-25BPC SSAI3-03-8BPC SSAI3-03-8BPC SSAI3-03-5BPC
0228363-MB	8/16/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.096 pg/g 0.80 pg/g 0.064 pg/g 0.26 pg/g 0.087 pg/g 0.16 pg/g	SSAI3-04-5BPC SSAI3-04-8BPC

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

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAI3-02-11BPC	1,2,3,4,6,7,8-HpCDD	0.27 pg/g	0.27U pg/g
	OCDD	1.0 pg/g	1.0U pg/g
SSAl3-02-14BPC	1,2,3,4,6,7,8-HpCDD	0.61 pg/g	0.61U pg/g
SSAI3-02-15BPC	1,2,3,4,6,7,8-HpCDD	0.18 pg/g	0.18U pg/g
	OCDD	1.2 pg/g	1.2U pg/g
SSAI3-02-19BPC**	OCDD	2.0 pg/g	2.0U pg/g
SSAI3-02-25BPC	1,2,3,4,6,7,8-HpCDD	0.12 pg/g	0.12U pg/g
	OCDD	1.6 pg/g	1.6U pg/g
SSAI3-02-5BPC_FD	1,2,3,4,6,7,8-HpCDD	0016 pg/g	0016U pg/g
	OCDD	0.93 pg/g	0.93U pg/g

` Sample	Compound	Reported Concentration	Modified Final Concentration
SSAI3-03-11BPC	OCDD	0.55 pg/g	0.55U pg/g
SSAI3-03-23BPC	OCDD	1.5 pg/g	1.5U pg/g
SSAI3-03-25BPC**	1,2,3,4,6,7,8-HpCDD	0.10 pg/g	0.10U pg/g
	OCDD	1.0 pg/g	1.0U pg/g
SSAI3-04-11BPC	1,2,3,4,6,7,8-HpCDD	0.20 pg/g	0.20U pg/g
	OCDD	1.0 pg/g	1.0U pg/g
SSAI3-04-14BPC	1,2,3,4,6,7,8-HpCDD	0.22 pg/g	0.22U pg/g
	OCDD	1.7 pg/g	1.7U pg/g
SSAI3-04-14BPC_FD	OCDD	0.55 pg/g	0.55U pg/g
SSAI3-04-15BPC	1,2,3,4,6,7,8-HpCDD	0.20 pg/g	0.20U pg/g
	OCDD	1.9 pg/g	1.9U pg/g
SSAI3-04-23BPC	1,2,3,4,6,7,8-HpCDD	0.18 pg/g	0.18U pg/g
	OCDD	0.75 pg/g	0.75U pg/g
SSAI3-04-25BPC	1,2,3,4,6,7,8-HpCDD	0.086 pg/g	0.086U pg/g
	OCDD	0.75 pg/g	0.75U pg/g
SSAI3-02-5BPC	1,2,3,4,6,7,8-HpCDD	0.28 pg/g	0.28U pg/g
	OCDD	1.9 pg/g	1.9U pg/g
SSAI3-02-8BPC	1,2,3,4,6,7,8-HpCDD	0.36 pg/g	0.36U pg/g
	OCDD	1.6 pg/g	1.6U pg/g
SSAI3-03-5BPC	1,2,3,4,6,7,8-HpCDD	. 0.35 pg/g	0.35U pg/g
	OCDD	2.4 pg/g	2.4U pg/g
SSAI3-03-8BPC	1,2,3,4,6,7,8-HpCDD	0.16 pg/g	0.16U pg/g
	OCDD	0.81 pg/g	0.81U pg/g
SSAI3-04-5BPC	1,2,3,7,8,9-HxCDD	0.18 pg/g	0.18U pg/g
	1,2,3,4,6,7,8-HpCDD	0.17 pg/g	0.17U pg/g
	OCDD	1.2 pg/g	1.2U pg/g
	1,2,3,4,7,8-HxCDF	0.22 pg/g	0.22U pg/g
	1,2,3,4,6,7,8-HpCDF	0.27 pg/g	0.27U pg/g
	OCDF	0.62 pg/g	0.62U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAI3-04-8BPC	1,2,3,7,8,9-HxCDD	0.26 pg/g	0.26U pg/g
	1,2,3,4,6,7,8-HpCDD	0.20 pg/g	0.20U pg/g
	OCDD	2.4 pg/g	2.4U pg/g
	1,2,3,4,7,8-HxCDF	0.21 pg/g	0.21U pg/g
	1,2,3,4,6,7,8-HpCDF	0.47 pg/g	0.47U pg/g

Sample EB-08072010 (from SDG G0H110567) 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-08072010	8/7/10	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	7.6 pg/L 1.5 pg/L 2.0 pg/L 1.0 pg/L 3.6 pg/L 9.5 pg/L	All samples in SDG G0H110575

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

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAI3-02-19BPC**	<sup>13</sup> C-OCDD	35 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-03-23BPC	<sup>13</sup> C-OCDD.	21 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-03-25BPC**	<sup>13</sup> C-OCDD	39 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-04-14BPC_FD	<sup>13</sup> C-OCDD	25 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAI3-04-15BPC	<sup>18</sup> C-OCDD	38 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-04-23BPC	<sup>13</sup> C-OCDD	34 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-03-5BPC	<sup>13</sup> C-OCDD	38 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAI3-04-5BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	₽
SSAI3-04-8BPC	<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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAI3-02-15BPC SSAI3-02-19BPC** SSAI3-02-25BPC SSAI3-02-25BPC_FD SSAI3-03-23BPC SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-5BPC SSAI3-02-8BPC SSAI3-03-8BPC SSAI3-03-5BPC	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H110575	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H110575	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

### XII. System Performance

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

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

Samples SSAl3-02-5BPC\_FD and SSAl3-02-5BPC and samples SSAl3-04-14BPC and SSAl3-04-14BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentra	tion (pg/g)	RPD	Difference		
Compound	SSAI3-02-5BPC_FD	SSAI3-02-5BPC	(Limits)	(Limits)	Flags	A or P
1,2,3,6,7,8-HxCDD	0.12	2.7U	-	2.58 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	0.13	0.10	-	0.03 (≤2.7)	_	
1,2,3,4,6,7,8-HpCDD	2.7U	0.28	-	2.42 (≤2.7)	-	•
OCDD	0.16	1.9	-	1.74 (≤5.4)	-	-
2,3,7,8-TCDF	0.93	0.54U	-	0.39 (≤0.54)	-	-
1,2,3,7,8-PeCDF	0.055	2.7U	-	2.645 (≤2.7)	-	-
2,3,4,7,8-PeCDF	0.089	2.7U	-	2.611 (≤2.7)	-	-
1,2,3,4,7,8-HxCDF	2.7U	0.16	-	2.54 (≤2.7)	-	-
1,2,3,6,7,8-HxCDF	2.7U	0.092	-	2.608 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDF	0.30	0.29	-	0.01 (≤2.7)	-	•
1,2,3,4,7,8,9-HpCDF	0.15	0.097	-	0.053 (≤2.7)	-	-
OCDF	0.65	0.65	<del>-</del>	0 (≤5.4)	_	-

	Concentra	tion (pg/g)	DDD	Difference		
Compound	SSAI3-04-14BPC	SSAI3-04-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,6,7,8-HxCDD	0.16	0.25	-	0.09 (≤2.7)	-	-
1,2,3,7,8,9-HxCDD	0.17	0.14	-	0.03 (≤2.7)	-	-
1,2,3,4,6,7,8-HpCDD	0.22	2.5U	-	2.28 (≤2.5)	-	-
OCDD	1.7	0.55	••	1.15 (≤5.3)	-	-

	Concentration (pg/g)					
Compound	SSAI3-04-14BPC	SSAI3-04-14BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDF	0.086	0.50U	-	0.414 (≤0.50)	-	-
1,2,3,4,7,8-HxCDF	0.31	0.13		0.18 (≤2.7)	-	-
1,2,3,6,7,8-HxCDF	0.12	0.085	-	0.035 (≤2.7)	-	
1,2,3,7,8,9-HxCDF	0.075	2.5U	-	2.425 (≤2.5)	-	-
1,2,3,4,6,7,8-HpCDF	0.56	0.32	-	0.24 (≤2.7)	•	-
1,2,3,4,7,8,9-HpCDF	0.095	2.5U	-	2.405 (≤2.5)	•	-
OCDF	0.98	0.73	•	0.25 (≤5.3)	_	-

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H110575

SDG	Sample	Compound	Flag	A or P	Reason
G0H110575	SSAI3-02-19BPC** SSAI3-03-23BPC SSAI3-03-25BPC** SSAI3-04-14BPC_FD SSAI3-04-15BPC SSAI3-04-23BPC SSAI3-03-5BPC SSAI3-04-5BPC SSAI3-04-8BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H110575	SSAI3-02-15BPC SSAI3-02-19BPC** SSAI3-02-25BPC SSAI3-02-5BPC_FD SSAI3-03-23BPC SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-5BPC SSAI3-02-8BPC SSAI3-03-8BPC SSAI3-03-5BPC	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (2nd column confirmation) (o)
G0H110575	SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-15BPC SSAI3-02-19BPC** SSAI3-02-25BPC SSAI3-02-25BPC SSAI3-03-11BPC SSAI3-03-14BPC SSAI3-03-23BPC** SSAI3-03-25BPC** SSAI3-04-14BPC SSAI3-04-14BPC SSAI3-04-14BPC SSAI3-04-15BPC SSAI3-04-25BPC SSAI3-04-25BPC SSAI3-04-25BPC SSAI3-04-5BPC SSAI3-04-5BPC SSAI3-04-5BPC SSAI3-02-5BPC SSAI3-03-5BPC SSAI3-03-8BPC SSAI3-03-5BPC SSAI3-04-8BPC	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason
G0H110575	SSAI3-02-11BPC SSAI3-02-14BPC SSAI3-02-14BPC SSAI3-02-15BPC SSAI3-02-15BPC SSAI3-02-25BPC SSAI3-03-11BPC SSAI3-03-14BPC SSAI3-03-23BPC SSAI3-03-25BPC** SSAI3-04-11BPC SSAI3-04-14BPC SSAI3-04-14BPC SSAI3-04-25BPC SSAI3-04-25BPC SSAI3-04-5BPC SSAI3-02-5BPC SSAI3-03-5BPC SSAI3-03-5BPC SSAI3-03-5BPC SSAI3-03-5BPC SSAI3-04-8BPC SSAI3-04-8BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H110575

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H110575	SSAI3-02-11BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.27U pg/g 1.0U pg/g	А	ы
G0H110575	SSAI3-02-14BPC	1,2,3,4,6,7,8-HpCDD	0.61U pg/g	А	bl
G0H110575	SSAI3-02-15BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.18U pg/g 1.2U pg/g	. А	b!
G0H110575	SSAI3-02-19BPC**	OCDD	2.0U pg/g	А	bl
G0H110575	SSAI3-02-25BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.12U pg/g 1.6U pg/g	Α	bl
G0H110575	SSAI3-02-5BPC_FD	1,2,3,4,6,7,8-HpCDD OCDD	0016U pg/g 0.93U pg/g	А	bl
G0H110575	SSAI3-03-11BPC	OCDD	0.55U pg/g	А	bl
G0H110575	SSAI3-03-23BPC	OCDD	1.5U pg/g	А	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H110575	SSAI3-03-25BPC**	1,2,3,4,6,7,8-HpCDD OCDD	0.10U pg/g 1.0U pg/g	А	bl
G0H110575	SSAI3-04-11BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.20U pg/g 1.0U pg/g	Α	bl
G0H110575	SSAI3-04-14BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.22U pg/g 1.7U pg/g	А	bl
G0H110575	SSAI3-04-14BPC_FD	OCDD	0.55U pg/g	A	bl
G0H110575	SSAI3-04-15BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.20U pg/g 1.9U pg/g	A	bl
G0H110575	SSAI3-04-23BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.18U pg/g 0.75U pg/g	A	bi
G0H110575	SSAI3-04-25BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.086U pg/g 0.75U pg/g	A	bl
G0H110575	SSAI3-02-5BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.28U pg/g 1.9U pg/g	A	bi
G0H110575	SSAI3-02-8BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.36U pg/g 1.6U pg/g	A	bl
G0H110575	SSAI3-03-5BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.35U pg/g 2.4U pg/g	A	bl
G0H110575	SSAI3-03-8BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.16U pg/g 0.81U pg/g	А	bi
G0H110575	SSAI3-04-5BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.18U pg/g 0.17U pg/g 1.2U pg/g 0.22U pg/g 0.27U pg/g 0.62U pg/g	A	ы
G0H110575	SSAI3-04-8BPC	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF	0.26U pg/g 0.20U pg/g 2.4U pg/g 0.21U pg/g 0.47U pg/g	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0H110575

No Sample Data Qualified in this SDG

	Tronox Northgate Henderson	
LDC #: <u>24089C21</u>	_ VALIDATION COMPLETENESS WORKSHEET	Date: 10/13//0
SDG #: <u>G0H110575</u>	Stage 2B/4	Page: /of /
Laboratory: Test America		Reviewer:
		2nd Reviewer:
METHOD: HRGC/HRMS Diox	xins/Dibenzofurans (EPA SW 846 Method 8290)	
The samples listed below were validation findings worksheets	e reviewed for each of the following validation areas. Validation fi	ndings are noted in attached

	Validation Area		Comments
1.	Technical holding times	A	Sampling dates: 8/1/10
11.	HRGC/HRMS Instrument performance check	A	
111.	Initial calibration	Δ	
IV.	Routine calibration/ <del>IC∀*</del>	A	
V.	Blanks	لىبى	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	ies
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	لىبى	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	5WK	Not reviewed for Stage 2B validation.
XII.	System performance	Α	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A-	
XIV.	Field duplicates	SW	P= 6, 19 12, 13
XV.	Field blanks	SIV	EB= EB-08072010

Note:

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

ND = No compounds detected R = Rinsate

FB = Field blank

SDG # GOHII 056 / D = Duplicate TB = Trip blank EB = Equipment blank

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

17		SOIL						
	1	SSAI3-02-11BPC	11	SSAI3-04-11BPC	21	حر SSAI3-03-5BPC	31 <b>/</b>	0225295-MB
	2	SSAI3-02-14BPC	12	SSAI3-04-14BPC 0	22 7	SSAI3-04-8BPC ~	32 <b>2</b>	0228363-MB
	3	SSAI3-02-15BPC	13	SSAI3-04-14BPC_FD P	23	SSAI3-03-8BPCMS	33	
u	4	SSAI3-02-19BPC**	14	SSAI3-04-15BPC	24	SSAI3-03-8BPCMSD	34	
	5	SSAI3-02-25BPC	15	SSAI3-04-23BPC	25	SSAI3-08-5BPCMS	35	
	6	SSAI3-02-5BPC_FD 0	16	SSAI3-04-25BPC	26	SSAI3-06-5BPCMSD	36	
	7	SSAI3-03-11BPC	172	SSAI3-04-5BPC	27		37	
	8	SSAI3-03-14BPC	18	SSAI3-02-8BPC	28		38	
	9	SSAI3-03-23BPC	19	SSAI3-02-5BPC 12 /	29		39	
] ر	10	SSAI3-03-25BPC**	20	SSAI3-03-8BPC	30		40	

Notes:		
	 ··········	 

### LDC #: 21089C2 \ SDG #: u cones

### VALIDATION FINDINGS CHECKLIST

Page: / of 2
Reviewer: /7
2nd Reviewer: 0

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

Validation Area	Yes	No	NA	Findings/Comments
l. 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) $\leq$ 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	Taung.			
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		1.114		Age State St
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?	1			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC #: 24089 C2 \
SDG #: 100 comm

### **VALIDATION FINDINGS CHECKLIST**

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

VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	004104 exclusive	electrone ecolores	Manager Code	
IX: Internal standards				
Were internal standard recoveries within the 40-135% criteria?	M	V	ļ	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?				<u> </u>
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?			ļ	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard ≥ 2.5?				
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	<i>&gt;</i>			
Was an acceptable lock mass recorded and monitored?		and a second	Security Sec	
XI. Compound quantitation/CRQLs		Get Many		
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?		. 1		
XII. System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.		-		
XV. Field blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

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

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

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Blanks

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

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

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

N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Blank analysis date:  $8//8// \odot$ Was the method blank contaminated? 0/  $\mathsf{Blank}$  extraction date: 8//3Y N N/A

Associated samples:

124-81,014

\* FAPC

58/1 D.28/4 2 6 0.080\* 11 2 6.53 18.0 0.16\*/4 0.93/4 0.18/4 h/sl·a 5 0.12\*/4 1.0/2 6.20/4 1.9/4 I Sample Identification 2 0.SS\* 9 ú 0.18\*/4 0.15\*/2 0.81/11 M/4.1 0.22\*/ 20 7 0.25\* 12 n/02.0 2 2.4/4 10/0/ 10.01 7 2/28.0 0.10\*/4 Ź 1.04/1 175.0 0. w) w) SX MB 0.85 411.0 15220 Blank ID 0.66 Compound U Ø Conc. units:

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".

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Page:

2nd Reviewer:\_ Reviewer:\_

\* EMPC

Blanks

Pléase 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)

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? Blank extraction date: 8//arphiN N/A

Blank analysis date: 8/20/10

Associated samples:

22

Sample Identification 2.4\*/4 0.2/4/4 0.20\*/4 p/ Lh·o A 0.17\* /2 10.27/4 0.62 ly 7 2 0.18/4 1 0.12 4:1 0.48 9.37 0.43 5.0 X e o 3 o Se 20 4 10000 0.80 \$ 2600 0228363 0.087 \* \* Blank ID 0.12 0.56 0.16 Compound Щ 9 W Ø 8 Conc. units:

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# 24089C2

### 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?

Brank units: pg/L Associated sample units: pg/g Sampling date() 8 11 0

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

EB = EB -08072010 SDG# GOH 110567

Associated Samples: FB

(X5Z) // W

Sample Identification L ľý 75.7 0 300 グス Ŋ ろっそ Blank ID して米 EB 2.0\* \*5: 5 0 Compound Ø Ø J 士

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:

27680/2 LDC #:

## **VALIDATION FINDINGS WORKSHEET**

Internal Standards

Reviewer:\_\_\_\_\_

Page:

Mease 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%)	mit: 40-135%)	Qualifications
		ħ	${\mathcal I}$	35	( 40-135)	1/41/P (i) own 9,0
					( 1 )	
					( )	
		8	I	2/	( )	
					( )	
		0)	I	39	( )	
				-	( )	
		(3	7	<b>Έ</b>	( )	
					( )	
		<i>ļ</i> .,	₽	38	( )	
		15	I	24	( )	
					( )	
		282	$\leftarrow$	3%	( )	
					( )	
		74 17	H	77	( )	
					<u></u>	
П		77	H	26	·	<b>→</b>
					(	
					( )	
		Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
∢	<sup>13</sup> C-2.3.7.8-TCDF	DF		K. 13C-1.2.3.4-TCDD	QΩ	
В	13C-2.3.7.8-TCDD	dd			НхСПЛ	
d	<sup>13</sup> C-12378-PeCDE	PACDE		M		
þ	<sup>13</sup> C-1,2,3,7,8-PeCDD	PACDD		Z		
Щ	13C-1,2,3,6,7,8-HxCDE	3-HxCDF		С		
Щ	13C-1,2,3,6,7,8-HxCDD	8-HxCDD		В		
d	13C-1,2,3,4,6,7,8-HpCDE	, 8-HpCDF		a		
$\exists$	13C-1,2,3,4,6,7,8-HpCDD	, 8-НрСDD		2		
+	1 13C OCDD					

### LDC# 24089C2)

## **VALIDATION FINDINGS WORKSHEET**

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Reviewer: Page:

2nd Reviewer:

Internal Standards

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

Pease 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? N N/A

#	Date	Lab ID/Reference	Internal Standard	% Reco	% Recovery (Limit: 40-135%)	Qua	Qualifications
		26	Н	37	(40-135	Jan 9 am	G SM .
					, )		
		×	1-1	30	) (	(	MS
					)	)	
		25	H	8		)	CISM
					)	(	
						(	
		220	5	35		) (	acm
					<b>&gt;</b>	)	
					)	(	
		T. Harden			)	(	
Ī					)		
					)	(	
					)		
					)		The Appendix Control of the Control
Ī					)	)	
T					)	)	
T					)	)	
					)	(	
		Internal Standards	Check Standard Used		Recovery Standards	Ch	Check Standard Used
4	<sup>13</sup> C-2.3.7.8-TCDF	DF		K. 13C-1.2.	<sup>13</sup> C-1.2.3.4-TCDD		
4	13C-2,3,7,8-TCDD	ad			<sup>13</sup> C-1 2 3 7 8 9-HxCDD		
þ	<sup>13</sup> C-1,2,3,7,8-PeCDE	PECDF		M			
4	<sup>13</sup> C-1,2,3,7,8-PeCDD	PeCDD		Z			
Щ	13C-1,2,3,6,7,8-HxCDF	3-HxCDE		q			
Ч	13C-1,2,3,6,7,8-HxCDD	3-HxCDD		а			
þ	13C-1234678-HpCDE	, 8-HpCDF		q			
크	13C-1,2,3,4,6,7,8-HpCDD	, 8-НрСDD		В			
$\frac{1}{2}$	╣			Н			Andread Andread Angree of the Control of the Contro

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: /of/ Reviewer: FT 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 Co

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		土	no and column	3,4,5,6,9,11,	(o) d own
			confirmation cas	12, 12, 18, 20,	1
			ner Hermen 2	, , , , , , , , , , , , , , , , , , , ,	
			f f		

Comments: See sample calculation verification worksheet for recalculations

LDC#:24089C21

### VALIDATION FINDINGS WORKSHEET \_\_\_\_Field\_Duplicates

Page:/of	
Reviewer:_	FI
d 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?

\* EMPC

	Concentra	tion pg/g)	(pg/g)	(pg/g)	Qualifications
Compound	6	19	Difference	Limits	(Parent Only)
D	0.12	2.7U	2.58	≤2.7	
E	0.13	0.10 🛠	0.03	≤2.7	
F	2.7∪	0.28	2.42	≤2.7	
G	0.16 🗶	1.9	1.74	≤5.4	
Н	0.93	0.54U	0.39	≤0.54	
ı	0.055 🜟	2.7U	2.645	≤2.7	
J	0.089 💥	2.7U	2.611	≤2.7	
к	2.7U	0.16	2.54	≤2.7	
L	2.7U	0.092	2.608	≤2.7	
0	0.30 🛠	0.29 🔻	0.01	≤2.7	
Р	0.15 *	0.097	0.053	≤2.7	. ,
Q	0.65 *	0.65	0	≤5.4	

LDC#: 24089C21

### VALIDATION FINDINGS WORKSHEET \_\_\_\_\_ Field Duplicates

Page: 2 of 2
Reviewer: F7
2nd Reviewer:



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

\* FMPC

	Concentra	tion pg/g)	(pg/g)	(pg/g)	Qualifications
Compound	12	13	Difference	Limits	(Parent Only)
D	0.16 🔺	0.25 💥	0.09	≤2.7	
E	0.17 🔏	0.14 🛠	0.03	≤2.7	
F	0.22 🔺	2.5U	2.28	≤2.5	
G .	1.7	0.55	1.15	≤5.3	
Н	0.086 🛠	0.50U	0.414	≤0.50	
к	0.31	0.13	0.18	≤2.7	
L	0.12	0.085	0.035	≤2.7	
N	0.075 💥	2.5U	2.425	≤2.5	
0	0.56	0.32 ⊁	0.24	≤2.7	
Р	0.095	2.5U	2.405	≤2.5	
Q	0.98	0.73 🛠	0.25	≤5.3	

V:\FIELD DUPLICATES\24089C21.wpd

LDC# 24089C2/ SDG#:

### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer. 2nd Reviewer:

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

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

calculations:

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

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

RRF =  $(A_{\nu})(C_{l_0})/(A_{l_0})(C_{\nu})$  average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (R12F3std)	RRF ( <i>pq2F                                    </i>	%RSD	%RSD
-	1421	0/12/1	2.3.7.8-TCDF ( <sup>13</sup> C-2.3.7.8-TCDF)	0.875	218.0	0.87	18.0	7.61	7.61
			2.3.7.8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.957	0.757	0.93	0.73	13.5	13.5
		<b>.</b>	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.107	1.107	61:1	1.13	12-5	12.5
		T	1.2.3.4.6.7.8-HpCDD (13C-1,2.4.6.7.8HpCDD)	1.026	1.026	1.07	1.07	13.6	/3.6
		T.	OCDE (13C.OCDD)	1.445	5 14.1	<u>/۰۶۰</u>	1.55	1.81	/-8/
2	1851	01/11/8	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.00/87	18100-1	7.80395	1.00395	4.19165	561.4
		`	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.16617	1-16617	1.15763	£7651.1	8.10393	401.8 ¢
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.14627	1.14627	761911	76/91.1	570 6 .8	708-8 6
		1	1.2.3.4.6.7.8-HpCDD (13C-1,2.4.6.7.8HpCDD)	1.08646	7,0801	1.44726	7.14h.1	74210.7	110.7 4
			OCDE (13C-OCDD)	Usyps!	C8 25.1	1.58 3/3	1-583/3	3.1203	2.8%
ო			2,3,7,8-TCDF (¹°C-2,3,7,8-TCDF)						
			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)						
			OCD# 430 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.

SDG# 24089(2)

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_y)(C_g)/(A_s)(C_y)$ 

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,

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
		Calibration		Average RRF	RRF	RRF		à
#	Standard ID	Date	Compound (Reference Internal Standard)	(initial)	(cc)	(၁၁)	%D	<b>0</b> %
_	eev-ss	01/81/8	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	518.0	84.0	0.78	10.6	10.6
	24:6		2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)	156.0	16.0	16.0	4.7	4.7
	•		1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.107	60.1	1.09	<i>\range 8-1</i>	8./
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1.026	1.0 کر	1.05	2.6	2.6
Н			OCDE (13C-OCDD)	544-1	1.69	(.69	17.3	17.3
7	cev-27	01/01/8	8/20//0 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.00187	78100-1	1/8/1	e 18	8.3
	11:110		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.16617	119914	1.16555	/ '0	7-0
H			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.14627	1. 4.14.27	1.133/6	1.7	1.1
H			1,2,3,4,6,7,8-HpCDD (¹³C-1,2,4,6,7,8,-HpCDD)	1.08646	1 1-08646	1.03340	9-8	2-6
			OCDE (13C-OCDD)	US4857	C+8/15-1/	1-39611	8-6	8-6
8			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)		16816:0A			
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		1.16553			
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)		1.13316			
┢			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)		07850.1			
		<b>T</b>	OCDF (¹3C-OCDD)		119651			

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.

24089C2/ te cons LDC#: SDG #:

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

2nd Reviewer.\_ Page:\_ 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:

トンプ

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

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

*ц* MS/MSD samples:

	ďS		Sample	Spiked Sample	Sample	Matrix Spike	Spike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Compound	Adi ( 129	Added ( 129./9.)	Concentration (Pg./9)		tration /9)	Percent Recovery	Recovery	Percent Recovery	Recovery	RPD	RPD
	O /	SD	٠٥,٥	2	MSD	Reported	Recalc.	Renorted	Recalc		*
2,3,7,8-TCDD	20.4	21.1	dN	17.9	2.61	88	8,8	76	76	2.8	8.3
1,2,3,7,8-PeCDD	102	106	VD	2//6	p.56	90	90	90	06	5%	5%
1,2,3,4,7,8-HxCDD	707	101	94	601	þΙΙ	101	101	801	801	2.6	4.7
1,2,3,4,7,8,9-HpCDF	94.3	94.3	6.14	94.3	8.16	76	26	68	2	0	0
	102	712	0.82	861	200	16	16	þЬ	16	99.0	0.66

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.

2408962 SDG #:\_\_ LDC#:

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

ŏ 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

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

LCS = Laboraotry control sample percent recovery

0225295 16> LCS ID:

LCSD = Laboratory control sample duplicate percent recovery

	8	ike	Spelked	amula	801	· ·	ICSD	C	US3/ICSD	CSD
Compound	<u> </u>	Added ( PQ / Q)	Concentration	tration	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	۵
	108	LCSD	o / sul	O	Reported	Recalc	Reported	Recalc	Reported	Recalculated
	7.0°C	NA	6.71	NA	06	90				
	20)		705	-	501	501				
	09/		43.4		86	93				
1,2,3,4,7,8,9-HpCDF	001		35.8		26	76				
	200	1	184	<b>→</b>	76	92	NA			
							\		:	

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

# lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Analyte	HPCDF HPCDF HPCDF HPCDD HPCDD HPCDD (S) HPCDD (S) NCDPE PFK	OCDF OCDF OCDD OCDD (S) OCDD (S) OCDP E PFK	
Elemental Composition	C <sub>12</sub> H <sup>36</sup> Cl <sub>1</sub> 37ClO C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> 37Cl <sub>2</sub> O 13 C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O 13 C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7ClO C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7ClO <sub>2</sub> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> 13 C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> 13 C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O <sub>2</sub>	C <sub>1</sub> .3°Cl,3°ClO C <sub>1</sub> .3°Cl,3°ClO C <sub>1</sub> .3°Cl,3°ClO <sub>2</sub> C <sub>1</sub> .3°Cl,3°Cl <sub>2</sub> O <sub>2</sub> 13°C <sub>1</sub> .3°Cl,0°ClO <sub>2</sub> 13°C <sub>1</sub> .3°Cl <sub>2</sub> O C <sub>1</sub> .3°Cl <sub>3</sub> .3°Cl <sub>2</sub> O	
Ol uol	M M M M M M M M M M M M M M M M M M M	M+2 M+4 M+2 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 471.7750 513.6775	
Descriptor	4	ιο 	
Analyte	TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HXCDPE	Pecdf Pecdf Pecdf (S) Pecdd (S) Pecdd (S) Pecdd (S) Pecdd (S) Pecdd (S)	HXCDF HXCDF HXCDF (S) HXCDD HXCDD HXCDD HXCDD (S) HXCDD (S) PFK
Elemental Composition	C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>4</sub> O C <sub>12</sub> H <sub>4</sub> <sup>3</sup> Cl <sub>4</sub> O 13C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>4</sub> O 13C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>4</sub> O C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> O C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> O 13C <sub>12</sub> H <sub>3</sub>	C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> ,7ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>3</sup> ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>3</sup> ClO <sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>3</sup> ClO C <sub>1</sub> F <sub>1</sub>	C <sub>12</sub> H <sub>2</sub> *C <sub>13</sub> *C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> *C <sub>13</sub> *C <sub>10</sub> G <sub>12</sub> H <sub>2</sub> *C <sub>11</sub> *C <sub>10</sub> G <sub>12</sub> H <sub>2</sub> *C <sub>11</sub> *C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> *C <sub>11</sub> *C <sub>10</sub> C <sub>12</sub> H <sub>2</sub> *C <sub>11</sub> *C <sub>10</sub> G <sub>12</sub> H <sub>2</sub> *C <sub>11</sub> *C <sub>10</sub>
Ol nol	W W W W W W W W W W W W W W W W W W W	M+2 M+4 M+2 M+2 M+2 M+2 M+2 M+2 M+3	M M M M M 4 2 2 4 2 4 2 4 2 4 4 4 4 4 4
Accurate mass <sup>(a)</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 403.8529 445.7555
Descriptor	-	N	ო

The following nuclidic masses were used: Ø

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

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

S = internal/recovery standard

LDC #:	24	1089Ca)	ļ
		cover	

### **VALIDATION FINDINGS WORKSHEET**

### Sample Calculation Verification

Page:	of/
Reviewer:_	F7
2nd reviewer:	V

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

	Y	N(	N/A
_	Y/	N	N/A

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

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

Sample I.D. # 4 OCDD:
Conc. = $(/543/3)$ , $(400)$ ) (3/535/00) $(1.09)$ $(10.18)$ $(0.9/2)$
= 1.956 pg/g

		Compound	С	Reported oncentration	Calculated Concentration ( )	Qualification
#	Sample ID	Compound				
,						

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

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 10, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H120594

### Sample Identification

SSAJ2-06-3BPC

SSAJ2-06-5BPC

SSAJ3-05-12BPC

SSAJ3-05-16BPC

SSAJ3-05-1BPC

SSAJ3-05-5BPC

SSAJ3-05-8BPC

SSAJ3-07-12BPC

SSAJ3-07-17BPC

SSAJ3-07-1BPC

SSAJ3-07-5BPC

SSAJ3-07-8BPC\*\*

EB-08102010

SSAJ3-05-12BPCMS

SSAJ3-05-12BPCMSD

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

### Introduction

This data review covers 14 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

- J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.
- J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.
- 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 and greater than or equal to 10 for each recovery and internal standard compound for samples on which Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

### V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0228254-MB	8/16/10	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	2.5 pg/L 2.0 pg/L 2.6 pg/L 7.7 pg/L 2.5 pg/L 1.9 pg/L 1.5 pg/L 2.6 pg/L 2.7 pg/L 2.7 pg/L 3.7 pg/L	All water samples in SDG G0H120594
0228379-MB	8/16/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.55 pg/g 0.061 pg/g 0.083 pg/g 0.045 pg/g 0.073 pg/g 0.14 pg/g 0.086 pg/g 0.31 pg/g	All soil samples in SDG G0H120594

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-08102010	OCDD	4.4 pg/L	4.4U pg/L
	1,2,3,7,8-PeCDF	5.2 pg/L	5.2U pg/L
	1,2,3,6,7,8-HxCDF	8.4 pg/L	8.4U pg/L
SSAJ3-05-16BPC	1,2,3,4,6,7,8-HpCDD	0.42 pg/g	0.42U pg/g
	OCDD	1.3 pg/g	1.3U pg/g
	2,3,4,6,7,8-HxCDF	0.20 pg/g	0.20U pg/g
	1,2,3,7,8,9-HxCDF	0.17 pg/g	0.17U pg/g
SSAJ3-07-17BPC	2,3,4,6,7,8-HxCDF	0.22 pg/g	0.22U pg/g
	1,2,3,7,8,9-HxCDF	0.30 pg/g	0.30U pg/g

Sample EB-08102010 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-08102010	8/10/10	OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	4.4 pg/L 7.8 pg/L 5.2 pg/L 12 pg/L 8.4 pg/L 19 pg/L 11 pg/L 55 pg/L	All soil samples in SDG G0H120594

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 percent recovery (%R) was not within QC limits for one compound, the LCS percent recovery (%R) was within QC limits and no data were qualified.

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAJ2-06-5BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	23 (40-135) 15 (40-135) 22 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-05-12BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 21 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

Sample	internal Standards	%R (Limits)	Compound	Flag	A or P
SSAJ3-05-16BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 16 (40-135) 25 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-05-1BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 19 (40-135) 37 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-05-5BPC			J (all detects) UJ (all non-detects)	P	
SSAJ3-05-8BPC	<sup>13</sup> C-OCDD	33 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ3-07-12BPC	<sup>13</sup> C-OCDD	24 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ3-07-17BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	23 (40-135) 12 (40-135) 20 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAJ3-07-1BPC	<sup>13</sup> C-OCDD	29 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ3-07-5BPC	18C-OCDD	22 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ3-07-8BPC**	18C-OCDD	23 (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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAJ3-07-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P
SSAJ3-05-5BPC	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	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 G0H120594	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H120594	All compounds reported by the lab 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H120594

		1			
SDG	Sample	Compound	Flag	A or P	Reason
G0H120594	SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-16BPC SSAJ3-05-1BPC SSAJ3-05-5BPC SSAJ3-07-17BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H120594	SSAJ3-05-8BPC SSAJ3-07-12BPC SSAJ3-07-1BPC SSAJ3-07-5BPC SSAJ3-07-8BPC**	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H120594	SSAJ3-07-1BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H120594	SSAJ3-05-5BPC	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H120594	SSAJ2-06-3BPC SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-16BPC SSAJ3-05-5BPC SSAJ3-05-8BPC SSAJ3-07-12BPC SSAJ3-07-17BPC SSAJ3-07-1BPC SSAJ3-07-5BPC SSAJ3-07-5BPC SSAJ3-07-8BPC** EB-08102010	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0H120594	SSAJ2-06-3BPC SSAJ2-06-5BPC SSAJ3-05-12BPC SSAJ3-05-16BPC SSAJ3-05-15BPC SSAJ3-05-5BPC SSAJ3-07-12BPC SSAJ3-07-17BPC SSAJ3-07-17BPC SSAJ3-07-15BPC SSAJ3-07-5BPC SSAJ3-07-8BPC** EB-08102010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	Α	Project Quantitation Limit (k)

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H120594

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H120594	EB-08102010	OCDD 1,2,3,7,8-PeCDF 1,2,3,6,7,8-HxCDF	4.4U pg/L 5.2U pg/L 8.4U pg/L	A	bl
G0H120594	SSAJ3-05-16BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.42U pg/g 1.3U pg/g 0.20U pg/g 0.17U pg/g	A	bl
G0H120594	SSAJ3-07-17BPC	2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.22U pg/g 0.30U pg/g	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H120594

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson**

LDC #: 24089DC21	VALIDATION COMPLETENESS WORKSHEET	
SDG #: G0H120594	Stage 2B/4	F
Laboratory: Test America		Rev
		2nd Rev

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	Δ	Sampling dates: 8/10/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	A	
IV.	Routine calibration <del>4€V</del>	Δ	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	sw	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB = 13

Note:

A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate FB = Field blank D = Duplicate

TB = Trip blank EB = Equipment blank

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

	SOIL +	اسر	ater				
1	SSAJ2-06-3BPC	11	SSAJ3-07-5BPC	<del>†</del> 21	0228379-MB	31	
2	SSAJ2-06-5BPC	12	SSAJ3-07-8BPC**	† 22	0228291-MB	32	
3	SSAJ3-05-12BPC	13	EB-08102010 V	23		33	
4	SSAJ3-05-16BPC,	14	SSAJ3-05-12BPCMS	24		34	
5	SSAJ3-05-1BPC	15	SSAJ3-05-12BPCMSD	25		35	
6	SSAJ3-05-5BPC~	16		26		36	
7	SSAJ3-05-8BPC	17		27		37	
8	SSAJ3-07-12BPC -	18		28		38	
9	SSAJ3-07-17BPC -	19		29		39	
10	SSAJ3-07-1BPC	20		30		40	

Notes:		

### LDC#: 24089pc2 | SDG#: \_\_\_\_\_ cone

### VALIDATION FINDINGS CHECKLIST

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.		985 st 1225	42,500	
III. 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?		<u> </u>		
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?			9/10/10/99	
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	1	ı	1	7
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		ı	i —	
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: 7
2nd Reviewer: 9

		Ari sele			
VIII. Regional Quality Assurance and Quality Control		<u> </u>	<u> </u>		
Were performance evaluation (PE) samples performed?		ļ	_	<del> </del>	
Were the performance evaluation (PE) samples within the acceptance limits?		<u> </u>		Ł	Producen v dipolitica de la companya
IX: Internal/standards					
Were internal standard recoveries within the 40-135% criteria?					
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	/	100 00 7030 0			
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?			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 $\pm$ 2 seconds (includes labeled standards)?	_	-			
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	_				
Was an acceptable lock mass recorded and monitored?					
XI. Compound quantitation/CRQLs					
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?					
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?					
XII. System performance					
System performance was found to be acceptable.					
XIII. Overall assessment of data					7 (* 12. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13. – 13.
Overall assessment of data was found to be acceptable.					
XIV. Field duplicates					10 de 20
Field duplicate pairs were identified in this SDG.					
Target compounds were detected in the field duplicates.			_	-	
XV. Field blanks	L	51			
Field blanks were identified in this SDG.		Annah Annah San			and the state of t
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

lotes:

LDC# 24089DC2)

## VALIDATION FINDINGS WORKSHEET

Blanks

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

> Pease 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)

Were all samples associated with a method blank?

Y N N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Was the method blank contaminated? 

Blank analysis date: \$\10\10 Blank extraction date: \_ 용니レ

all waler (bl) EMPC \* Associated samples:

Conc. units: pa L.		·			чения при	\
Compound	Blank ID	××		Sample Identification	ítion	
	0228254 MB	-MB	13			
D	2.5	12.5				
נע)	2.0 *	0.01			,	
ΙL	7.6*					
G	7.7	38.5	4.4+/4			
I	2,5	13.5	b/z.s			
<b>Y</b>	¥ b:1	9.6				
Γ	1.9		8.4/4			
W	1.5 *	7.5	1			
2	2.6	13	1			
θ	2.7 *	13.5	1			
م	2.0	01	1			
8	3.7*	18.5	\			
	·					
:						

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".

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### VALIDATION FINDINGS WORKSHEET Blanks

Page: 2nd Reviewer.\_ Reviewer:

7

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

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

Were all samples associated with a method blank? M/A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Was the method blank contaminated? Blank extraction date: ४ N/N N/A

Conc. units: pa

Blank analysis date: 8/2/ 2/2/

Associated samples:

EMPC

\*

All 801/2 ( bd)

Compound	Blank ID	×.5			Sai	Sample Identification	ion		
	0228379-MB	-MB	7	Ь					
1	P1.0	1.0	h/zh.0						
6	0.55 A	2.15	1.344						
٧٢	0.061	0.305	t						
	0.083	0.415	1						
M	0.045*	0.22	0.20/y	n/*22.0					
Z	0.073	0.365	0.17/W	10.30/u					
Ø	6.14	1.0	. (	-					
٥	0.086	0.43							
প্র	16.0	1.55	ı						

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# 24089DC2/

## VALIDATION FINDINGS WORKSHEET FIELD Blanks

FMPC

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

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

Blank units: Paragociated sample units: Paragociated

Sampling date: 0 2 10 10 Sampling date: 0 Circle one) Field Blank / Rinsate / Other.

All 80:15 (25x)

Field blank type: (circle one) Field Blank / Rinsate / Other:	Field Blank / I	ک Associated Samples:	amples:	<   \sqrt{\sq}\}}\sqrt{\sq}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}}	(XSK) <	(	
Compound	Blank ID		Sample Identification	ntification			
	[3						
9	4.4*						
H	*8.1						
H	2.5						
K	71						
1	४.५						
φ	61						
٩							
&	55						

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

14089DC21 LDC #:\_

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

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

MS/MSD. Soil / Water.

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

	5.			T							Ī												
ations	nison De																						
Qualifications	Jan 6	0																					
	3																						
Associated Samples	8																						
)	(	Î ^	)	`	)	(	(	ĺ		)	)	)	)	(	)	).	(	(	(	(	(	^	
RPD (Limits)	)		)	)	)	)	)	)	)	)	)	)	)	)		)	)	<u> </u>	)	)	)	)	
	1	_	^	(	(	-	<u> </u>	_	,	^	(	^	^	_	^	(	(	^	^	^	^	^	)
MSD %R (Limits)	11-5-1	-	_	J	)	<u> </u>	<b>-</b>	_	_	J	_	_	_	_	_		_	_	<u> </u>	_		_	J
%	150																						
		1	_	^	( )	^	^	ľ		^	ſ^	^		^	_		^	^	^	^	^		
MS %R (Limits)	_	-	_	_	_	_	_	,		_	_	_	_	_	_	_	    	_	_	_	_	_	
*																							
Compound	8	1																					
OI OSW/SW	) I P																						
Date	200																						
#																							

LDC# 240x9 DC2

## VALIDATION FINDINGS WORKSHEET

Page: /of\_

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  $\geq$  10?

N/N/A Ϋ́

*	Date	Lab ID/Reference	Internal Standard	% Recovery	% Recovery (Limit: 40-135%)	Qualifications	ations
		2	H	43	( fo-135 )	JMJP (i	OWAL F
			I	15	(   )		୍ ଜ, ହ
			6	72	( )		9,49
					( )		
		3	Н	35	(		
			1	21	)		
			b b	34	( )		
					( )		
		Ъ	H	28	( )		
			J	9/	( )		
П			6	メ	(		
					(		
		5	#	39	(		
			Ĭ	61	( )		-
			9	37	(		
					( )		
		9	Ħ	39	)		
			-	8/	( )		
T			\$	37		Þ	
					( )		
		Internal Standards	Check Standard Used		Recovery Standards	Check	Check Standard Used
A	<sup>13</sup> C-2.3.7.8-TCDF	,DF		K. 13C-1.2.3.4-TCDD	TCDD		
В	13C-2,3.7,8-TCDD	OD		1 13C-1237	<sup>13</sup> C-1 2 3 7 8 9-HxCDD		
d	13C-12,3,7,8-PeCDF	PACDE		M			
d	<sup>13</sup> C-12378-PeCDD	Pecdo		Z			
Ц	13C-1,2,3,6,7,8-HxCDE	3-HxCDE		q			
Щ	13C-1,2,3,6,7,8-HxCDD	3-HxCDD	approach to debate	Д.			
d	13C-1,2,3,4,6,7,8-HpCDE	7,8-HpCDE		d			
뒥	13C-1,2,3,4,6,7,8-HpCDD	7,8-HpCDD		В			
+	13C OCDD						

LDC # 24089 DC2 /

## **VALIDATION FINDINGS WORKSHEET**

Page: 2 of 3

Internal Standards

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

Y N/A/A

Are all internal standard recoveries were within the 40-135% criteria?

Y N/A/A

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

Date Lab	Lab	Lab ID/Reference	internal Standard	% "	covery (Limit: 40-135%)		ations
			H	"	33 (40-13	( 56/	M3// (C) CUMICGO
		—					
<i>⊗</i>	3		<b>T</b>	6	٥, / ١		À
e A	1		#	23	) 6	(	(C) gual F
			I	12	7	(	( d, Q
			6	2	) Οι	(	9,9
					•	(	
<i>a</i> )	Q)		Н	0	39 (	Î	(F) gual G, B
						(	•
			I		77		
G -	C -		F		4		
			-				*
					)	) (	
						) (	
					)	) [	
Internal Standards	Internal Standards		Check Standard Used		Recovery Standards	rds	Check Standard Used
<sup>13</sup> C-2.3,7,8-TCDF	DF			X,	<sup>13</sup> C-1.2.3.4-TCDD		
<sup>13</sup> C-2 3 7 8-TCDD	ממנ			- 1	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		
<sup>13</sup> C-12378-PeCDF	PECDF			Ø			
<sup>13</sup> C-1 2 3 7 8-PeCDD	Pecdo	- 1		Z			
13C-123678-HxCDF	3-HxCDF			q			
<sup>13</sup> C-1,2,3,6,7,8-HxCDD	3-HxCDD			В			
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	7,8-HpCDE			q			
<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD	7,8-НрСDD			В			
				=			_

LDC # 246890C2/

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

Page: 3of 3 Reviewer: Znd Reviewer: Z

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". メ んん/A N/A N/A

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	Was the S/N ratio all internal standard peaks > 10?
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Qualifications	no gue MS	0 1			Ш	USM Law p on	7														Check Standard Used									
% Recovery (Limit: 40-135%)	5 ( 40-135 )	( 1 ) 31	) hE	( )	· ·	( ) 22	( <b>→</b> ) 68	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	Recovery Standards	<sup>13</sup> C-1.2.3.4-TCDD	<sup>13</sup> C-1,2,3,7,8,9-HxCDD		- Carrier - Carr					
Internal Standard % R						T											,				Check Standard Used	¥	) <sub>E1</sub> 1	M	Z	O	а	а	В	
Lab ID/Reference						51															Internal Standards	)F	dc dc	aCDF	еСDD	HXCDF	HXCDD	8-HnCDF	8-HnCDD	
# Date																						A. 13C-2.3.7.8-TCDF	_		H					H

### LDC# 24089DC2/

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

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

Y N/A Wer

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).

17		· ·			
	Date	Sample ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
					,
			All compounds reported as EMPC	Ali	JK detects (k)
		HIKLOPO	x lol cal Range	01	1/paet (e)
		9,8	7	9	)   pax (c)

Comments: See sample calculation verification worksheet for recalculations

LDC# 24087 DC 2/ SDG# ALL COURT

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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

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

RRF =  $(A_{\nu})(C_{s_{\nu}})/(A_{l_{v}})(C_{\chi})$  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<sub>s</sub> = Area of associated internal standard C<sub>s</sub> = Concentration of internal standard S, X = Mean of the RRFs

Recalculated 3.6K o o %RSD 3.37 Reported %RSD るるか Ø. 7.0 3. 7.5 RRF (*RPF3*std) Recalculated 1.02 .35 9:10 23.0 0.97 .0. RRF (*PRF* 3 std) Reported 101 0.97 . 3 S 07: 0.98 Ó Average RRF (initial) Recalculated 0.995 0.983 1.056 1.163 1.073 1.370 Average RRF (initial) 1.050. Reported 1.370 0.983 163 566.0 .072 Compound (Reference Internal Standard) 1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD) 1,2,3,4.8,7,8-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-HxCDB (13C-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-TCDD ("C-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) 2,3,7,8-TCDF (13C-2,3,7,8-TCDF) OCDE (13C\_OCDD) OCDE (3C OCDD) OCDF (13C-OCDD) 01/92/L Calibration Date Standard ID 60100 PBris 1047 45 #

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

LDC#: 24089 DCV, SDG#: 44 cons

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_x)/(A_x)(C_x)$ 

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

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

 $A_{\rm ls}$  = Area of associated internal standard  $C_{\rm ls}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	<b>Q</b> %
-	2-120	01/27/8	8/23/10 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	566.0	96.0	26.0	3.0	3.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.383	46.0	160	4.9	4.9
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.163	1.17	1.17	0.7	0.7
1			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	220.1	1.04	1.04	7.6	2.6
			OCDF (13C-OCDD)	1.370	1.42	1.42	3.4	3.4
7	cev-2	01/17/8	8/21/10 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)		66.0	66.0	6.0	6.0
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)		16.0	16.0	1.	/ /
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)		61.1	(1.13	2.6	2-6
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)		1.06	1.06	6.0	6.0
٦İ			CODE (30-00D)	Å	76.1	95.1	6,3	6.3
3	261-2	01/62/8	8/29/10 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.056	1.05	1.05	6.0	8.0
	DBINS		2,3,7,8 TODD (*C-2,3,7,8-TØDD)					
			1,2,3,6,7,8-HxCDD (3-7-1,2,3,6,7,8-HxCDD)					
寸			1,2,3,4,6,Z,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCBF ("C-OCDD)					

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. Comments:

LDC # 24089 DC2/ et cons SDG #:

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

2nd Reviewer: Reviewer:

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

using the following calculation:

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

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

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

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

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

괴 MS/MSD samples: \_

		1	1	1	T	<u> </u>	Г	T	T	T	T	T	T
Recalculated	RPD		4.3	3.)	2.6	2.5	7.7						
Reported	RPD		7.3	3.	2	الم	1.7						
Matrix Spike Duplicate	Percent Recovery	Pocalc	٥	107	801	136	asl						
Matrix Spik		å		701	801	130	æ31						
Matrix Spike	Percent Recovery	Recalc	301	bal	116	139	871						
Matrix		Renorted	801	601	116	139	971					·	
Sample	Concentration ( DS Q	Jid Msn	T.41	113	114	200	719						
Spiked Sample		) I	23.3	117	124	Son	569						
Sample	Concentration (ロス) め	0 101	0.53	7.	1.4	52	300						
Spike	(g)	MSD	6.02	101	->	<b>~</b>	bor						
	Adde (	) I MS	1.12	105	7	4	300				-		
	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 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#: 240891 DC2/ SDG#: 24 couch

# 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: SSC = Spiked sample concentration SA = Spike added

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 022 8379 - 10/2

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

Recalculated I CS/I CSD RPD Reported Recalc Percent Recovery CSD 45 <u>0</u> 00 <u>=</u> Percent Recovery 5 113 00 <u>و</u> = 5 00 CSD **∢** 2 Spiked Sample Concentration <u>g</u> 2-1-6 109 113 23 CSD なな Spike Added 0.002 0.001 00.0 100.0 J. O. Compound 1,2,3,4,7,8,9-HpCDF 1,2,3,4,7,8-HxCDD 1,2,3,7,8-PeCDD 2,3,7,8-TCDD 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 ecalculated results.

# Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

E P			
Analyte	HPCDF HPCDF HPCDF HPCDD HPCDD HPCDD HPCDD (S) HPCDD (S)	000F 000F 000D 000D 000D (S) 000D (S) PFK	
Elemental Composition	C <sub>12</sub> H*C <sub>13</sub> ,7ClO C <sub>12</sub> H*C <sub>13</sub> ,7ClO '3C <sub>12</sub> H*C <sub>13</sub> ,0ClO '3C <sub>12</sub> H*C <sub>13</sub> ,7ClO '3C <sub>12</sub> H*C <sub>13</sub> ,7ClO C <sub>12</sub> H*C <sub>13</sub> ,7ClO '3C <sub>12</sub> H*C <sub>13</sub> ,7ClO '3C <sub>12</sub> H*C <sub>13</sub> ,7ClO C <sub>12</sub> H*C <sub>13</sub> ,7ClO C <sub>12</sub> H*C <sub>13</sub> ,7ClO	G12 wG1,37ClO G12 wG1,87ClO G12 wG1,37ClO G12 wG1,37ClO 19C12 wG1,37ClO 19C12 wG1,37ClO C12 wG1,37ClO C12 wG1,37ClO	
Ol nol	M M M H + 2	M+4 M+4 M+2 M+4 M+4 LOCK	
Accurate Mass <sup>(s)</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 471.7750 513.6775	
Descriptor	4	ഗ 	-
Analyte	TCDF TCDF (8) TCDP (8) TCDD (9) TCDD (8) TCDD (8) TCDD (8) HXCDPE	Pecdr Pecdr Pecdr (S) Pecdd Pecdd Pecdd (S) Pecdd (S) Pecdd (S)	HXCDF HXCDF HXCDF (3) HXCDD HXCDD HXCDD (5) HXCDD (5) OCDPE
Elemental Composition	C <sub>12</sub> H, <sup>35</sup> Cl,O C <sub>12</sub> H, <sup>35</sup> Cl,O 13C,2H, <sup>35</sup> Cl,O 13C,2H, <sup>35</sup> Cl,O C <sub>12</sub> H, <sup>35</sup> Cl,O <sub>2</sub> C <sub>12</sub> H, <sup>35</sup> Cl,O <sub>2</sub> 13C,2H, <sup>35</sup> Cl,O <sub>2</sub> 13C,2H, <sup>35</sup> Cl,O <sub>2</sub> 13C,2H, <sup>35</sup> Cl,O <sub>2</sub> C <sub>12</sub> H, <sup>35</sup> Cl,O <sub>3</sub> C <sub>12</sub> H	C,2H,3°Cl,3°ClO C,2H,3°Cl,3°ClO 19°C,2H,3°Cl,3°ClO 19°C,2H,3°Cl,3°ClO C,2H,3°Cl,3°ClO <sub>2</sub> C,2H,3°Cl,3°ClO <sub>2</sub> 19°C,2H,3°Cl,3°ClO <sub>2</sub> C,2H,3°Cl,3°ClO C,2°H,3°Cl,3°ClO C,2°H,3°Cl,3°ClO	C, 1, 1, 20 C, 1, 20 C, 2, 1, 20 C, 2, 2, 20 C, 2, 20 C, 20 C, 2, 20 C,
Ol nol	X	M+2 M+4 M+2 M+2 M+4 M+4 M+2 COCK	M+42 M+22 M+44 M+44 LOCK
Accurate mass®	303.9016 305.8987 315.9419 317.9389 319.8965 321.8936 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 403.8529 445.7555
Descriptor	-	Q	en

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

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

O = 15,994915  $^{3}CI = 34.968853$   $^{3}CI = 36.965903$ 

S = internal/recovery standard

LDC #:	24089DC2	
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### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:_	of
Reviewer:	FI
2nd reviewer:	W.

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

Y	h	N/A
(Y/	N	N/A

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

Concer	Concentration = $(A_{*})(I_{*})(DF)$ $(A_{*})(RRF)(V_{o})(\%S)$					
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured				
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard				
1,	=	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.				

Cxample.
Sample I.D. #12 , OCPP :
Conc. = (1894618) (4000) (13/0900 1.20) (10.02) (0.939
= 16.248 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	·				
<b></b>					

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 9 through August 10, 2010

**LDC Report Date:** 

October 15, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H180547

Sample Identification

SB03-24BPC

SB01-24BPC\*\*

**SB02-24BPC** 

SB03-24BPCMS

SB03-24BPCMSD

<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.

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 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	
0235250-MB 8/23/10		OCDD	1.1 pg/g	All samples in SDG	
		1,2,3,4,6,7,8-HpCDF	0.22 pg/g	G0H180547	

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

Sample EB-08102010 (from SDG H0H120594) 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-08102010	8/10/10	OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	4.4 pg/L 7.8 pg/L 5.2 pg/L 12 pg/L 8.4 pg/L 19 pg/L 11 pg/L 55 pg/L	SB01-24BPC** SB02-24BPC

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 for several compounds. Since the sample concentration was greater than the spiked concentration, no data were qualified.

# VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0235250-LCS	1,2,3,7,8,9-HxCDD	79 (80-143)	All samples in SDG G0H180547	J- (all detects) UJ (all non-detects)	Р

# VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

# X. Target Compound Identifications

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

# XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SB03-24BPC SB02-24BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
0235250-MB	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
0235250-MB	All compounds reported by the lab 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

# XIII. Overall Assessment of Data

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

# XIV. Field Duplicates

No field duplicates were identified in this SDG.

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H180547

SDG	Sample	Compound	Flag	A or P	Reason
G0H180547	SB03-24BPC SB01-24BPC** SB02-24BPC	1,2,3,7,8,9-HxCDD	J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (l)
G0H180547	SB03-24BPC SB02-24BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H180547

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H180547

No Sample Data Qualified in this SDG

Reviewer:							
SDG#	#: G0H180547		age 2B/4	Re	Page: /of / eviewer:		
LDC#	:24089E21		thgate Henderson LETENESS WORKSH	IEET	Date: 10/13/		

	Validation Area		Comments
l.	Technical holding times	A	Sampling dates: 8/9 - 8/10/10
II.	HRGC/HRMS Instrument performance check	A	
111.	Initial calibration	А	
IV.	Routine calibration/ICV-	Δ	
V.	Blanks	رىسى	
VI.	Matrix spike/Matrix spike duplicates	یسی	
VII.	Laboratory control samples	رسى	LC>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	یسی	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	5/N	EB= FB-08102010 >DG #60H120594

Note:

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

D = Duplicate TB = Trip blank EB = Equipment blank

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

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

	<u> </u>					
1	SB03-24BPC	11	0235250 -MB	21	31	
2	SB01-24BPC**	12		22	32	
3	SB02-24BPC	13		23	33	•
4	SB03-24BPCMS	14		24	34	,
5	SB03-24BPCMSD	15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

Notes:			

LDC #: 24089 E2/ SDG #: pu cones

# VALIDATION FINDINGS CHECKLIST

	Page:_	1	_of_	2
	Reviewer:		Ex	
2nd	Reviewer:		<u>q</u>	

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% ?	/		<u> </u>	
Is the static resolving power at least 10,000 (10% valley definition)?			<u> </u>	
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?	<u> </u>		L	
III. Initial calibration			r	
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?			1, <sub>1, 1</sub> , 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2,	
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?		gargassan.	ag about	
V. Blanks	i -	Ī		Г
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				Г
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.iLaboratory control samples	i —	r	T	j
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?	ı			

LDC #: 2408 9EZ SDG #: pu comes

# VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: 5
2nd Reviewer: 5

			31. 5.	Janes (TIII) Janes (TIII)		i Desgr			
VIII. Regional Quality Assurance and Quality Control				<del> </del>		us in guesta fusió	<u> </u>	<u>griss (1)</u>	
Were performance evaluation (PE) samples performed?  Were the performance evaluation (PE) samples within the acceptance limits?	<del> </del>		-	-					
IX: Internal standards	<u> </u>	1	<u> </u>						
Were internal standard recoveries within the 40-135% criteria?	<u> </u>	ļ						1000 to	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?					1				
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?		<u> </u>	ļ						
Was the Ion Abundance Ratio for the two quantitation ions within criteria?									
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?									
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?	_								
For PCDF identification, was any signal (S/N ≥ 2.5, at ± 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									2
Field duplicate pairs were identified in this SDG.			-						
Target compounds were detected in the field duplicates.									
XV:Field blanks				ı					
Field blanks were identified in this SDG.									
Target compounds were detected in the field blanks.									

# **VALIDATION FINDINGS WORKSHEET**

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

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

Notes:

1298045	
LDC#:	

# VALIDATION FINDINGS WORKSHEET Blanks

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".

Were all samples associated with a method blank? Z Z Z Z Z Z

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y/N N/A

Blank analysis date: 8/27/Was the method blank contaminated? Elank extraction date: arkappa'

Associated samples:

					:									
	ation													
	Sample Identification													
	Samp							 						
1														,
		<i>/</i> -S	5.5	1.1										
	Q	52 gC												
	Blank ID	apes 8 20	1.1	0.22										
1	6													
09/6	Compound		9	<b>P</b>										
units:	Com													
Conc.														

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 # 24089 E2

# **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: Bfank units:

Sampling date:  $\frac{1}{\sqrt{8/|\omega|/c}}$ 

EB= EB-08102010

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

Ī		IT -	1	Т	T	_	<del></del>	T	T	T	<del></del>	<del></del>	T	i	T	T	T	<del></del>	T	<del>T -</del>
	ation																			
	Sample Identification																			
	Blank ID	BP.	4.4*	7.8*	5.5	13	4.8	61	[/	52							-			
	pun																			
	Compound		6	#	FI	X	7	Ф	Р	প										CROL
L																				<u> </u>

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

LDC#: 24089E2

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

2nd Reviewer:\_ 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".

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

MS/MSD. Soil / Water.

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

#	Date	MS/MSD ID	Compound	l	MS %R (Limits)	MSD %R (Limits)	its)	RPD	RPD (Limits)	Associated Samples	Qualifications
-		ST h	fener	1/0 m	Recom	to tria			(	2	10 gual
			,	<i>'.</i>	RI	sw)	could	ie h	(itmit )	)	parlit 74x
_				•	( )	)	(		( )		1 spife Ant.
Н					( )	)	(		( )		, /
					( )	)	(		( )		
					( )	)	(		( )		
-					( )	)	^		( )		:
H					(						
					( )	)	(				
					( )	)	)		( )	100 100 100 100 100 100 100 100 100 100	
					( )	)	) (		( )		
					( )	)	(		)		
					( )	)	(		)		
					( )	)	(		)		
Н					( )	)	)		,	(	
$\dashv$	-				( )				)		
$\vdash$					( )	)	)		)		
					( )	)	(		,		
					( )	)	(		,	(	
					( )	)	(		)	(	
					( )	)	)		)	(	
					( )	)	(		)	(	
				,	( )		(		)		

24089E2 Le cons LDC #: SDG#:

# VALIDATION FINDINGS WORKSHEET **Laboratory Control Samples (LCS)**

2nd Reviewer:\_ 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".

Y N N/A

Was a LCS required?
Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

	L																									
Qualifications	nodsw d/sm/s	(I).,	(no cesp)																							
Associated Samples	)mo																									
RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	. (	( )	( )	( )	( )	( )	( )
LCSD %R (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	· ·	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
LCS %R (Limits)	19 (80-143)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
Compound	E																									
Lab ID/Reference	623 5350 · 1C >																									
# Date																										

LDC# 24089 E2/ SDG# Les core

# 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

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	(2) mod/C	an in the second		J/Adet. (Sp)	l		1 K det (R)	The second secon			
Associated Samples	6 1/			SW-aseseto			<i>^</i>				
Finding	x'd cal Range			Compel. superted below			longod uportul as EMPC				
Comple 4D	#, O, Q										
Date	·										
*											

Comments: See sample calculation verification worksheet for recalculations

# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET



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

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

calculations:

RRF =  $(A_{\rm h})(C_{\rm le})/(A_{\rm le})(C_{\rm s})$  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,

 $A_{\rm b}$  = Area of associated internal standard  $G_{\rm b}$  = 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 ( PAF3std)	RRF (RPF 3std)	%RSD	%RSD
-	1047	0//08/8	8/30// 2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	691.1	6911	1.209	1.26.09	75:5	25.5
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.35	1.325	1.2487	Car!	70.7	4:00
			1,2,3,6,7,8-HxCDD (°3C-1,2,3,6,7,8-HxCDD)	1-165	59/1	1.2152	1-2/52	6.20	6.20
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	08/:/	08/./	1.253	1.2034	29.5	295
			OCDE (13C.OCDD)	768.1	1.892	1.9979	1.9979	6.95	(6.7)
7	(on	01/12/8	8/31//C 2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	628-0	618-0	26.0	26.0	1.3	1.3
	DBINS		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 ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDE (13C.OCDD)						
3			2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)						
			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 ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-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

1DC# 24089E2/ SDG# 44 com

# VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_y)/(A_s)(C_x)$ 

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

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

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

					Reported	Recalculated	Ranorted	Recalculated
#	Standard ID	Calibration	Commound (Reference Internal Standard)	Average RRF	RRF	RRF	, A	à
			Compound (reperior mental Standard)	(mittal)	(22)	(22)	7.00	O%
-	UEN-50	01/1/6	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.149	1.140	1.17	0.3	6.3
	8E 15:6		2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.352	51.1	5/-/	8.7	8.7
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.165	81.1	8/-/	7.7	1.7
			1,2,3,4,6,7,8-HpCDD (¹³C-1,2,4,6,7,8,-HpCDD)	081.1	02.1	1.20	1.0	.6
			OCDF (3C-OCDD)	768.1	/.36	1.36	٧٠٠	5.
2	1	01/12/8	8/3//U 2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.169	1.169	7/1	0.7	6.7
	8:17:01		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.87	1.387	7-14	8.9	6.8
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.165	39/1	7.23	5.8	25
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	081.1	081:1	1.1/	٦. ٦	2.7
			OCDE (19C-OCDD)	1.×92	168./	1.81	4.5	4.5
က	aer	01/1/6	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.879	0.1	7.0	75.51	13.4
	DBIK							-
			1,2,3,6,7,8-HxCDD(42-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			Sept (13c-ocpp)					

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 ecalculated results

LDC# 24089E, SDG#:

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

Page:\_ 2nd Reviewer. 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)

b

T <sub>g</sub>				Ī	Ī		T	Ī	T	Τ		T
Recalculated	RPD				1							
Reported	RPD	1			15/							
Matrix Spike Duplicate	Percent Recovery	Recalc			ph							
Matrix Spik	Percent	Reported			pp							
Matrix Spike	Percent Recovery	Recalc			90							
Matrix	Percent F	Renorted			0%							
Sample	Concentration ( 29/9)	MSD			882							
				7.2.7	. <del>0/</del>							
Sample	Concentration ( 29/9)	000			Ohz							
ike	Added (P4/97)	/ WSD			501							
Ś	Ad )	) WS			103							
	Compound	100	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 Matrix Spike/Matrix Spike Duplicate findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% 4Rent >41/50 of the recalculated results.

24089E2 SDG #: Les yours LDC#

# Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

o 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 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 I \* 2/(LCS + LCSD)

234- Oxc 25 E0

LCS ID:

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

Recalculated US21/SD RPD Reported Recalc Percent Recovery TCSD Reported 5 bog Recalc 66 X 90 Percent Recovery K 156 Reported مر و 100 2 USD とな Spiked Sample Concentration 25:36 5.86 18.9 106 2/7 501 dsol <u>ና</u> 10/50 Spike Added, 200 100 00/ 20 001 Compound 1,2,3,4,7,8,9-HpCDF 1,2,3,4,7,8-HxCDD 1,2,3,7,8-PeCDD 2,3,7,8-TCDD 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.

# lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

	<u></u>			
	Analyte	HPCDF HPCDF HPCDF HPCDD HPCDD HPCDD (3) HPCDD (5) NCDPE PFK	ocdf ocdf ocdd ocdd ocdd (s) ocdd (s) bcdpe PfK	
	Elemental Composition	C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7ClO C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7ClO 10,2H <sup>45</sup> Cl <sub>18</sub> 7ClO 10,2H <sup>45</sup> Cl <sub>18</sub> 7ClO C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7ClO C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7ClO 10,2H <sup>45</sup> Cl <sub>18</sub> 7Cl <sub>2</sub> O 10,2H <sup>45</sup> Cl <sub>18</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7Cl <sub>2</sub> O C <sub>12</sub> H <sup>45</sup> Cl <sub>18</sub> 7Cl <sub>2</sub> O	C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>37</sup> ClO C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>27</sup> Cl <sub>2</sub> C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>27</sup> Cl <sub>2</sub> C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>27</sup> Cl <sub>2</sub> <sup>15</sup> C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>27</sup> Cl <sub>2</sub> <sup>16</sup> C <sub>12</sub> <sup>25</sup> Cl <sub>3</sub> <sup>27</sup> Cl <sub>2</sub>	
	Ol nol	M M M M M M M M M M M M M M M M M M M	M + 4 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 471.7750 513.6775	
	Descriptor	4	ທ	-
	Analyte	TCDF TCDF (8) TCDF (8) TCDD TCDD TCDD (8) TCDD (8) HXCDPE PFK	PecDF PecDF (S) PecDF (S) PecDD PecDD (S) PecDD (S) PecDD (S)	HXCDF HXCDF HXCDF (\$) HXCDD HXCDD HXCDD HXCDD (\$) HXCDD (\$) CCDPE
	Elemental Composition	C <sub>12</sub> H <sub>4</sub> *Cl <sub>1</sub> O C <sub>12</sub> H <sub>4</sub> *Cl <sub>3</sub> O 'S <sub>12</sub> H <sub>4</sub> *Cl <sub>3</sub> O 'S <sub>12</sub> H <sub>4</sub> *Cl <sub>4</sub> O 'S <sub>12</sub> H <sub>4</sub> *Cl <sub>4</sub> O C <sub>12</sub> H <sub>4</sub> *Cl <sub>4</sub> O 'SC <sub>12</sub> H <sub>4</sub> *Cl <sub>2</sub> O 'SC <sub>12</sub> H <sub>4</sub> *Cl <sub>2</sub> O 'SC <sub>12</sub> H <sub>4</sub> *Cl <sub>3</sub> OCl <sub>2</sub> O C <sub>2</sub> C <sub>13</sub> OCl <sub>2</sub> O	C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> J <sup>3</sup> ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> C <sub>1</sub> J <sup>3</sup> ClO 16 <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>2</sub> J <sup>3</sup> ClO 16 <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>2</sub> J <sup>3</sup> ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> J <sup>3</sup> ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> J <sup>2</sup> ClO 16C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> J <sup>2</sup> ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> J <sup>2</sup> ClO C <sub>12</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> J <sup>2</sup> ClO C <sub>2</sub> F <sub>13</sub>	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 10 <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> O 10 <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> O 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 <sub>2</sub> C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO <sub>2</sub> 10 <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO <sub>2</sub> 10 <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O
9	Ol noi	M M M M M M M M M M M M M M M M M M M	M M H + 2 A + 2 A + 4 A	M + 4 M + 2 M + 2 M + 2 M + 4 M + 4 M + 4 M + 4
Accesso magain	Acculate Illass	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	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555
Descriptor			0	ო

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 #:	24	089E	2/
SDG #:			

# **VALIDATION FINDINGS WORKSHEET**

# Sample Calculation Verification

Page:	of	_/
Reviewer:	F	2
2nd reviewer:_	Ø	

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

Y	N	N/A
$\overline{V}$	N	N/A

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

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

Concentration =  $(A_{\bullet})(I_{\bullet})(DF)$  $(A_{\bullet})(RRF)(V_{\circ})(\%S)$ 

A<sub>x</sub> = Area of the characteristic ion (EICP) for the compound to be measured

A<sub>ss</sub> = Area of the characteristic ion (EICP) for the specific internal standard

1 = Amount of internal standard added in nanograms (ng)

V<sub>o</sub> = Volume or weight of sample extract in milliliters (ml) or grams (g).

RRF = Relative Response Factor (average) from the initial calibration

Df = Dilution Factor.

%S = Percent solids, applicable to soil and solid matrices only.

Example:

Sample I.D. #2 . OC PD

= 758 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
==+					
+					
					-
					<u> </u>
					<del>                                     </del>
-					
					1
					1

# Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 13, 2010

**LDC Report Date:** 

October 19, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H190584

# Sample Identification

SSAK3-08-0BPC

SSAL4-04-0BPC

SSAL4-05-0BPC

SSAN6-09-0BPC

SSAN6-08-0BPC

SSAN5-04-0BPC

SSAO3-05-0BPC\*\*

SSAO3-04-0BPC

SSAO4-06-0BPC

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

## Introduction

This data review covers 9 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

## III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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
0235250-MB	8/23/10	OCDD 1,2,3,4,6,7,8-HpCDF	1.1 pg/g 0.22 pg/g	All soil samples in SDG G0H190584

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

No field blanks were identified in this SDG.

# VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds. Since the sample concentration was greater than the spiked concentration, no data were qualified.

# VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0235250LCS	1,2,3,7,8,9-HxCDD	79 (80-143)	All samples in SDG G0H190584	J- (all detects) UJ (all non-detects)	Р

# VIII. Regional Quality Assurance and Quality Control

Not applicable.

## IX. Internal Standards

All internal standard recoveries were within QC limits.

# X. Target Compound Identifications

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

# XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAL4-04-0BPC SSAL4-05-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAO3-04-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	Р
SSAN5-04-0BPC	OCDD	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р
SSAO4-06-0BPC	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 G0H190584	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H190584	All compounds reported by the lab 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 Stage 4 review was performed. Raw data were not evaluated for the samples reviewed by Stage 2B criteria.

## XIII. Overall Assessment of Data

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

# XIV. Field Duplicates

No field duplicates were identified in this SDG.

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H190584

		1			
SDG	Sample	Compound	Flag	A or P	Reason
G0H190584	SSAK3-08-0BPC SSAL4-04-0BPC SSAL4-05-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAN5-04-0BPC SSAO3-05-0BPC** SSAO3-04-0BPC SSAO4-06-0BPC	1,2,3,7,8,9-HxCDD	J- (all detects) UJ (all non-detects)	Р	Laboratory control samples (%R) (I)
G0H190584	SSAL4-04-0BPC SSAL4-05-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAO3-04-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H190584	SSAN5-04-0BPC	OCDD	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190584	SSAO4-06-0BPC	OCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190584	SSAK3-08-0BPC SSAL4-04-0BPC SSAL4-05-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAN5-04-0BPC SSAO3-05-0BPC** SSAO3-04-0BPC SSAO4-06-0BPC	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0H190584	SSAK3-08-0BPC SSAL4-04-0BPC SSAL4-05-0BPC SSAN6-09-0BPC SSAN6-08-0BPC SSAN5-04-0BPC SSAO3-05-0BPC** SSAO3-04-0BPC SSAO4-06-0BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H190584

No Sample Data Qualified in this SDG

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H190584

No Sample Data Qualified in this SDG

SDG # _abora	t:24089F21 #:G0H190584 atory: Test America	<del></del>	ALIDATIO	N COMI S	PLETEI tage 2B		SHEET	Date: 10 / 19/1 Page: 1 of 1 Reviewer: 197 2nd Reviewer: 19
	IOD: HRGC/HRMS Did							
i ne sa /alidat	amples listed below we tion findings worksheet	re revi s.	ewed for ea	ich of the i	following	validation areas	. Validation find	dings are noted in attached
	Validatio	n Area					Comments	
1.	Technical holding times	Δ	Sampling	dates: &//	3/10			
- 11.		HRGC/HRMS Instrument performance check			1			
III.	Initial calibration			Δ				
IV.	Routine calibration/ <del>ICV</del>			Α				
V.	Blanks			SW				
VI.	Matrix spike/Matrix spike o	Matrix spike/Matrix spike duplicates			SBC	03-24BPC	MS/D	
VII.	Laboratory control sample			<i>ال</i> ای		ふ	, -	
VIII.	Regional quality assurance		uality control	N				
IX.	Internal standards	-		٧.				
Χ.	Target compound identific	ations		<b>A</b>	Not reviewed for Stage 2B validation.			
XI.	Compound quantitation an		.s	SW	Not reviewed for Stage 2B validation.			
XII.	System performance			Δ	Not reviewed for Stage 2B validation.			
XIII.	Overall assessment of dat	a		Δ	Transfer of Stage 22 Validation			
XIV.	Field duplicates	,		12				
				N				
XV.	Field blanks			10	<u> </u>			
ote:	A = Acceptable N = Not provided/applicab SW = See worksheet	le	R = Rin	o compound sate eld blank	s detected	D = Duplio TB = Trip EB = Equi		
alidate	d Samples: ** Indicates san	nple und	lerwent Stage	4 validation				
1 / 5	SSAK3-08-0BPC	11 /	0235	250	21		31	
2 8	SSAL4-04-0BPC	12			22		32	
3 8	SSAL4-05-0BPC	13			23		33	
1 S	SSAN6-09-0BPC	14			24		34	
5 S	SSAN6-08-0BPC	15			25		35	
s s	SSAN5-04-0BPC	16			26		36	
' s	SSAO3-05-0BPC**	17			27		37	
3 S	SSAO3-04-0BPC	18			28		38	
s	SSAO4-06-0BPC	19			29		39	
10		20			30			

# LDC #: 24089 F2 | SDG #: ru cones

# VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
ITechnical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
III. 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			4.15	
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				
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 he QC limits?				

LDC#: 24089F2] SDG#: u comes

## **VALIDATION FINDINGS CHECKLIST**

Page: 2 of 2
Reviewer: 5
2nd Reviewer: 4

VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?		lane recovering	25 W 30 Ext 1 W	765 775 F.
IX: Internal standards				
Were internal standard recoveries within the 40-135% criteria?				
Was the minimum S/N ratio of all internal standard peaks ≥ 10?				
X. Target compound identification				
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?		-		
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?			_	
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding 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.	<u> </u>			
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.		P		

# VALIDATION FINDINGS WORKSHEET

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

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

Notes:

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TDC

# **VALIDATION FINDINGS WORKSHEET**

Blanks

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

> 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)

Were all samples associated with a method blank?

N N A A N A

Was a method blank performed for each matrix and whenever a sample extraction was performed?

Blank analysis date: 8 /27 // Was the method blank contaminated? Blank extraction date: 8/3 3/ Conc. units: A/N A

Associated samples:

\* EMPC

A// 801/5

Compound	Blank ID	5×		Sa	Sample Identification	ion		-
	OW-05 ESE EO	OW-d						
9	1.1	5.5						
Ф	0.22 *	1-1						
		-						
						,		

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".

1	
F	
8	
3	
N	
#	1
2	

# VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

2nd Reviewer:\_\_ Page: 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 associated

MS/MSD. Soil / Water.

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?

	ı —	_		1	Ť	T	Т	T	II .	T	Т	Т	T	<del>-</del>	Т	Τ	ır —	T	T	1	Т	Т	<del>_</del>
Qualifications	I ame								The state of the s														
Associated Samples	2002	MALLA .			Administration of the Control of the									TANAMATA TA									
RPD (Limits)	rioh ( )	( ) Sy'w,	( )		( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(
MSD %R (Limits)	were	6R 4 R		( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
MS %R (Limits)	ed group de	( , )	( )	( )	( )	( )	( )		( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Compound	Sene																						
MS/MSD ID	5803-34BPC	MSID																					
Date																							
#																							

LDC# 24087F2 SDG#:

# **VALIDATION FINDINGS WORKSHEET** Laboratory Control Samples (LCS)

Page: Reviewer.\_ 2nd Reviewer:

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

Y N N/A Y N/A

Was a LCS required? Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

						1									. '									ı		
ď	(1) d/cm/-r	ins 10 out no	1cs\$																							
Associated Samples	4//																									
RPD (Limits)		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
LCSD %R (Limits)		( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
LCS %R (Limits)	79 (80-143	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
Compound	Ē																									
Lab ID/Reference	577 05 25 2 20																									
# Date																										

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

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 N N/A

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

#	Date	Lompo Sample ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	Ail	JK detects (k)
		Д, Ө.Н	x of colfange	8,5,4,8,4	J/Polet (C)
			,	, , , ,	•
		6	7	9	1 (Peut (C)
		Ø	<i>^</i>	Ь	) /Palet (c)
					,
					:

Comments: See sample calculation verification worksheet for recalculations

LDC# 24089 F2) SDG#:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

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

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

RRF = (A<sub>x</sub>)( $C_{s_y}$ )( $C_{s_y}$ )( $C_{s_y}$ ) average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

 $A_{\rm is}=$  Area of associated internal standard  $C_{\rm is}=$  Concentration of internal standard X = Mean of the RRFs A<sub>x</sub> = Area of compound,
C<sub>x</sub> = Concentration of compound,
S = Standard deviation of the RRFs,

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (PPF 2 std)	RRF ( <i>RRF</i> 3std)	%RSD	%RSD
-	7401	01/29/	IL	0.875	0.875	0.87	78.0	7.11	14.2
				156.0	726.0	0.93	0.73	/3.5	13.5
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.242	1.242	1.27	1.27	2.6	13.6
			1,2,3,4,6,7,8-HPCDD (13C-1,2,4,6,7,8,-HpCDD)	1.026	1.026	1.07	1.07	13.6	13.6
			OCDE (13C. OCDD)	1.445	1.445	اري /	1.55	18.	18.1
2	1081	0/22/2	7/26/0 (2,3,7,8-TCDF (3C-2,3,7,8-TCDF)	1.036	1.056	7.07	10.1	3.32	3.32
	DBIN	,,	2, <del>8,7,8-TCDD (*C-</del> 2,3,7, <del>8,7C</del> DD)						
			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)						
			OCDE (190, OOPIN)						
8			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			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 (3C-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.

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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

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

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

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

 $A_{\rm is}$  = Area of associated internal standard  $G_{\rm is}$  = Concentration of internal standard X = Mean of the RRFs  $\begin{aligned} A_x &= A \text{rea of compound,} \\ C_x &= Concentration of compound,} \\ S &= S \text{tandard deviation of the RRFs,} \end{aligned}$ 

_	_			Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (PLF3std)	RRF (RDF 2std)	"RSD	
-	1047	0//05/8	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.169	6911	1.20	1.26.09	25.0	200
		`	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	7.82	1.352	1.2487	Lan.	4.00	4:00
T			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1-165	1.165	1.2152	1.2152	6.20	6.20
T			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	08/1	2/-/	1.255	1.2634	29.5	562
			OCDE (3C OCDD)	7.83.1	1.892	1.9979	1.99.7	26.9	10/
2	(cor	(11/18/8	8/3//() 23.7,8-TCDF (*6-2.3.7.8-TCDF)	618-0	618-0	092	180	1.7	7.3/
	DBINS	) . ,	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	, , ,		2/:2	27.0	/ //	/./>
			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)						
			OCDE (13C. OCDD)						
က			2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)						
			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 ("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.

SDG# 2408787

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_s)/(A_s)(C_x)$ 

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

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

 $A_{\rm is}$  = Area of associated internal standard  $C_{\rm is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>Q</b> %	Q%
-	aev-14	C/Le/8	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.875	760	76.0	7.11	6.11
		·	2,3,7,8-TCDD (¹3C-2,3,7,8-TCDD)	0.957	0.96	260	7.0	4.0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.107	21.1	1.17	2:7	2:5
			1,2,3,4,6,7,8-HpCDD (¹³C-1,2,4,6,7,8,-HpCDD)	1.026	1.12	1.12	9.6	3.6
			OCDF (¹3C-OCDD)	1.445	1.72	1.72	4.6	19.4
2	cev-2	01/6/6	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.05%	1.01	/0/	4.8	4.8
	DB225		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)					
			1,2,3,4,6,7,8,4pCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDE (**C-OCDD)					
3			2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (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)					

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 Comments:

123680h LDC #: SDG #:

## Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

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METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_x)(C_s)/(A_s)(C_x)$ 

Where:

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF  $A_x$  = Area of compound,  $A_y$  = Concentration of compound,  $G_s$ 

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	□%	<b>G</b> %
-	as-190	01/1/6	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	641.1	6211	1.17	6.3	6.9
	8E 15:6		2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.8%	51.1	5/-/	8.2	4.8
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.165	1.18	8/-/	2.1	1.7
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	081.1	07:1	1.20	9.1	1.6
			OCDE (¹³C-OCDD)	768.1	1.36	1.36	/٠۶	5: /
2	eev-15	01/18/8		1.167	691.1	1.14	0.7	6.7
	10:11:8	•	2,3,7,8-TCDD (¹3C-2,3,7,8-TCDD)	1.87	1.32.7	7.14	8.9	6.8
		<del>,</del>	1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1-165	39/1	7.23	S: 8	RS
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	08/:1	081.1	1.2/	1.8	2.7
			OCDE (13C-OCDD)	1.X92	168.	1.81	5.4	4.5
3	aes	01/1/6	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	0.879	1.0	1.0	13.4	13.4
	DBUK		(2,3,7,8 TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)				-	
			1,2,3,6,7,8-HxCDD (3-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			Sept (13c-ocpp)					

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#: 24089E2/

## VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample Results Verification

Page: /of\_ 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 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 I \* 2/(LCS + LCSD)

25- OSC 25 E EO

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

		petelu									
USJ I/SD	RPD	Recalculated									
SOI		Reported									
CSD	Recovery	Recalc									
Ül	Percent Recovery	Reported					24				
S.	ecovery	Recalc	35	901	66	Sh	201				
SOI	Percent Recovery	Reported	25	90/	66	26	80/				
amole	ration	U icsn	NA				1				
SpikedS	Concentration (P2/9)	10.1	18.9	901	5.86	S:56	217				
ike	Added, (99/9)	// 0 1 CSD	NΑ				<i>^</i>				
28	Ad Ad	) SOI	20	001	001	00/	202				
	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.

# Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

DF (S) 407.7818  DF (S) 417.8250  DD (S) 423.7767  DD (S) 423.7767  DD (S) 423.7767  DD (S) 423.7767  DD (S) 441.7428  CDF (S) 441.7428  CDF (S) 441.7428  CDF (S) 459.737  CDF (S) 469.737  CDF (S) 469.736  CDF (S) 613.6775  CDF (S)	Descriptor Accurate mass <sup>(x)</sup>	Ol nol	Elemental Composition	Analyte	Descriptor	Accurate Mass(8)	4		
η         M+2         Grave, στος στος στος στος στος στος στος στος	016	Σ	1 S C C S	1001	loudinana	Accurate Mass.	Ol nol	Elemental Composition	Analyte
Mathematical Control of the contro	3987 1419	M+2	C12H, 45C1, 40C10	TCDF	4	407.7818 409.7788	M + A 2 +	C <sub>12</sub> H <sup>35</sup> Cl <sub>8</sub> 7ClO	HpCDF
M	389	M+2	13C, H, 3CI, 3CIO	TCDF (S)		417.8250	Σ	13C12H3C1,O	HPCDF (S)
M+2   C <sub>12</sub> H <sub>2</sub> C <sub>12</sub> O'10 <sub>2</sub>   TODD   425.7737     M	355 25	≥ ;	C <sub>12</sub> H, <sup>sc</sup> C <sub>1</sub> O <sub>2</sub>	TCDD (3)		419.8220	M+2	13C <sub>12</sub> H <sup>25</sup> Cl <sub>8</sub> 37ClO	HpCDF
## M+2   "C_R_H_WCI_NTO_2"   TODD (S)   435.8169   ## M+2   "C_R_H_WCI_NTO_2"   TODD (S)   435.8169   ## M+2   C_R_H_WCI_NTO_2"   TODD (S)   437.8140   ## C_R_H_WCI_NTO_2"   TODD (S)   437.8140   ## C_R_H_WCI_NTO_2"   PECDF   PECD	8 89	Z + Z	C12H, 3C1, 3/C102	TCDD		425.7737		C;H,C,C,C,C	HPCDD
M+2   C <sub>1</sub> L <sup>1</sup> <sup>4</sup> <sup>4</sup> Cl <sub>3</sub> <sup>4</sup> ClO   HXDPE   HX	38	M+2	13C, H, 3C, 3CO,	1CDD (S)		435.8169		<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>8</sub> 37ClO <sub>2</sub>	HpCDD (S)
M+2   C <sub>1</sub> c <sub>1</sub> c <sub>1</sub> c <sub>2</sub> c <sub>1</sub> c <sub>2</sub> c <sub>1</sub> c <sub>2</sub> c <sub>1</sub> c <sub>2</sub> c <sub>2</sub> c <sub>2</sub> c <sub>2</sub> c <sub>3</sub> c <sub>2</sub> c <sub>2</sub> c <sub>3</sub> c <sub>2</sub> c <sub>3</sub>	64 7921	M+2	Cr.H.*Cl.37ClO	HXCDPE		479.7165	⊼ + + 4 +	13C12H <sup>23</sup> C1 <sup>23</sup> C1 <sup>2</sup> O <sup>2</sup> C1 H <sup>23</sup> C1 3C1 O	HPCDD (S)
7         M+2         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rClO         PecDF         5         441.7428           7         M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDF         5         443.7399           M+2 <sup>16</sup> C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDF         5         443.7399           M+4 <sup>16</sup> C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDF         5         443.7399           M+2         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDD         471.7750         471.7750           M+2         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDD         871.7750         471.7750           M+2         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PecDD         871.7750         871.8775           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PerCDD         871.8775         1422.9278           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PerCD         PerCDD         871.8775           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PerCD         PerCDD         871.8775           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>4</sub> 3rCl <sub>2</sub> O         PerCDD         PerCDD         871.8775           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>3</sub> arCl <sub>2</sub> O         PerCDD         PerCDD         PerCDD           M+4         C <sub>12</sub> H <sub>3</sub> eCl <sub>3</sub> arCl <sub>2</sub> O         PerCDD         PerCDD         PerCDD	Ī	5	<u>.</u>	PFK		[430.9728]	LOCK	CgF 17 C12 C12 C	PFK
M+2   10,12   14,3   15,2   14,3   15,2   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3   14,3   15,3	76	M+2	C12H3*C147CIO	PeCDF	က	441.7428	M+0	Ci 2%	
M+4 (197,7377  M+4 (197,7377  M+4 (197,7377  M+5 (197,1971)  M+6 (197,1971)  M+7 (197,1971)  M+7 (197,1971)  M+8 (197,1971)  M+1 (197,1971)  M+2 (197,1971)  M+4 (197,1971)  M+4 (197,1971)  M+5 (197,1971)  M+6 (197,1971)  M+7 (197,1971)  M+8 (197,1971)  M+9 (197,1971)  M+1 (197,1971)  M+1 (197,1971)  M+2 (197,1971)  M+2 (197,1971)  M+4 (197,1971)  M+5 (197,1971)  M+6 (197,1971)  M+7 (197,1971)  M+7 (197,1971)  M+8 (197,1971)  M+9 (197,1971)  M+1 (197,1971)  M+1 (197,1971)  M+2 (197,1971)  M+3 (197,1971)  M+4 (197,1971)  M+5 (197,1971)  M+6 (197,1971)  M+7 (197,1971)  M+7 (197,1971)  M+8 (197,1971)  M+9 (197,1971)  M	: 8	M + 4	12 12 12 12 12 12 12 12 12 12 12 12 12 1	PecDF		443.7399		C1, 20, 20, 20, 20, 20, 20, 20, 20, 20, 20	000
M+2 C <sub>12</sub> H <sub>3</sub> sC <sub>13</sub> C <sub>10</sub> C <sub>10</sub> PeCDD A93.738 A49.738 A49.738 A49.739 A4	0	M+4	10 To	Pecor (s)		457.7377		C <sub>12</sub> 351,37ClŌ2	OCDD
M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub> PeCDD A71.7750 M+2 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub> PeCDD (S) A71.7750 M+4 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub> PeCDD (S) A71.7750 M+2 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub> PeCDD (S) A71.7750 M+2 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub> PFK  M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDF M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDF M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDF M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD M+4 C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD M+4 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD (S) M+4 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD (S) M+4 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD (S) M+4 ( <sup>13</sup> C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O OCDPE	<b>6</b>	M+2	C <sub>12</sub> H <sub>3</sub> *CI <sub>1</sub> *CIO <sub>3</sub>	Pecdo (3)		459.7348		C <sub>12</sub> **Cl <sub>2</sub> *7Cl <sub>2</sub> O <sub>2</sub>	OCDD
M+2         "C <sub>12</sub> H <sub>3</sub> *C( <sub>1</sub> *7C( <sub>10</sub> )         PeCDD (S)         513.6775           M+4         "C <sub>12</sub> H <sub>3</sub> *C( <sub>1</sub> *7C( <sub>10</sub> )         PeCDD (S)         [422.9278]           M+2         C <sub>12</sub> H <sub>3</sub> *C( <sub>1</sub> *7C( <sub>10</sub> )         PFK         PFK           M+2         C <sub>12</sub> H <sub>3</sub> *C( <sub>1</sub> *7C( <sub>10</sub> )         HxCDF         HxCDF           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *7C( <sub>10</sub> )         HxCDF         HxCDF           M+2         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         HxCDF         HxCDF           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         HxCDD         HxCDD           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         HxCDD         HxCDD           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         HxCDD         HxCDD           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         HxCDD         HxCDD           M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         C <sub>10</sub> C <sub>10</sub> M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         C <sub>10</sub> C <sub>10</sub> M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         C <sub>10</sub> C <sub>10</sub> M+4         C <sub>12</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         C <sub>10</sub> C <sub>10</sub> M+4         C <sub>10</sub> H <sub>2</sub> *C( <sub>1</sub> *C( <sub>10</sub> )         C <sub>10</sub> C <sub>10</sub> M+4         C <sub>10</sub> H <sub>2</sub> *C( <sub>10</sub> *C( <sub>10</sub> )         C <sub>10</sub>	19 a	4+ X	C <sub>12</sub> H <sub>3</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O <sub>2</sub>	PeCDD		471.7750		13C <sub>12</sub> 33Cl <sub>2</sub> 37ClO <sub>2</sub>	(s) acoo
M+2	. თ	M+2	10, 12, 13, 10, 10, 10, 10, 10, 10, 10, 10, 10, 10	PeCDD (S)		513.6775		C. 35 1 37 1 0	OCDD (S)
2] LOCK C <sub>1</sub> F <sub>1</sub> C <sub>1</sub> C <sub>2</sub> C <sub>3</sub> C <sub>4</sub>	4	M+2	C.H. 401 37010	PeCDD (S)		[422.9278]		C12 (18 (12)	PEK
M+2 C <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rClO HXCDF M+4 C <sub>12</sub> H <sub>2</sub> scl <sub>4</sub> rCl <sub>2</sub> O HXCDF M (16 <sub>12</sub> H <sub>2</sub> scl <sub>4</sub> rCl <sub>2</sub> O HXCDF M+2 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rCl <sub>2</sub> O HXCDF M+4 C <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rClO HXCDF M+4 C <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rClO <sub>2</sub> HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rClO <sub>2</sub> HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rClO <sub>2</sub> HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rCl <sub>2</sub> O HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rCl <sub>2</sub> O HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rCl <sub>2</sub> O HXCDD M+4 (16 <sub>12</sub> H <sub>2</sub> scl <sub>3</sub> rCl <sub>2</sub> O OCDPE	92]	Lock	0,F <sub>13</sub> (1,6 (1)	PFK AT				2	
M+2 C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO HXCDF  M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO HXCDF  M+2 '3C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> O HXCDF  M+2 '3C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> O HXCDF  M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO HXCDD  M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO HXCDD  M+4 C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O HXCDD  M+4 (3C <sub>12</sub> H <sub>2</sub> *Cl <sub>3</sub> *Cl <sub>2</sub> O OCOPE									
M 15°, 1°, 2°, 2°, 2°, 2°, 2°, 2°, 2°, 2°, 2°, 2	<b>Σ</b> δ		C <sub>12</sub> H <sub>2</sub> *Cl <sub>2</sub> *ClO C <sub>1</sub> H <sub>2</sub> *Cl <sub>3</sub> *ClO	HXCDF					
M+2 '3C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7ClO HXCDF M+2 C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7ClO HXCDF M+4 C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7ClO <sub>2</sub> HXCDD M+2 '3C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7ClO <sub>2</sub> HXCDD M+4 '3C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7Cl <sub>2</sub> O HXCDD M+4 '3C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7Cl <sub>2</sub> O HXCDD M+4 C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7Cl <sub>2</sub> O HXCDD M+4 C <sub>12</sub> H <sub>2</sub> 3C <sub>15</sub> '7Cl <sub>2</sub> O OCDPE	68		O.O. T. T. O.O.	HYCOF (%)					
M+2 C <sub>12</sub> H <sub>2</sub> GCl <sub>2</sub> Cl <sub>2</sub> Cl <sub>2</sub> M+4 C <sub>12</sub> H <sub>2</sub> GCl <sub>2</sub> Cl <sub>2</sub> M+2 G <sub>12</sub> H <sub>2</sub> GCl <sub>2</sub> Cl <sub>2</sub> HXCDD M+2 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> GCl <sub>2</sub> TCl <sub>2</sub> HXCDD M+4 <sup>13</sup> C <sub>12</sub> H <sub>2</sub> GCl <sub>3</sub> TCl <sub>2</sub> HXCDD M+4 C <sub>12</sub> H <sub>2</sub> GCl <sub>3</sub> TCl <sub>2</sub> O CDPE	0		<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>32</sup> Cl <sub>2</sub> <sup>37</sup> ClO	HXCDF (S)					
M+4 C <sub>12</sub> H <sub>2</sub> sCl <sub>4</sub> sCl <sub>2</sub> O <sub>2</sub> HXCDD M+2 'sC <sub>12</sub> H <sub>2</sub> sCl <sub>3</sub> sCl <sub>6</sub> sCl <sub>0</sub> HXCDD M+4 'sC <sub>12</sub> H <sub>2</sub> sCl <sub>3</sub> sCl <sub>2</sub> O <sub>2</sub> HXCDD M+4 C <sub>12</sub> H <sub>2</sub> sCl <sub>3</sub> sCl <sub>2</sub> O <sub>2</sub> OCDPE	9 6	X+2	C <sub>12</sub> H <sub>2</sub> *Cl <sub>5</sub> *ClO <sub>2</sub>	HXCDD					
M+4 13C <sub>12</sub> H <sub>2</sub> 3C <sub>1</sub> C <sub>1</sub> C <sub>2</sub> HXCDD HXCDD M+4 C <sub>12</sub> H <sub>2</sub> 3C <sub>1</sub> C <sub>2</sub> C <sub>2</sub> C <sub>2</sub> C <sub>2</sub> C <sub>3</sub>	•		C12H2 2C12O2	HXCDD			-		
M+4 C <sub>12</sub> H <sub>2</sub> C <sub>2</sub> C <sub>2</sub> C <sub>3</sub> C <sub>2</sub>			12' 12' 12' 12' 12' 12' 12' 12' 12' 12'	HXCDD (S)					
a O XOO	10.3		C <sub>12</sub> H <sub>2</sub> 3C <sub>18</sub> C <sub>12</sub> C <sub>2</sub> C <sub>2</sub> C <sub>12</sub> C <sub>12</sub> C <sub>12</sub> C <sub>12</sub> C <sub>12</sub> C		-				
27 60	<u></u>		C,F,	PFK			·		

(a) The following nuclidic masses were used:

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

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

S = internal/recovery standard

LDC #:	2	4089F	2/
_		cover	

Dilution Factor.

Df

%S

## **VALIDATION FINDINGS WORKSHEET**

## Sample Calculation Verification

Page:	_/_of	
Reviewer:_	F	2
2nd reviewer:_		<u>/</u>

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

Percent solids, applicable to soil and solid matrices

Y N N/A Were all reported results recalculated and Were all recalculated results for detected t	verified for all level IV samples? arget compounds agree within 10.0% of the reported results?
Concentration = $(A)(I_*)(DF)$	Example:
$(A_{\pm})(RRF)(V_o)(\%S)$ A = Area of the characteristic ion (EICP) for the	Sample I.D. # 10:

#	Sample ID	Compound		Reported Concentration ( )	Calculated Concentration ( )	Qualification
	Odnipio io					
<b> </b>						
			,			
-						
1						

## Laboratory Data Consultants, Inc. Data Validation Report

**Project/Site Name:** 

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 13, 2010

LDC Report Date:

October 15, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H190600

## Sample Identification

SSAL3-07-0BPC

SSAK8-10-0BPC\*\*

SSAK8-09-0BPC

SSAK8-07-0BPC

SSAN6-03-0BPC

RSAN7-0BPC

SSA07-03-0BPC

SA44-0BPC

SSAP3-01-0BPC

SSAO4-01-0BPC\*\*

SSAK3-05-0BPC

SSAK3-07-0BPC

SSAK8-07-0BPCMS

SSAK8-07-0BPCMSD

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

### Introduction

This data review covers 14 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

### III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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
0237235-MB	8/25/10	OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.23 pg/g 0.13 pg/g 0.10 pg/g 0.39 pg/g 0.11 pg/g 0.70 pg/g	All samples in SDG G0H190600

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

No field blanks were identified in this SDG.

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were not within QC limits for several compounds. Since the sample concentration was greater than the spiked concentration, no data were qualified.

## VII. Laboratory Control Samples (LCS)

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

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAK8-10-0BPC**	<sup>13</sup> C-OCDD	35 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAK8-09-0BPC	<sup>13</sup> C-OCDD	34 (40-135)	OCDD	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
SSAK8-07-0BPC	<sup>13</sup> C-OCDD	27 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAO7-03-0BPC	<sup>13</sup> C-OCDD	38 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAK3-05-0BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDD <sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 27 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

## X. Target Compound Identifications

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

## XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria ·	Flag	A or P
SSAL3-07-0BPC SSAK8-09-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects)	Р
SSAK8-10-0BPC** SSAK8-07-0BPC	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
RSAN7-0BPC SA44-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	Р
SSAO4-01-0BPC**	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р

Sample	Compound	Finding	Criteria	Flag	A or P
SSAK3-05-0BPC SSAK3-07-0BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H190600	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H190600	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

## XII. System Performance

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

## XIII. Overall Assessment of Data

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

## XIV. Field Duplicates

No field duplicates were identified in this SDG.

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H190600

SDG	Sample	Compound	Flag	A or P	Reason
G0H190600	SSAK8-10-0BPC** SSAK8-09-0BPC SSAK8-07-0BPC SSAO7-03-0BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H190600	SSAK3-05-0BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0H190600	SSAL3-07-0BPC SSAK8-09-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190600	SSAK8-10-0BPC** SSAK8-07-0BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190600	RSAN7-0BPC SA44-0BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H190600	SSAO4-01-0BPC**	OCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190600	SSAK3-05-0BPC SSAK3-07-0BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H190600	SSAL3-07-0BPC SSAK8-10-0BPC** SSAK8-09-0BPC SSAK8-07-0BPC SSAN6-03-0BPC RSAN7-0BPC SSAO7-03-0BPC SA44-0BPC SSAP3-01-0BPC SSAO4-01-0BPC** SSAK3-05-0BPC SSAK3-07-0BPC	All compounds reported below the PQL.	J (all detects)		Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason
G0H190600	SSAL3-07-0BPC SSAK8-10-0BPC** SSAK8-09-0BPC SSAK8-07-0BPC SSAN6-03-0BPC RSAN7-0BPC SSAO7-03-0BPC SA44-0BPC SSAP3-01-0BPC SSAO4-01-0BPC** SSAK3-05-0BPC SSAK3-07-0BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H190600

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H190600

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

		1 11-
LDC #: 24089G21	VALIDATION COMPLETENESS WORKSHEET	Date: 10/14/jū
SDG #: <u>G0H190600</u>	Stage 2B/4	Page: / of /
Laboratory: Test America		<del>-</del>
METHOD: HRGC/HRMS Diox	ins/Dihenzofurans (EDA SW 846 Mothed 9200)	Reviewer:

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	Δ	Sampling dates: 8 13 10
11.	HRGC/HRMS Instrument performance check	Δ	
111.	Initial calibration	Δ	
IV.	Routine calibration/ <del>ICV</del>	Δ	
V.	Blanks	سی	
VI.	Matrix spike/Matrix spike duplicates	~SW	
VII.	Laboratory control samples	A	lc>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

N.	احا	te		

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 Stage 4 validation

		solv					
,	1	SSAL3-07-0BPC	14 11	SSAK3-05-0BPC	21	0237235-MB	31
4	2	SSAK8-10-0BPC**	17 12	SSAK3-07-0BPC	22	-	32
6	3	SSAK8-09-0BPC	13	SSAK8-07-0BPCMS	23		33
8	4	SSAK8-07-0BPC	14	SSAK8-07-0BPCMSD	24		34
10	5	SSAN6-03-0BPC	15		25		35
11	6	RSAN7-0BPC	16		26		36
12	7	SSAO7-03-0BPC	17		27		37
13	8	SA44-0BPC	18		28		38
14	9	SSAP3-01-0BPC	19		29		39
15	10	SSAO4-01-0BPC**	20		30		40

Notes:	

## LDC #: 24089 Q2 | SDG #: m cones

## VALIDATION FINDINGS CHECKLIST

	Page:	/	_of_	2
	Reviewer:		E	
2nd	Reviewer:		4	

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				Language Commons
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% ?				
ls 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 ≥ 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	<u> </u>			
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		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?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC #: 24089 Q2 SDG #: 100 comm

## VALIDATION FINDINGS CHECKLIST

Page:_	2-of_	2_
Reviewer:	E	<u>,                                    </u>
2nd Reviewer:	D	

VIII. Regional Quality Assurance and Quality Control				
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?	Mar	/	1	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	$\perp_{\nu}$	<u> </u>		
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?			ļ	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?			<u> </u>	79 79 713 404 7 7 3 44 4 5 7 4 4 4 5 7 4 4 4 5 7 4 4 4 5 7 4 4 4 5 7 4 4 5 7 4 5 7 4 5 7 4 5 7 4 5 7 4 5 7 4 5 7 4 5 7 4 5 7 4 7 4
Was the signal to noise ratio for each target compound and labeled standard $\geq 2.5$ ?				
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ seconds RT) detected in the corresponding PCDPE channel?				
Was an acceptable lock mass recorded and monitored?				
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII System performance				
System performance was found to be acceptable.				
XIII. Overall assessment of data			1	
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates			I	
Field duplicate pairs were identified in this SDG.		_		
Target compounds were detected in the field duplicates.			-	
XV. Fleid blanks				
Field blanks were identified in this SDG.			ł	
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:

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## VALIDATION FINDINGS WORKSHEET Blanks

Page:\_ 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". Were all samples associated with a method blank? ∀ Z

N/A

X/N N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed? Was the method plank contaminated?

\* FMPC

Associated samples: Blank analysis date: 9/3/0Blank extraction date:\_ Conc. units: pa

Г			T	T	T	П	Г	 Τ		T	<u> </u>	 	T	Ι		Г	
					-												
ion									-								
Sample Identification						-											
Sa																	
XS	-MB	1.15	0.65	2.0	56-1	6.56	3.5										
Blank ID	OH-8527550	0.23 #	0.73	0.10 ₩	6.39	0.11 #	C <del>L</del> .0										
Compound			K	7	Ф	Р	<i>ه</i>										
Сот		8		-			-										

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#: 240 89 92/

## VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

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

MS/MSD. Soil / Water.

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

Qualifications	no qual 101,n	(parket 74X)	Lut x'as	ľ																			
Associated Samples	ħ																						
RPD (Limits)	o/o pag	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )
MSD %R (Limits)	nds % R +	(imit)	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
MS %R (Limits)	1	outsid.	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )
Compound	rene	wen																					
MS/MSD ID	11+51																						
Date																							
#																_							

LDC# 2408962/

## **VALIDATION FINDINGS WORKSHEET**

Internal Standards

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".

Y N N/A

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

*	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	40-135%)	Qualifications
$\dashv$		4	H	35	( SE1-Op)	1/11/18 (i) one G,B
					( 1 )	, ,
		3	<b>~</b>	34		
		4	<b>↑</b>	27	(	
		7	<i>^</i>	38		<b>→</b>
					(	
			Н	32		1 1 mm (1)
$\exists$			I	72		4,8
			9	34	( >	9,P
	, :				(	•
		(3	7	33		no apred m>
			T	32	(	
			П	39		
			B	33	( )	
					(	•
		<del> </del>	I	37		USM Lamp OM
		Internal Standards	Check Standard Used	Rec	Recovery Standards	Check Standard Used
A.	<sup>13</sup> C-2.3.7.8-TCDF	DF		K. 13C-1,2,3,4-TCDD		
В	13C-2,3,7,8-TCDD	DD			טט:	
4	<sup>13</sup> C-1 2 3 7 8-PeCDE	PECDE		M		
4	<sup>13</sup> C-1 2 3 7 8-PeCDD	PECDD		Z		
Щ	<sup>13</sup> C-1,2,3,6,7,8-HxCDE	-HxCDF		O		
щ	13C-1,2,3,6,7,8-HxCDD	-HxCDD		а		
G	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDE	, 8-нрСDF		q		
ㅋ	13C-1,2,3,4,6,7,8-HpCDD	, 8-НрСDD		В		
1	13C OCDD			]		

# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

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

Y N N/A

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).

		spounds			
#	Date	Sample-IB	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	IIA	J/A detects (sp)
				entremental Andrews Andrews and the Control of the	
			All compounds reported as EMPC	All	JK detects (k)
		H. O, P, Q	x'd cal lange	X.6,1	1/P dut (e)
		Ø, Ø	7	2,4	
		1			
		H, O, A	7	8'9	
		Ø,	7	01	
		H, K, O, P, B	~	71 11 21	

Comments: See sample calculation verification worksheet for recalculations

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of A Reviewer: 2nd Reviewer:

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

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

RRF =  $(A_s)(C_{s_s})/(A_{s_s})(C_s)$  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,

 $A_{l_{b}} = Area \ of \ associated \ internal \ standard$   $G_{l_{b}} = Concentration \ of \ internal \ standard$  s,  $X = Mean \ of \ the \ RRFs$ 

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (RCF3 std)	RRF (PRF3 std)	%RSD	%RSD
1	7421	01/20/1	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.050	1.052	101	1.02	3.32	3.32
	Surga	<u>.</u>	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)						
		· · · · ·	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)						
			OCDE (13C OCDD)						
2	141	2/29/5	8/34/0 2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	691-1	1.169	6021	6072.1	7555	5:52
			2,3,7,8-TCDD (¹C-2,3,7,8-TCDD)	1.22	1,252	1-2887	[882.1	0.4	X
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.165	1.165	252-1	15/e-1	6.20	9.9
		·····	1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	081-1	081-1	1.2654	15 x.1	562	275
			OCDE (13C.OCDD)	1.892	1492	1.9979	1.99.79	6.95	6.95
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD ( <sup>3</sup> C-2,3,7,8-TCDD)						
		<del></del>	1,2,3,6,7,8-HxCDD ( <sup>3</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)						
			OCDE (3C-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# 24087921/ SDG# 140 com

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA Method TO-9A)

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

% Difference = 100 \* (ave. RRF - RRF)/ave. RRF RRF =  $(A_{\nu})(C_{s})/(A_{is})(C_{\nu})$ 

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,

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

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF	RRF	RRF	<b>U</b> %	٧,
T	gen-19	01/4/6	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.056	1.12	1.12	۲۰۶	6.3
	DB 225	· ·	-2,3,7,8-TCbB (130-2,3,7,8-1CBD)					
			1,2,3,6,7,8-HxCDD (2,5,3,6,7,8-HxCDD)					
Ī			1,2,3,4,6,2,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			Contraction of the contraction o					
2	/E-NOO	9/3/10	9/3/10 2,3,7,8-TCDF (³C-2,3,7,8-TCDF)	1.169	7.0%	1.08	8-6	7.8
		· ·	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.352	61-1	61-1	7-5	/3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1-165	1.12	71-1	3-7	3.7
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	081-1	8/-/	81.1	0.3	6-3
			OCDF (13c-OCDD)	768-1	1.62	1.62	/4.3	14.3
3			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)					
		· · · · ·	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)					

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. Comments:

LDC# 24089921 te con SDG#

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

2nd Reviewer:\_ 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:

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

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

40 MS/MSD samples:

$\Box$					<u> </u>	Ι		i	Ĭ	<u> </u>		<u> </u>
Recalculated	RPD	1 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	77	//	77	11	6					
Reported	RPD	1000	22	//	27	77	S					
- Duplicate	Recovery	Recalc	277	hal	187	26.30	Obis					
Matrix Spike Duplicate	Percent Recovery	Reported		401	181	0802	060					
Matrix Spike	Percent Recovery	Recalc	163	20	///	263	0					
Matrix	Percent F	Reported	591	22	611	327	0					
Sample	ncentration	// MSD		346	118	99.501	\$0001S					
Spiked Sample	Concentration ( Pg /9)	<i>) (</i> MS	tt 93.9	311	182	0/8	DODIS 00172					
Sample	Concentration (PN/91)		19	Ope	120	0017	000ah					
ike	Added	/U MSD	1.02	001	7	4	1 oc					
ds	Added ( 03/7	O /	20.0	8.66	1	7	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 Matrix Spike/Matrix Spike Duplicate findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC#: 240 89 G2, SDG#: 444 Couch

# 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 W

Where: SSC = Spiked sample concentration SA = Spike added

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

LCS = Laboraotry control sample percent recovery

LCS ID: 02 37235- 163

LCSD = Laboratory control sample duplicate percent recovery

	8	الم	Spiked	alumat	80	<i>u</i>	USDI	C	US2/1/CSD	csp
Compound	Ag G	Added (0%/%)	Concentration (PS/9)	tration (9)	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	۵۰
	) r (S) l	رار ۱ CSD	.0/	Jusp	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	S. 65	42	6.81	AΛ	56	36				
1,2,3,7,8-PeCDD	001		92.9		93	23				
1,2,3,4,7,8-HxCDD	001		24.7		95	95				
1,2,3,4,7,8,9-HpCDF	001		001		001	001				
OCDF	Oot	1	89/	1	<i>þ8</i>	Z	245			
							\		,	
								-		

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.

Elemental Composition
C,z,t,*sCi,O C,z,t,*sCi,**C10 'sC,z,t,*sCi,O 'sC, t, *sCi,**Cl,O
13C <sub>12</sub> H, 3C <sub>13</sub> (5) C <sub>12</sub> H, 3C <sub>13</sub> (5) C <sub>14</sub> H, 3C <sub>13</sub> (6) HXCDPE C <sub>15</sub> F <sub>13</sub> PFK
C. H. 3 C. 1 C
C12H2**C12**C102***C12HXCDD C12H2**C13**C12O***C12O

(a) The following nuclidic masses were used:

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

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

S = internal/recovery standard

LDC #:	24	1089	92/
SDG #:			,

## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:_	of	
Reviewer:_	F	<u> </u>
2nd reviewer:_	W.	!
	•	

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

/ <sub>Y</sub>	h	N/A
$(\cancel{y}$	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	tration	=
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard
l,	=	Amount of internal standard added in nanograms (ng)
V <sub>°</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.
<b>%</b> S	=	Percent solids, applicable to soil and solid matrices only.

Example: Sample I.D.  $\frac{2}{2}$ .  $\frac{20173}{1852239}$ .  $\frac{129}{10.150}$ :  $\frac{1517.66}{1520.85}$ 

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	વ				
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## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 23, 2010

**LDC Report Date:** 

October 15, 2010

Matrix:

Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H250498

Sample Identification

EB-08232010

### Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

## I. Technical Holding Times

All technical holding time requirements were met.

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

## II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

### III. Initial Calibration

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

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

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

## IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

### V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0260402	9/2/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-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	1.6 pg/L 1.1 pg/L 1.0 pg/L 8.0 pg/L 29 pg/L 1.7 pg/L 2.0 pg/L 1.7 pg/L 1.6 pg/L 2.2 pg/L 1.8 pg/L 4.0 pg/L	All samples in SDG G0H250498

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-08232010	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.97 pg/L 0.67 pg/L 1.3 pg/L 4.4 pg/L 2.9 pg/L 1.7 pg/L 1.6 pg/L 3.2 pg/L 2.0 pg/L 8.1 pg/L	0.97U pg/L 0.67U pg/L 1.3U pg/L 4.4U pg/L 2.9U pg/L 1.7U pg/L 1.6U pg/L 3.2U pg/L 2.0U pg/L 8.1U pg/L

Sample EB-08232010 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-08232010	8/23/10	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.97 pg/L 0.67 pg/L 1.3 pg/L 4.4 pg/L 2.9 pg/L 1.7 pg/L 1.6 pg/L 3.2 pg/L 2.0 pg/L 8.1 pg/L	No associated samples in this SDG

## VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable.

## VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0260402	1,2,3,6,7,8-HxCDF	136 (76-133)	All samples in SDG G0H250498	J+ (all detects)	Р

## VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

All internal standard recoveries were within QC limits.

## X. Target Compound Identifications

Raw data were not reviewed for this SDG.

## XI. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H250498	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0H250498	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

Raw data were not reviewed for this SDG.

## XII. System Performance

Raw data were not reviewed for this SDG.

## XIII. Overall Assessment of Data

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

## XIV. Field Duplicates

No field duplicates were identified in this SDG.

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H250498

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H250498	EB-08232010	1,2,3,6,7,8-HxCDF	J+ (all detects)	Р	Laboratory control samples (%R) (I)
G0H250498	EB-08232010	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0H250498	EB-08232010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А	Project Quantitation Limit (k)

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H250498

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H250498	EB-08232010	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	0.97U pg/L 0.67U pg/L 1.3U pg/L 4.4U pg/L 2.9U pg/L 1.7U pg/L 1.6U pg/L 3.2U pg/L 2.0U pg/L 8.1U pg/L	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0H250498

No Sample Data Qualified in this SDG

## Tronox Northgate Henderson

	_ VA		N COMF	PLETENE	SS WORK	SHEET	Date: 10/1
			3	stage 2B			Page: <u>    /</u> of_ Reviewer: <i></i>
OD: HRGC/HRMS Diox	kins/Γ	)ibenzofurar	ns (FPA SI	// 846 Met	hod 8290\		2nd Reviewer:
					·		
ımples listed below wer ion findings worksheets	e revi	ewed for ea	ch of the f	ollowing va	ilidation areas.	Validation finding	ngs are noted in attache
	•						
Validation	Area					Comments	
Technical holding times			Δ	Sampling da	ates: g	23/10	
HRGC/HRMS Instrument p	erform	ance check	Δ				
Initial calibration			Δ				
Routine calibration/I <del>C</del> ∀			Δ				
Blanks			SW				
Matrix spike/Matrix spike du	ıplicate	es	N	6C	Sampl	<u>.</u>	
Laboratory control samples			چس	Le	(1)		
Regional quality assurance	and qu	uality control	N				
Internal standards			Α				
Target compound identifica	tions		N				
Compound quantitation and	CRQL	.s	N				
System performance			N				
Overall assessment of data			A				
Field duplicates			N				
				EB:	<del></del>		
A = Acceptable N = Not provided/applicable SW = See worksheet	•	R = Rins	o compounds sate		D = Duplic TB = Trip b	olank	
samples: walk							
B-08232010	11	026040	)2_	21		31	
	12			22		32	·
	13			23		33	
	14			24		34	
	15		4	25		35	
	16			.26		36	
	17			27		37	
	18			28		38	
	19			29		39	
	20			30		40	
	E G0H250498 atory: Test America  OD: HRGC/HRMS Diox amples listed below wer ion findings worksheets  Validation  Technical holding times  HRGC/HRMS Instrument p Initial calibration  Routine calibration/PCV  Blanks  Matrix spike/Matrix spike du Laboratory control samples  Regional quality assurance Internal standards  Target compound identifica  Compound quantitation and System performance  Overall assessment of data  Field duplicates  Field blanks  A = Acceptable N = Not provided/applicable	atory: Test America  OD: HRGC/HRMS Dioxins/Damples listed below were revision findings worksheets.  Validation Area Technical holding times HRGC/HRMS Instrument perform Initial calibration Routine calibration/Pev Blanks Matrix spike/Matrix spike duplicate Laboratory control samples Regional quality assurance and quality assuranc	E: G0H250498 attory: Test America  OD: HRGC/HRMS Dioxins/Dibenzofurar amples listed below were reviewed for earlien findings worksheets.  Validation Area  Technical holding times  HRGC/HRMS Instrument performance check Initial calibration  Routine calibration/IPCV  Blanks  Matrix spike/Matrix spike duplicates  Laboratory control samples  Regional quality assurance and quality control internal standards  Target compound identifications  Compound quantitation and CRQLs  System performance  Overall assessment of data  Field duplicates  Field blanks  A = Acceptable ND = Nc R = Rinc SW = See worksheet FB = Field Samples:  B-08232010 11 024046  12 13 14 15 16 16 17 18 18 19	E GOH250498 atory: Test America  OD: HRGC/HRMS Dioxins/Dibenzofurans (EPA S) amples listed below were reviewed for each of the fion findings worksheets.  Validation Area  Technical holding times  A HRGC/HRMS Instrument performance check Initial calibration  Routine calibration/PeV  A Blanks  Matrix spike/Matrix spike duplicates Laboratory control samples  Regional quality assurance and quality control Internal standards  A Target compound identifications  Compound quantitation and CRQLs  System performance  N Overall assessment of data  Field duplicates  N = Acceptable N = Not provided/applicable SW = See worksheet  D = No compounds R = Rinsate FB = Field blank  A Samples:  B-08232010  11  D240402  12  13  14  15  16  17  18  19	Stage 2B atory: Test America  OD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Met amples listed below were reviewed for each of the following varion findings worksheets.  Validation Area  Technical holding times  A Sampling de HRGC/HRMS Instrument performance check Initial calibration  Routine calibration/IEV  Blanks  Matrix spike/Matrix spike duplicates  N & C C Laboratory control samples  Regional quality assurance and quality control Internal standards  Target compound identifications  Compound quantitation and CRQLs  System performance  N Overall assessment of data  Field duplicates  Field duplicates  Field duplicates  A = Acceptable N = Not provided/applicable SW = See worksheet  De See worksheet  Target compounds detected R = Rinsate F = Field blank  B-08232010  11	Stage 2B atory: Test America  OD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290) amples listed below were reviewed for each of the following validation areas. ion findings worksheets.  Validation Area  Technical holding times  A Sampling dates: 8 HRGC/HRMS Instrument performance check A Initial calibration  Routine calibration/IeV  A Blanks  Matrix spike/Matrix spike duplicates Laboratory control samples  Regional quality assurance and quality control Internal standards  A Target compound identifications  Compound quantitation and CRQLs  System performance  Overall assessment of data  A = Acceptable N = Not provided/applicable SW = See worksheet  A = Acceptable SW = See worksheet  Target compounds detected A D = Duplic B = Field blank  Target compounds detected C = D = Duplic B = Field blank  D = Duplic B = Field blank  Target compounds  A = Acceptable SW = See worksheet  A = Acceptable SW = See worksheet  Target compounds  A = Acceptable SW = See worksheet  A = Rinsate FB = Field blank  TB = Trip t FB = Field blank  TB = Trip t FB = Equir  A = Acceptable SW = See worksheet  Target compounds detected A D = Duplic B = Trip t FB = T	Stage 2B  atory. Test America  OD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)  amples listed below were reviewed for each of the following validation areas. Validation finding in findings worksheets.  Validation Area  Comments  Technical holding times  A Sampling dates: \$ 23 10

## 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 Pacine
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF
		•		

Notes:

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## VALIDATION FINDINGS WORKSHEET

Reviewer:\_ 2nd Reviewer:\_

Page:

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)

Was a method blank performed for each matrix and whenever a sample extraction was performed? Were all samples associated with a method blank?

Was the method blank contaminated? N N/A

Conc. units: 🗷

Blank analysis date: 4/10/10 9210 Blank extraction date:

Associated samples:

AL

EMPC \*

Sample Identification 1.3\*/1 4.4\* 3-2\*/4 11/\*/.8 M/\*L60 0.67\*/1 2.0/4 2.9/4 1.7 /U 1.6\*/2 £0.0 0.0 \ \ \ \ . ં & O 5.0 e J SiS 70 Ұ 었 0560402 \* Blank ID \*01 \* ·0\* 4.0 & 0 6 0,4 ڊ 1 Compound ≨ Z Ф 8 ত 1 Q سا ၂

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#: 14089#2

## **VALIDATION FINDINGS WORKSHEET** Field Blanks

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

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

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

FB Biank units: po/L Associated sample units: つん Sampling date: 0 8 23 0 C Field blank type: (circle one) Field Blank / Rinsate / Other:

EMPC \*

old blank type: (circle one	e) Field Blank / F	Field blank type: (circle one) Field Blank / Rinsate / Other: モウ Associated Samples:
Compound	Blank ID	Sample Identification
Ω	0.97*	
תו	0.01*	
<b>L</b>	1.3*	
þ	4.4*	
<b>Y</b>	P.4	
	1.7	
7	¥ 9.1	
Ф	3.2 *	
<b>12</b>	0.2 20	
Ø	* 1.8	
THE PERSON NAMED IN COLUMN NAM		
CRQL		

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

ne comer LDC #. 24089H2 SDG#:

VALIDATION FINDINGS WORKSHEET

2nd Reviewer: O Reviewer: 🚽

Laboratory Control Samples (LCS)

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

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

Was a LCS required? Y N N/A

Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

Qualifications 17/P due (no Leso Associated Samples RPD (Limits) LCSD %R (Limits) 136 (76-133 LCS %R (Limits) Compound Lab ID/Reference 0260402 01/01/10 Date #

LDC #: 24089H2/

# 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 M/A Were t

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 Associated Samples Qualifications	A11 J/A0	below the Pal 1		compounds reported All JR dat (K)						
	All compounds re	below the PQL		M chupound	Day E Mpc					
Sample ID										
*										

Comments: See sample calculation verification worksheet for recalculations