

# Laboratory Data Consultants, Inc.

7750 El Camino Real, Ste. 2L Carlsbad, CA 92009

Phone 760.634.0437

Web www.lab-data.com

Fax 760.634.0439

Northgate Environmental Management, Inc.

December 8, 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 October 27, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

# LDC Project # 24445:

SDG#

Fraction

G0H120523, G0H280490, G0I020523, G0I140549

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
- 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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LDC #: <u>24445</u> SDG #: <u>G0H120523</u>, <u>G0H280490</u>, <u>G0I020523</u>, <u>G0I140549</u> Page: 1 of I Reviewer: <u>JE</u> 2nd Reviewer: BC

# Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I: Completeness				
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	x		<u> </u>	
III. EDD Lab Anomalies				
Were EDD anomalies identified?		Х		
If yes, were they corrected or documented for the client?		•	Х	See EDD_discrepancy_ form_LDC24445_120710.doc
IV. EDD Delivery				
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: August 9, 2010

**LDC Report Date:** December 7, 2010

Matrix: Soil/Water

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0H120523

Sample Identification

SSAQ4-08-10BPC\*\* SSAQ4-08-10BPC\_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010

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

## Introduction

This data review covers 4 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 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 2.0 pg/L 3.7 pg/L	All water samples in SDG G0H120523
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	All soil samples in SDG G0H120523

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		Reported	Modified Final
	Compound	Concentration	Concentration
EB-08092010	1,2,3,6,7,8-HxCDD	1.6 pg/L	1.6U pg/L
	1,2,3,7,8,9-HxCDD	1.2 pg/L	1.2U pg/L
	1,2,3,4,6,7,8-HpCDD	2.4 pg/L	2.4U pg/L
	OCDD	4.3 pg/L	4.3U pg/L
	1,2,3,7,8-PeCDF	11 pg/L	11U pg/L
	2,3,4,6,7,8-HxCDF	2.6 pg/L	2.6U pg/L
	1,2,3,7,8,9-HxCDF	2.5 pg/L	2.5U pg/L
	1,2,3,7,8,9-HpCDF	10 pg/L	10U pg/L

Sample EB08092010 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
EB08092010	8/9/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,4,6,7,8-HyCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	1.1 pg/L 1.6 pg/L 1.2 pg/L 2.4 pg/L 4.3 pg/L 5.7 pg/L 11 pg/L 6.9 pg/L 14 pg/L 11 pg/L 2.6 pg/L 2.5 pg/L 2.7 pg/L 48 pg/L	All soil samples in SDG G0H120523

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

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

# VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAQ4-08-1BPC	<sup>13</sup> C-OCDD	35 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAQ4-08-5BPC	<sup>13</sup> C-OCDD	21 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

# 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
SSAQ4-08-10BPC**	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)	Р
S\$AQ4-08-10BPC_FD S\$AQ4-08-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	Р
SSAQ4-08-5BPC	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 G0H120523	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 G0H120523	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 SSAQ4-08-10BPC\*\* and SSAQ4-08-10BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concent	ration (pg/g)				
Compound	SSAQ4-08-10BPC	SSAQ4-08-10BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	3.6	2.7	29 (≤50)	-	-	-
1,2,3,7,8-PeCDD	23	16	36 (≤50)	<del>-</del>	-	
1,2,3,4,7,8-HxCDD	15	12	22 (≤50)	<del>-</del>	-	-
1,2,3,6,7,8-HxCDD	13	25	63 (≤50)	*	J (all detects)	Α
1,2,3,7,8,9-HxCDD	17	12	34 (≤50)		•	<u>.</u>
1,2,3,4,6,7,8-HpCDD	120	110	9 (≤50)	-	-	-
OCDD	190	180	5 (≤50)	-	-	-
2,3,7,8-TCDF	120	99	19 (≤50)	٠	-	-
1,2,3,7,8-PeCDF	450	380	17 (≤50)	-	-	<u>-</u>
2,3,4,7,8-PeCDF	220	190	15 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	670	650	3 (≤50)	-	· <u>-</u>	-
1,2,3,6,7,8-HxCDF	670	590	13 (≤50)	-		-
2,3,4,6,7,8-HxCDF	150	150	0 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	110	98	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	2700	2700	0 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1100	1100	0 (≤50)	-	-	-
OCDF	12000	13000	8(≤50)	<u>-</u>	-	-

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H120523

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H120523	SSAQ4-08-1BPC SSAQ4-08-5BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	₽	Internal standards (%R) (i)
G0H120523	SSAQ4-08-10BPC**	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	Ρ	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-10BPC_FD SSAQ4-08-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-5BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H120523	SSAQ4-08-10BPC SSAQ4-08-10BPC_FD	1,2,3,6,7,8-HxCDD	J (all detects)	А	Field duplicates (RPD) (fd)

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H120523

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H120523	EB-08092010	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	1.6U pg/L 1.2U pg/L 2.4U pg/L 4.3U pg/L 11U pg/L 2.6U pg/L 2.5U pg/L 10U pg/L		bl

# Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H120523

No Sample Data Qualified in this SDG

# **Tronox Northqate Henderson**

LDC #: 24445A21	VALIDATION COMPLETENESS WORKSHEET
SDG #: G0H120523	Stage 2B/4
Laboratory: Test America	<u> </u>

	Date:	12/	01/1
	Page:_	_/of_	_
	Reviewer:	7	<u> </u>
2nd	Reviewer:	1	
		7	

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

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

	Validation Area		Comments
l.	Technical holding times	Δ	Sampling dates: 8 9 10
11.	HRGC/HRMS Instrument performance check	Δ	
III,	Initial calibration	Δ	
IV.	Routine calibration# <del>CV</del>	4	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	2	client speciful
VII.	Laboratory control samples	Ą	LCS 1
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	يىي	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	Δ	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	D = 1,2
XV.	Field blanks	SW	EB = S

Note:

A = Acceptable N = Not provided/applicable

ND = No compounds detected

D = Duplicate TB = Trip blank

SW = See worksheet

R = Rinsate FB = Field blank

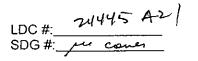
EB = Equipment blank

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

+ water

,	JUIL T WAL					
1 1	SSAQ4-08-10BPC**	村!	0230135	21	31	1
2	SSAQ4-08-10BPC_FD	122	0228254	22	32	2
3 1	SSAQ4-08-1BPC	13		23	33	3
4	SSAQ4-08-5BPC ✓	14		24	34	4
5 7	EB-08092010	15	·	25	35	5
6		16		26	36	6
7		17		27	37	7
8		18		28	38	8
9		19		29	39	9 .
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Notes:		



# **VALIDATION FINDINGS CHECKLIST**

	Page:_	1	_of	2-
	Reviewer:		17	
2nd	Reviewer:		0/	
			7	

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	7	:		
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) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	_			
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound $\geq$ 2.5 and for each recovery and internal standard $\geq$ 10?				
IV: Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	_			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?				
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks	13 61 14 FC 25 18			
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	2500 2500		ovala Mesa	
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		NG-GS.		
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 #: 24445 AZ | SDG #: \_\_\_\_\_\_\_\_\_

# **VALIDATION FINDINGS CHECKLIST**

Page:	<u>2</u> of	2_
Reviewer:		5_
2nd Reviewer:		

				v
VIII. Regional Quality Assurance and Quality Control	<u>.</u>		<del>.</del>	
Were performance evaluation (PE) samples performed?	_		_	
Were the performance evaluation (PE) samples within the acceptance limits?	<u>E</u> (\$1. ~ c)	ক্রান্ত্রনার করে করে করে করে করে করে করে করে করে কর		
IX: Internal/standards	<u> </u>			
Were internal standard recoveries within the 40-135% criteria?	ļ.			
Was the minimum S/N ratio of all internal standard peaks ≥ 10?		1	<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?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	/	<u> </u>		
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 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?	Ļ		<u> </u>	
XI. Compound quantitation/CRQLs			( (/ :	
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	_			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	_			
XII System performance				
System performance was found to be acceptable.		-		
XIIII: Overall assessment of data				
Overall assessment of data was found to be acceptable.	-			
XIV:Fieldiduplicatess = 12				Marke
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XVdField/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)

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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2nd Reviewer:\_ Reviewer: FT

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?

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

Was the method blank contaminated? X M N/A V N/A

Blank analysis date: 8/21/10 Blank extraction date: 8 16 10

Associated samples:

all water

Sample Identification 49/4 2.6/4 2.5/4 4.3/4 7.4 1.2/11 2 5 2 0228254-4B Blank ID 13. ý c , 2.5 ڊ ن 9,0 らな 7 4 7.7 6: Š Conc. units: pg/ Compound JŁ Σ 2 P H 0 دنا J 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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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?

 $\sqrt{N N/A}$  Was the method blank contaminated? Blank extraction date:  $\sqrt{|16|}$  b Blank analysi N N/A

Blank analysis date: ≼ 2011∪

Associated samples:

75x

Sample Identification 9.27 ゞ 0.435 4.0 0.48 ò <u>و</u> ة ゲゲ 0228363-MB Blank ID 0.004 0.006 0.087 0.16 0.12 0.70 0, &*O* Conc. units: pa a Compound ۵ প্ত W ۰ U.  $\sigma$ 

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# 24445 A2/

# VALIDATION FINDINGS WORKSHEET Field Blanks

2nd Reviewer: Page:\_ Reviewer: FT

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

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# 24445 A2/

# VALIDATION FINDINGS WORKSHEET Internal Standards

Reviewer: FT 2nd Reviewer: Q

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?

XVXX Y/N N/A

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

ন ত **Check Standard Used** Qualifications **OLA** 9/ [N] Recovery Standards 40-125 % Recovery (Limit: 40-135%) <sup>13</sup>C-123789-HxCDD <sup>13</sup>C-1,2,3,4-TCDD 7 Д d d Check Standard Used Internal Standard Lab ID/Reference Internal Standards 13C-1,2,3,4,6,7,8-HpCDD <sup>13</sup>C-1234678-HpCDE <sup>13</sup>C-1 2 3 6 7 8-HxCDD <sup>13</sup>C-123678-HxCDE <sup>13</sup>C-1,2,3,7,8-PeCDE 13C-12378-PeCDD 13C-2 3 7 8-TCDD <sup>13</sup>C-2,3,7,8-TCDF Date

# Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

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".

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 footong the compound?

		7	<del>                                     </del>	T	<del></del>	T		Т	T	T	<del></del>	7	$\overline{}$	Т	Т	T	T
essary).		Adamironions	(de) stanta Vic				JK detects (k)		1 Par					*			
to reflect all sample dilutions and dry weight factors (if necessary).	Associated Samulas	All	The second secon				All				7 2		7				
were adjusted to reflect all sample difutior	Finding	All compounds reported below POL					All compounds reported as EMPC		x1d cas conce		<b>→</b>		>				
Soulpoully qualification and CRQLS were adjusted	Pamon Sample ID								0, P, Q		<i>و</i> , ه		Ø				
	Date															-	
	*	·															

Comments: See sample calculation verification worksheet for recalculations

LDC#:24445A21

# **VALIDATION FINDINGS WORKSHEET** Field Duplicates

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nd Reviewer: 7
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METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

(ta)

	Concentrat	ion (nala)	%RPD	(ng/g)	(ng/g)	Qualifications
Compound	1		%KPD ≤50	(pg/g) Difference	(pg/g) Limits	(Parent Only)
Compand	1	2		Difference	Lillius	(Parent Only)
Α	3.6	2.7	29			
В	23	16	36		,	
c	15	12	22			
٥	13	25	63			J/A dot
ш	17	12	34			·
F	120	110	9			
G	190	180	5			
н	120	99	19			
1	450	380	17			
J	220	190	15			
К	670	650	3			
L	670	590	13			
M	150	150	0			
N	110	98	12			
0	2700	2700	0			
Р	1100	1100	0			
Q	12000	13000	8		•	

V:\FIELD DUPLICATES\24445A21.wpd

1DC#: 24445A2/ SDG#: 210 count

# VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: /of/ Reviewer: /=7\_ 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:

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

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

A<sub>is</sub> = Area of associated internal standard C<sub>is</sub> = Concentration of internal standard X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#1	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF	RRF	%RSD	%RSD
-	164	Chorle	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.003	1.003	1.0226	1.03%	3./3	3.13
		::	2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.172	2/-/	06401	0610-1	6:11	6://
		· · · ·	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.130	1-130	1.083/	1:80.1	20.3	なら
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.079	6601	6/10-1	1.07/3	X & 1	PE-/
	-		OCDE (3C,OCDD)	1.467	1.467	1.4726	1.4726	3.12	3.12
2	1451	01/27/1	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	2220	CT8-0	18-0	28-0	14.2	7:3/
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	0.957	0.957	0.93	0.93	/3.2	(3-3)
		<del></del>	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.107	1.107	61.1	61.1	_s·e/	5-61
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.020	1.026	1.07	1.07	/3.6	13.6.
	1		OCDE (13C. OCDD)	1.445	534.1	٠.٤٧	/-کئ	/ × · /	//.//
ြ	7471	01191/8	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.00187	181001	1.00395	1.00397	4.19465	561-4
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.16617	(1991.1	1.15763	1-15763	8.1039	101.8
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1-14627	1.14627	761711	96191.1	1590E.8	8-306
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	82001	1.08648	1.10877	61.10879	6.01727	6.017
			OCDF (1°C-OCDD)	75865.1	754 KS-1	7.58313	E1885-1	8. 41 szs <sup>-</sup>	715-8

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

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# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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Page:	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>k</sub> = Area of compound, C<sub>k</sub> = Concentration of compound, S = Standard deviation of the RRFs,

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

 $\label{eq:RF} $RF = (A_y)(C_s)/(A_{ls})(C_x)$ average $RF = 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 ( むろう std)	RRF ( 0% 3std)	%RSD	%RSD
-	ICAL	1/20/10	7/2 <i>C/IO</i> 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	250-1	1.052	1.020		3.32	3.32
	pBirs		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)						
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)						
			1,2,3,6,7,8-HxCDD (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)						
ო			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 (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 (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.

SDG#: 24 com

# VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: /of / Reviewer: /F7 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 =  $(A_{\lambda})(C_{k_{\lambda}})/(A_{k_{\lambda}})(C_{\lambda})$ 

Where: ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A<sub>x</sub> = Area of compound, A<sub>x</sub> = Concentration of compound, C<sub>x</sub>

 $A_{is}$  = 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)	Q%	Q%
_	dev 17:33	01/08/8	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.05	0.96	26.0	8.7	1.3
	DBN		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)					
			OCDE (13C-OCDD)					
2	aen 8:40	01/21/8	8/24/10 2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	22.0	28.0	85.0	0	б
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	7.56.0	0.87	0.87	2.6	しょ
			1,2,3,6,7,8-HXCDD (13C-1,2,3,6,7,8-HXCDD)	1.107	601	1.03	サ・C	4.0
		<del></del>	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.026	1.07	1.07	4.2	4.7
			ocpe (¹c-ocpp)	1.445	1.58	1. CB	9.2	4.2
53	aru 21:35	01/01/8	8/20/O 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1. 00/87	hhоою 'o	0.9004	10.7	1001
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.16617	1.0963	1.0963	0.9	6-0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.14627	50h91-1	1.16495	ン・	9.1
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.08048	1.080709	1.0879	0.5	5-0
			OCDF (13C-OCDD)	CS4827	1.44/162	1.44/62	6.9	6.9

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

LDC# 74445A2

# Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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Page:	Reviewer:	2nd Reviewer:

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

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

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

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF  $A_x$  = Area of compound,  $A_y$  = Concentration of compound,  $C_y$  = Concentration of compound, 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)	%D	Q%
Ψ-	Oct 22:09 8/21/10	CIlIA	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	6.003	1.06	1.06	5.3	٤.٦
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	7.14s	201	1.07	6.2	4.9
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	0.61.1	/se:/	\mathcal{K}./	13.2	7.5/
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	620.1	80.1	1.08	0.5	0.5
			OCDE (13C-OCDD)	1.467	1.67	1.69	14.9	6.41
2			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)					
			OCDE (13C-OCDD)					
က			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)					

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.

SDG #: 1447 172/ SDG #: 644 1944

# VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

rage: \_\_or\_\_ 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 = 8

Where: SSC = Spiked sample concentration SA = Spike added

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

6958260

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	S	lke	Spiked 5	Sample	\$J1	S	I CSD	າກ	I CS/I CSD	CSD
Compound	Ad (09%	Added (now and )	Concentration (アペール)	tration	Percent Recovery	ecovery	Percent Recovery	acovery	RPD	Q
	0 )	U CSD	108	l Csn	Raportad	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.02	4 2	(-1.)	MΑ	` <i>\$</i>	γ,				
1,2,3,7,8-PeCDD	001	•	ر. لاد	-	12	1.8				
1,2,3,4,7,8-HxCDD	0.01		93.6		pb	þb				
1,2,3,4,7,8,9-HpCDF	0 9 1		الا. ما		10	٦٩				
ocpF	002	7	169	<del></del>	hχ	٦ لا	Z A Z			
-									,	

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

	Analyte	HPCDF HPCDF (S) HPCDF HPCDD HPCDD HPCDD (S) HPCDD (S) HPCDD (S)	OCDF OCDF OCDD OCDD OCDD (S) OCDP (S) PFK	
	Elemental Composition		C <sub>12</sub> <sup>20</sup> Cl <sub>7</sub> <sup>37</sup> ClO C <sub>12</sub> <sup>20</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> <sup>20</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O <sup>10</sup> C <sub>12</sub> <sup>20</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O <sup>10</sup> C <sub>12</sub> <sup>20</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O C <sub>12</sub> <sup>20</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	
	Ion ID	M+4 M+2 M+2 M+2 M+4 M+4 M+4 M+4	M M + 4 M M + 4 M M + 4 M M + 4 M M + 4 C C K	
	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	ເດ 	·
	Analyte	TCDF TCDF (S) TCDD (S) TCDD TCDD TCDD TCDD (S) HCDPE PFK	PecDF PecDF (S) PecDF (S) PecDD PecDD PecDD PecDD PecDD PecDD PecDD PecDD (S) PecDD (S) PecDD (S)	HXCDF HXCDF HXCDF (S) HXCDF (S) HXCDD HXCDD HXCDD (S) HXCDD (S)
	Elemental Composition	C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>1</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>1</sub> O <sup>16</sup> C <sub>2</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O <sup>16</sup> C <sub>2</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O <sup>16</sup> C <sub>2</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O C <sub>12</sub> H <sub>4</sub> <sup>36</sup> Cl <sub>2</sub> O	C <sub>1</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>2</sub> Cl <sub>2</sub> H <sub>3</sub> <sup>3</sup> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>2</sub> Cl <sub>3</sub> Cl <sub>3</sub> Cl <sub>2</sub> Cl <sub>3</sub>	C <sub>1</sub> , H, wCl, vClO C <sub>1</sub> , H, wClO C
	Ci noi	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+2 M+4 M+2 M+2 LOCK	M M M M M M M M M M M M M M M M M M M
Accimpto mose(8)	Accided III 435	303.9016 305.8987 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	339,8597 341,8567 351,9000 353,8970 355,8546 357,8516 367,8949 369,8919 409,7974 [354,9792]	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 445.7555
Descriptor		-	N	က

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 <sup>10</sup>C = 13.003355 F = 18.9984

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

S = internal/recovery standard

LDC #:_	24	445AZ	-/
		cover	/

Sample Calculation Verification

Page:_		
Reviewer:	F	7
2nd reviewer:	0	
_	7	

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

NY	N/A
Y/N	N/A

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

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

Concentration =  $(A_k)(I_k)(DF)$  $(A_k)(RRF)(V_o)(\%S)$ 

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. #/ 2,3,7,8-7CPD

Conc. = (4277440), (2000)) ( ) 2182/7700) ( 1.14 ) ( 10.36) ( 0.4/9)

= 3.6 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#1 2, 3, 7, 8- TCDF	(0B225)		
					·
		= 202503800 (	2000		
		= 202503800 ( (334668000)(1	06)(10.36)	0.919	
		= 120	pg/g		
			ļ		
					1
					-
$\vdash$	<u></u>				
<b> </b>	<del></del>				
	-			<del> </del>	
			<del> </del>		-

# Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

August 26, 2010

LDC Report Date:

December 8, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0H280490

# Sample Identification

BDT-2-S-20-10BPC

BDT-2-S-20-12BPC

BDT-2-S-20-14BPC\*\*

BDT-2-S-20-2BPC

BDT-2-S-20-4BPC

BDT-2-S-20-6BPC

BDT-2-S-20-6BPC FD

BDT-2-S-20-8BPC

BDT-2-S-15-10BPC

BDT-2-S-15-12BPC

BDT-2-S-15-14BPC\*\*

BDT-2-S-15-2BPC

BDT-2-S-15-4BPC

BDT-2-S-15-6BPC

BDT-2-S-15-8BPC

BDT-2-S-15-2BPC FD

BDT-2-S-20-10BPCMS

BDT-2-S-20-10BPCMSD

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

## Introduction

This data review covers 18 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 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
0257312-MB	9/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.081 pg/g 1.4 pg/g 0.089 pg/g 0.23 pg/g	All samples in SDG G0H280490

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. Although the MS/MSD percent recovery (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

# VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
BDT-2-S-20-10BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 37 (40-135)	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
BDT-2-S-20-14BPC**	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 39 (40-135)	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
BDT-2-S-15-10BPC	<sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	38 (40-135)	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	₽

# 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
BDT-2-S-20-2BPC BDT-2-S-20-4BPC	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
BDT-2-S-15-2BPC	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
BDT-2-S-15-4BPC BDT-2-S-15-2BPC_FD	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
All samples in SDG G0H280490	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 G0H280490	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 BDT-2-S-20-6BPC and BDT-2-S-20-6BPC\_FD and samples BDT-2-S-15-2BPC and BDT-2-S-15-2BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concent	ration (pg/g)				
Compound	BDT-2-S-20-6BPC	BDT-2-S-20-6BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	1.4	1.6	13 (≤50)	-	-	<u>.</u>
1,2,3,7,8-PeCDD	4.5	5.1	12 (≤50)	-	-	_
1,2,3,4,7,8-HxCDD	3.8	4.3	12 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	7.0	7.9	12 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	5.6	6.6	16 (≤50)	-	_	-
1,2,3,4,6,7,8-HpCDD	22	25	13 (≤50)	-	-	-
OCDD	49	64	27 (≤50)	-	-	-
2,3,7,8-TCDF	33	38	14 (≤50)	-	-	-
1,2,3,7,8-PeCDF	53	60	12 (≤50)	-	-	-
2,3,4,7,8-PeCDF	29	33	13 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	96	100	4 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	65	69	6 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	21	25	17 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	9.8	11	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	280	310	10 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	120	120	0 (≤50)	-		
OCDF	590	660	11 (≤50)	-	-	-

	Concent	ration (pg/g)		Difference			
Compound	BDT-2-S-15-2BPC	BDT-2-S-15-2BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
2,3,7,8-TCDD	12	11	9 (≤50)	-	-	-	
1,2,3,7,8-PeCDD	42	40	5 (≤50)	-	-	-	
1,2,3,4,7,8-HxCDD	41	34	19 (≤50)	-	-	-	
1,2,3,6,7,8-HxCDD	68	70	3 (≤50)	-	-	-	
1,2,3,7,8,9-HxCDD	55	56	2 (≤50)	-	-	-	
1,2,3,4,6,7,8-HpCDD	230	220	4 (≤50)	-	-	-	
OCDD	620	540	14 (≤50)	<del>-</del>	-	_	
2,3,7,8-TCDF	300	270	11 (≤50)	-		-	
1,2,3,7,8-PeCDF	490	440	11 (≤50)	-	-		
2,3,4,7,8-PeCDF	270	240	12 (≤50)	-	-	-	
1,2,3,4,7,8-HxCDF	920	870	6 (≤50)	-	-	-	
1,2,3,6,7,8-HxCDF	550	570	4 (≤50)	_	-	-	
2,3,4,6,7,8-HxCDF	170	180	6 (≤50)	-	-	-	
1,2,3,7,8,9-HxCDF	86	100	15 (≤50)	-	-	-	
1,2,3,4,6,7,8-HpCDF	2800	2400	15 (≤50)	-	-	-	
1,2,3,4,7,8,9-HpCDF	1100	1000	10 (≤50)	_	-		
OCDF	6300	7000	11 (≤50)	-	-	-	

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H280490

		1	<u>-</u>	<u> </u>	
SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-14BPC**	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)
G0H280490	BDT-2-S-15-10BPC	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280490	BDT-2-S-20-2BPC BDT-2-S-20-4BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-2BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-4BPC BDT-2-S-15-2BPC_FD	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)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-2BPC BDT-2-S-15-6BPC BDT-2-S-15-6BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC_FD BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	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 G0H280490

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H280490

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** T

LDC #: 24445B21	VALIDATION COMPLETENESS WORKSHEE
SDG #: G0H280490	Stage 2B/4
Laboratory: Test America	<u> </u>
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Date:	12/01/10
Page:_	<u>/</u> of/
Reviewer:	<u> </u>
2nd Reviewer:	<u></u>
	9

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 26 10
II.	HRGC/HRMS Instrument performance check	Δ	
111.	Initial calibration	Д	
IV.	Routine calibration <del>/ICV</del>	4	
V.	Blanks	پىي	
VI.	Matrix spike/Matrix spike duplicates	يىرى	
VII.	Laboratory control samples	A.	w
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	ىسى	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	ريري	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	5~	D=6,7 18,14
XV.	Field blanks	2	

Note:

A = Acceptable

N = Not provided/applicable

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank

SW = See worksheet

FB = Field blank

EB = Equipment blank

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

	<u> </u>						
1	BDT-2-S-20-10BPC	11	BDT-2-S-15-14BPC**	21	0259197	31	"
2	BDT-2-S-20-12BPC	12	BDT-2-S-15-2BPC ()	22	025 7312	32	
3	BDT-2-S-20-14BPC**	13	BDT-2-S-15-4BPC	23		33	
4	BDT-2-S-20-2BPC	14	BDT-2-S-15-6BPC	24		34	
5	BDT-2-S-20-4BPC	15	BDT-2-S-15-8BPC	25		35	
6	BDT-2-S-20-6BPC	16	BDT-2-S-15-2BPC_FD	26		36	
7	BDT-2-S-20-6BPC_FD	17	BDT-2-S-20-10BPCMS	27	<u>.</u>	37	
8	BDT-2-S-20-8BPC	18	BDT-2-S-20-10BPCMSD	28		38	
9	BDT-2-S-15-10BPC	19		29		39	
10	BDT-2-S-15-12BPC	20		30		40	

Notes:			
	 		•

### LDC #: 24445 B2/ SDG #: pu comes

### VALIDATION FINDINGS CHECKLIST

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		<i>:</i>		
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?			<u> </u>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?			<u> </u>	
Was the mass resolution adequately check with PFK?			<u> </u>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration		···	<del></del>	
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?	_			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?				
IV: Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?				
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?				
Did all routine calibration standards meet the Ion Abundance Ratio criteria?		لببا		
V. Blanks		100 M 100 M		
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?		************		
Mi. 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 s Laboratory control samples		MESSE MESS		
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 #:_	24445 B2
SDG #:_	per comes

### **VALIDATION FINDINGS CHECKLIST**

Page:2_of²	_
Reviewer:	
2nd Reviewer:	

		:		
VIII. Regional Quality Assurance and Quality Control	<u>/</u>	1	· 	· · · · · · · · · · · · · · · · · · ·
Were performance evaluation (PE) samples performed?	ļ	ļ		·
Were the performance evaluation (PE) samples within the acceptance limits?				45.4
IX:/Internal standards			7 (26.1) \$1.3 /	
Were internal standard recoveries within the 40-135% criteria?				
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	/	<u> </u>	<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?				
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?	_			
All System performances				
System performance was found to be acceptable.				
XIII:Overall assessment of data			artika - Sila	
Overall assessment of data was found to be acceptable.				
XIV/stield:duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			<u> </u>	
Welfield 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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### VALIDATION FINDINGS WORKSHEET Blanks

2nd Reviewer: Reviewer: FT

\* EMPC

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

Blank extraction date: 에너티

Blank analysis date: 9 26 10

(= | | Associated samples:\_\_

									-					
								<b>,</b>						
	Sample Identification													
<b>!</b> •	Sample													
								,						
_														
		MPS 5X	504.0	L	244.0	51.1								
\ \{\frac{1}{4}}	Blank ID	035 7312- MPS 5X	6.08 ⊀	1.4 *	₩ 680.0	6.23								
Conc. units: 129 a	O. O. Compound		<b>1</b>	p	Ф						,			

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# 24445 B2/

### 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". N N/A

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

MS/MSD. Soil / Water.

N/A

Y/N/N/A

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

#	Date	OI OS/WSW	Compound	MS %R (Limits)	s mits)	8	(5	RPD (Limits)	Associated Samples	Qualifications
П		X1 + L1	4	1)0/0 T	٦ +	0878 %		ware ( )	1	NO grad LESIN
			butside.		( <del>),</del>	)	(	( )	-	Λ
П					(	)	^	( )		
$\Box$				)	(	)	^	( )		
				)	)	)	)	( )		
				)	(	)	)	( )	,	
				)	(	)	(	( )		
				)	1	)				
				)	)	)	^	( )		
				)	(	)	(	( )	:	
				)	(	)	^	( )		
				)	(	)	^	( )		
				)	(	)	^	( )		
				)	)	)	^	( )		
				<b>—</b>	`	)		( )		
								( )		
П				)	(	)	^	( )		
				<u> </u>	^	Ú	^	( )		
				`		)		( )		
				`	^	)		( )		
				)	(	)	(	( )		
					^	V		( )		
			T	)	(	)	`	( )		

## LDC# 24445B2/

### VALIDATION FINDINGS WORKSHEET Internal Standards

Page: /of/ Reviewer: F 2nd 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)

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

#

Check Standard Used Qualifications 3 Recovery Standards 하 교소 % Recovery (Limit: 40-135%) <sup>13</sup>C-1.2.3.4-TCDD 0 なる 30 'n 27 6 Check Standard Used Internal Standard W J ত J Lab ID/Reference Internal Standards M 3 9 13C-2.3.7.8-TCDE Date Y N N/A

ď

ž

13C-1,2,3,4,6,7,8-HpCDD <sup>13</sup>C-1234678-HpCDE <sup>13</sup>C-1 2 3 6 7 8-HxCDD <sup>13</sup>C-1 2 3 6 7 8-HxCDE

<sup>13</sup>C-12378-PeCDD <sup>13</sup>C-1 2 3 7 8-PeCDE 13C-2 3 7 8-TCDD

13C-1,2,3,7,8,9-HxCDD

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# VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: of Reviewer: FT

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).

Associated Samples Qualifications				All JK defects (k)		4, 5 11P det (e)		12		13, 16		
Finding	All compounds reported below PQL			All compounds reported as EMPC		Exid cal Range	)	->		->		
 GOVADO GENERAL SERVICES						Ø		H B, P, G	-	# g, &		
# Date	-											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24445B21

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

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

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

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

(	fd	)
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	Concentra	tion (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	6	7	≤50	Difference	Limits	(Parent Only)
Α	1.4	1.6	13	-34c		
В	4.5	5.1	12			
С	3.8	4.3	12			
D	7.0	7.9	12			
E	5.6	6.6	16			
F	22	25	13		·	
G	· 49	64	27			
н	33	38	14			
ı	53	60	12			
J	29	33	13			
к	96	100	4			
L	65	69	6			
М	21	25	17			
N	9.8	11	12			
0	280	310	10			
Р	120	120	0			
Q	590	660	11			

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LDC#: 23906B4

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

Page:_	<u>/of_/</u>
Reviewer:	FT
2nd Reviewer:	0
	7

METHOD: Metals (EPA Method 6020/7000)

AN NA Y N NA

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

	Concentra	tion (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	12	16	≤50	Difference	Limits	(Parent Only)
А	12	11	9 .			
В	42	40	5			
С	41	34	19			
D	68	70	3			,
E	55	56	2			
F	230	220	4			
G	620	540	14			
Н	300	270	11		,	
1	490	440	11			
J	270	240	12			
К	920	870	6			
L	550	570	4			
М	170	180	6			
N	86	100	15			
o	2800	2400	15			
Р	1100	1000	10			
Q	6300	7000	11			

V:\FIELD DUPLICATES\24445B21.wpd

LDC#: 24445B 2/ SDG#: 444 court

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of 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:

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

 $A_{s} = Area \ of \ compound, \\ C_{s} = Concentration \ of \ compound, \\ S = Standard \ deviation \ of \ the \ RRFs, \\ X = Mean \ of \ the \ RRFs$ 

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)		RRF (@\$3 std)	%RSD	%RSD
L	DB 255 1CAL	1	H	1.052	1.056	1.02	40.1	332	3.32
			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 ('3C-1,2,4,6,7,8,-HpCDD)						
			OCDE (19C-OCDD)						
~	7851	01/08/8	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.169	1.169	1.2609	1.2009	5,52	25.25
			2,3,7,8-TCDD ('3C-2,3,7,8-TCDD)	1,22,2	クスン	1.2887	1.2887	004	00-h
<u> </u>			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	7.505	7:207	1.452	1.452	5-2-7	5.27
<u> </u>			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	081.1	081.1	1.2654	1.2634	29.5	79-5
			OCDE (13C.OCDD)	1-892	768-1	1.9979	1.9979	6-95	6.9
က			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 ( <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.

LDC #. 24445 B2/ ee cons SDG#:

### Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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

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

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A<sub>x</sub> = Area of compound, A<sub>s</sub> C<sub>x</sub> = Concentration of compound, C<sub>s</sub> Where:

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

1								
					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	<b>Q</b> %	Q%
-	11.12 100	01/1/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.050	61-1	61:1	6.6/	6.61
	posse		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)					
Í			OCDE (13C-OCDD)					
2	CON 27:42 9/28/10	01/24/6	2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)	1.169	01.1	01.1	6.3	6.3
		,	2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)	7.25	61.1	1.14	€.6	9.3
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	701	8/:1	31.1	0.1	0.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	081:1	101	1.1	2.3	6.4
			OCDE (13C-OCDD)	768.1	1.77	1.77	9.9	9.9
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 (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

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

2nd Reviewer: Reviewer: Page:

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)

+ | 8

Recalculated <u>م</u> RPD 3 7 Reported 72 n e 7 RPD 7 9 Matrix Spike Duplicate Recalc 200 Percent Recovery % √ 9 757 23 Reported K W tes  $\frac{\mathcal{Q}}{\mathcal{O}}$ Y Z Recalc Percent Recovery 9 135 7 B 2 Matrix Spike Reported 102 13.  $\mathcal{C}_{\mathcal{O}}$ 4 30 0821 アイン 47.4 89.8 MSD 308 Spiked Sample Concentration 4919 17.3 145 8 8 8 79.8 섥 Sample Concentration 3 ة 0 0.36 7 ナニ \_\_\_\_ <u>ه</u> 4 ٩ dSM 20 23 Spike Added 829 <u>-</u> Sol 6 성 77 1,2,3,4,7,8,9-HpCDF 1,2,3,4,7,8-HxCDD Compound 1,2,3,7,8-PeCDD 2,3,7,8-TCDD 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.

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## Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

| |-|rage: 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 | \* 2/(LCS + LCSD)

215120

LCS ID: \_\_

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery.

	Sc	Ke	Spiked 5	ample	SJI	S	1 ຕຣກ	O.	USJ ISJ I	csn
Compound	A 67	Added (%)	Concentration (pg   み)	tration	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	۵
	) ( ) (	/ U	0,01	l csn	Reported	Recalc	Reported	Racaic	Reported	Recalculated
2,3,7,8-TCDD	0.0r	λV	3.L1	NΑ	85	**				
1,2,3,7,8-PeCDD	001	~	95.6		93	ભુ				
1,2,3,4,7,8-HxCDD	0 01		95.96		96	36				
1,2,3,4,7,8,9-HpCDF	0 이		4·h8		μB	8.4				
OCDF	2002	<del>-&gt;-</del>	251	<b>→</b>	2	72	¥ 2			
-										

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

# ions monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ol no!	Elemental Composition	Analyte	non-ultiple				
-	200 004		nomeodine -	Allalyte	Descriptor	Accurate Mass <sup>(4)</sup>	lon ID	Elemental Composition	Analyte
-	305.8987	¥ M	C <sub>12</sub> H <sub>3</sub> *C <sub>1</sub> O	TCDF	4	407.7818	M+2	C. H <sup>36</sup> Cl 37ClO	0001
	315,9419	Σ		100F		409.7788	M+4	C.H.*CI.*CI.O	TOOG I
	317,9389	M+2	13C1,H,3CL,3CIO	100F(8)		417.8250	Σ	13C <sub>12</sub> H <sup>az</sup> Cl,O	HPCDF (S)
	319,8965	Σ	C, H, SCI, C,	(S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> CI <sub>3</sub> 37CIO	HPCDF
	321.8936	M+2	C.H.**CI,**\C_10,	TCDD		423.7707	M+2		HpCDD
	331.9368	≅	13C, H, 35C, C,	TCDD (S)		425.7.37	M+4	C <sub>12</sub> H <sup>32</sup> Cl <sub>5</sub> 3 <sup>2</sup> Cl <sub>2</sub> O <sub>2</sub>	Hecdo
	333,9338	M+2	13C; H, acl, aclo,	(S) CODE		450.6169	M+2	_	HeCDD (S)
	375,8364	M+2	C, H, "Cl, "Clo	HYCOPE HYCOPE		437.8140	M+4	_	HeCDD (S)
<del></del>	[354.9792]	LOCK	, E. C.	PFK		4/9./163	¥+ 4+		
				<u>:</u>		[430.97.28]	LOCK	· · · · ·	PFK
٥	330 8507								
l	341.8567	7+N	0 0/5/12/20	PeCDF	ນ	441.7428	M+2	0.000	1
	351 9000	† C		PeCDF		443.7399			OCDF
	353 8970	7+1/1	5 7 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	PeCDF (S)	-	457.7377			OCDF
	25E 8E46	4+1	0,20, 1, 20, 20, 20, 20, 20, 20, 20, 20, 20, 20	PecDF (S)		459 7348			ocoo
	357 0616	M+2	C <sub>12</sub> H <sub>3</sub> *C  <sub>4</sub> *ClO <sub>2</sub>	PecDD		469.7780			0000
	007.0010	M+4	C <sub>12</sub> H <sub>3</sub> 3Cl <sub>3</sub> O <sub>2</sub>	PecDD		471 7750	M+12	-	(s) agoo
	307,0348	M+2	13C12H3*C12,	Pecdo (S)		513 677E			(s) agoo
	369.8919	M+4	13C1, H, 43C1, 37C1, O,	Pecho (S)		700,00701		_	DCDPE
	409.7974	M+2	C,H,*Cl,*ClO,	HnCDPE		[422.32/0]	Ž O Z		PFK
	[354.9792]	LOCK	, E	PFK					
8	373.8208	M+2	1 C	1.00					
_	375.8178	M+4	C 18 0 30 0	TXCDT					
	383,8639	2		HXCDF					
	385.8610	₩+2	1212 CO	HXCDF (S)					
	389.8156	M + 1		HXCDF (S)			•		
	391.8127		C T 3C 3C C	HXCDD	_				
•	401.8559		15. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	HXCDD (e)					
	403,8529	M+4	13C, H, 35C, 37C, O	HyCDD (8)					=
	445.7555		C <sub>12</sub> H <sub>2</sub> <sup>#</sup> Cl <sub>2</sub> of cl <sub>2</sub> O						·
	[430.9728]	Lock	C,F1,	PFK I					-
					_				=

The following nuclidic masses were used:

**®** 

825 O = 15,994915 0000  $^{36}CI = 34,968853$  $^{355}$   $^{37}CI = 36,965903$ 

H = 1,007825 C = 12.000000 <sup>13</sup>C = 13.003355 F = 18.9984

S = internal/recovery standard

LDC #:_	2	4445B	2/
		cover	,

### **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:_		/
Reviewer:	F	2_
2nd reviewer:	- · · · · · · ·	
	/	

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

Y N N/A

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

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

Concentration = (A)(L)(DF)

(A\_)(RRF)(V<sub>s</sub>)(%S)

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

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

 $l_x$  = Amount of internal standard added in nanograms (ng)

V<sub>a</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

Example:

Sample I.D. #3 . 2, 3, 7, 8- TCRD

Conc. = (342808) (2000) ( 167 448500 (1.25) (10.38) (0.434)

= 0.34 ·pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2, 3, 7, 8 - TCDF	(DB225		
		- 17379670 (2 466918000 (1.0	(las		
		466918000 (1.0	6) (10.38)C	p.93y)	
		- 7.2	44 pg/g		
			ļ		
	<u> </u>				-
					,
		·	<del> </del> -		<u> </u>
·	-				
					_

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

August 31, 2010

LDC Report Date:

December 8, 2010

Matrix:

Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0l020523

Sample Identification

EB-08312010

### 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
02651243-MB	9/8/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.7 pg/L 39 pg/L 4.2 pg/L	All samples in SDG G0l020523

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-08312010	1,2,3,4,6,7,8-HpCDD	3.5 pg/L	3.5U pg/L
	OCDD	34 pg/L	34U pg/L
	1,2,3,4,6,7,8-HpCDF	7.8 pg/L	7.8U pg/L

Sample EB-8312010 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-08312010	8/31/10	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	1.2 pg/L 2.3 pg/L 2.2 pg/L 3.5 pg/L 3.4 pg/L 2.9 pg/L 4.2 pg/L 4.2 pg/L 4.2 pg/L 2.4 pg/L 2.4 pg/L 7.8 pg/L 2.8 pg/L 12 pg/L	No associated samples in this SDG

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0251243-LCS	2,3,4,6,7,8-HxCDF	138 (80-137)	All samples in SDG G0I020523	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 G0l020523	All compounds reported below the PQL.	J (all detects)	Α

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G01020523	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

### XII. System Performance

Raw data were not reviewed for this SDG.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I020523

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G01020523	EB-08312010	2,3,4,6,7,8-HxCDF	J+ (all detects)	Р	Laboratory control samples (%R) (I)
G01020523	EB-08312010	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0I020523	EB-08312010	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 G0I020523

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I020523	EB08312010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.5U pg/L 34U pg/L 7.8U pg/L	А	ы

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0I020523

No Sample Data Qualified in this SDG

### **Tronox Northgate Henderson** ΞT

_DC #: <u>24445C21</u>	_ VALIDATION COMPLETENESS WORKSHEE
SDG #: G0I020523	_ Stage 2B
_aboratory: <u>Test America</u>	<u> </u>

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2nd	Reviewer:	1	11	
			y	

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 3 1 10
II.	HRGC/HRMS Instrument performance check	Δ	'
10.	Initial calibration	Δ	
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	Les sample
VII.	Laboratory control samples	SW	Les
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
Χ.	Target compound identifications	N .	
XI.	Compound quantitation and CRQLs	N	,
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	sw	EB-

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: سعقاد

	<del>~~~</del> .					
1 \	EB-08312010	11	8/25/10	21	31	
2		12\	0251243	22	 32	
3		13		23	 33	
4		14		24	34	
5		15		25	35	
6		16		26	 36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	 40	

Notes:		1

## 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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LDC #:_

## VALIDATION FINDINGS WORKSHEET

2nd Reviewer.\_ Reviewer: FT

> Phease 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

YN N/A

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

Blank analysis date: 9|21|10Was the method blank contaminated? Blank extraction date:\_

Associated samples:

(pg) 11 (

Sample Identification 7.8/5 2,5/4 24/4 E X Sp21 200 Blank ID 4.7 1.8 50 Compound Conc. units: Ф ড

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 # 24445 62)

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

Page: Reviewer: FT 2nd Reviewer:

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

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

Sampling date:

PAN-A-	Sample Identification																4	
Associated Samples:	Sa																	
Slank / Rinsate / Other: EB	k ID	/ # Einters 2 200																
Field blank type: (circle one) Field Blank / Rinsate / Other:	Compound Blank ID		7:1	D 2.3	Ê 7.2	F 3.5	५५ न	I 2.9	K	4.7	M 3.0	かて	7.8	8.7 2.8	۶ 7			CRQL

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

LDC #: 2444562/ 2

## VALIDATION FINDINGS WORKSHEET

2nd Reviewer. Reviewer: FT

Laboratory Control Samples (LCS)

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 LCS required? Y/N N/A

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?

MO RY D P m cosp, Qualifications + Associated Samples = RPD (Limits) LCSD %R (Limits) (80-137) LCS %R (Limits) 35 Compound  $\leq$ 225 1243 -Les Lab ID/Reference Date \*

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

**Collection Date:** 

September 10, 2010

LDC Report Date:

December 8, 2010

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0I140549

### Sample Identification

SSAJ8-03-1BPC

SSAJ8-03-3BPC

SSAJ8-03-3BPC FD

SSAJ8-03-5BPC

SSAJ8-03-8BPC

SSAJ8-03-10BPC\*\*

\$\$A07-07-0BPC\*\*

EB-09102010

SSAO7-07-0BPCMS

SSAO7-07-0BPCMSD

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

### Introduction

This data review covers 9 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 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
0263138-MB	9/20/10	1,2,3,4,7,8-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 OCDF	0.063 pg/g 0.11 pg/g 0.64 pg/g 0.062 pg/g 0.058 pg/g 0.040 pg/g 0.048 pg/g 0.14 pg/g 0.13 pg/g	All soil samples in SDG G0I140549

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
SSAJ8-03-1BPC	OCDD	3.1 pg/g	3.1U pg/g
SSAJ8-03-3BPC	OCDD	3.2 pg/g	3.2U pg/g
SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD	0.11 pg/g	0.11U pg/g
	OCDD	0.84 pg/g	0.84U pg/g
SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD	0.057 pg/g	0.057U pg/g
	OCDD	1.0 pg/g	1.0U pg/g
	2,3,4,6,7,8-HxCDF	0.16 pg/g	0.16U pg/g
	1,2,3,7,8,9-HxCDF	0.14 pg/g	0.14U pg/g
SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD	0.55 pg/g	0.55U pg/g
	OCDD	0.93 pg/g	0.93U pg/g
	2,3,4,6,7,8-HxCDF	0.10 pg/g	0.10U pg/g
	1,2,3,7,8,9-HxCDF	0.076 pg/g	0.076U pg/g

Sample EB09102010 was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

## VI. Matrix Spike/Matrix Spike Duplicates

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

## VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAJ8-03-1BPC	<sup>13</sup> C-OCDD <sup>13</sup> C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 37 (40-135)	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
SSAJ8-03-3BPC	<sup>13</sup> C-OCDD	34 (40-135)	O'CDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ8-03-5BPC	<sup>13</sup> C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAJ8-03-10BPC**	<sup>13</sup> C-OCDD	32 (40-135)	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAO7-07-0BPC**	<sup>13</sup> C-OCDD	334 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

## X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which 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
SSAJ8-03-10BPC**	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	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0l140549	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 G01140549	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

Samples SSAJ8-03-3BPC and SSAJ8-03-3BPC\_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

Concentration (pg/g)		DDD	D.W.			
Compound	SSAJ8-03-3BPC	SSAJ8-03-3BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.26	0.091	-	0.169 (≤0.55)	-	-
1,2,3,7,8-PeCDD	0.68	0.21	-	0.47 (≤2.8)	-	-

	Concenti	ration (pg/g)	RPD	Difference		
Compound	SSAJ8-03-3BPC	SSAJ8-03-3BPC_FD	(Limits)	(Limits)	Flags	A or P
1,2,3,4,7,8-HxCDD	0.50	0.11	-	0.39 (≤2.8)	-	- 1
1,2,3,6,7,8-HxCDD	1.0	0.29	-	0.71 (≤2.8)	-	-
1,2,3,7,8,9-HxCDD	0.81	0.17	-	0.64 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDD	3.4	0.87	-	2.53 (≤2.8)	-	
OCDD	3.2	0.84	-	2.36 (≤5.5)	-	-
2,3,7,8-TCDF	6.0	1.2	-	4.8 (≤0.55)	J (all detects)	Α
1,2,3,7,8-PeCDF	11	2.2	-	8.8 (≤2.8)	J (all detects)	А
2,3,4,7,8-PeCDF	6.0	<b>1.1</b>	-	4.9 (≤2.8)	J (all detects)	Α
1,2,3,4,7,8-HxCDF	16	3.0	-	13 (≤2.8)	J (all detects)	Α
1,2,3,6,7,8-HxCDF	13	2.7	-	10.3 (≤2.8)	J (all detects)	Α
2,3,4,6,7,8-HxCDF	2.8	0.63	-	2.17 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	2.5	0.62	,	1.88 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	51	10	-	41 (≤2.8)	J (all detects)	Α
1,2,3,4,7,8,9-HpCDF	21	4.6	-	16.4 (≤2.8)	J (all detects)	Α
OCDF	110	20 .	-	90 (≤5.5)	J (all detects)	Α

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0l140549

SDG	Sample	Compound	Flag	AorP	Reason (Code)
G0l140549	SSAJ8-03-1BPC	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)
G0l140549	SSAJ8-03-3BPC SSAJ8-03-5BPC SSAJ8-03-10BPC** SSAO7-07-0BPC**	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0I140549	SSAJ8-03-10BPC**	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (e)
G0l140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0l140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А	Project Quantitation Limit (k)
G0l140549	SSAJ8-03-3BPC SSAJ8-03-3BPC_FD	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects)	А	Field duplicates (RPD) (fd)

## Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0I140549

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I140549	SSAJ8-03-1BPC	OCDD	3.1U pg/g	А	bl
G0l140549	SSAJ8-03-3BPC	OCDD	3.2U pg/g	Α	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0l140549	SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD OCDD	0.11U pg/g 0.84U pg/g	А	bi
G0l140549	SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.057U pg/g 1.0U pg/g 0.16U pg/g 0.14U pg/g	A	ы
G0l140549	SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.55U pg/g 0.93U pg/g 0.10U pg/g 0.076U pg/g	À	ы

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0l140549

No Sample Data Qualified in this SDG

## **Tronox Northgate Henderson**

LDC #: 24445D21 **VALIDATION COMPLETENESS WORKSHEET** SDG #: G0I140549 Stage 2B/4 Laboratory: Test America

Date: 12/01/	10
Page: <u>/</u> _of <u>/</u> _	
Reviewer:	
2nd Reviewer:	

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

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

	Validation Area		Comments
<u>l.</u>	Technical holding times	4	Sampling dates: 9//0//U
II.	HRGC/HRMS Instrument performance check	Δ	7
_81.	Initial calibration	Δ	·
IV.	Routine calibration/ICV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LC>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	لىپى	·
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	Δ	
XIV.	Field duplicates	<u>\$</u>	D=2,3
XV.	Field blanks	NN	EB-8

Note: A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

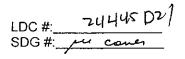
TB = Trip blank

EB = Equipment blank

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

		GOU + Wall					
,	1 1	SSAJ8-03-1BPC	11	0263138.	21	31	
4	2 1	SSAJ8-03-3BPC	12	0281099	22	32	
5	3 \	SSAJ8-03-3BPC_FD	13		23	33	
ب	4 ١	SSAJ8-03-5BPC	14	'	24	34	
7	5 <b>\</b>	SSAJ8-03-8BPC	15		25	35	
8	6 I	SSAJ8-03-10BPC**	16		26	36	
7	7	SSAO7-07-0BPC**	17		27	37	
u	82	EB-09102010	18		28	38	
	9	SSAO7-07-0BPCMS	19		29	39	
	10	SSAO7-07-0BPCMSD	20		30	40	

Notes:			
	•		



## **VALIDATION FINDINGS CHECKLIST**

	Page:_	o	f_2
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2nd	Reviewer:		)
	_	7	

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	1	·		·
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq$ 25% ?		_		
is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?				
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?				,
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?				
IV: Continuing calibration		• • • •		
Was a routine calibration performed at the beginning and end of each 12 hour period?	,,,,,	<u>-</u>		
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			er. I	
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?				
M. Matrix spike/Matrix spike duplicates			ide of Norma	
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?	<u>Da</u>			
VII: Laboratory control samples 2		MOSGO METRICA		
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 #:_	24445 D2
SDG #:_	per comes

## **VALIDATION FINDINGS CHECKLIST**

	Page:_	2-of_	2_
	Reviewer:	F	_
2nd	Reviewer:	1	_

	·			
VIII. Regional Quality Assurance and Quality Control		Γ	Γ.	
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	9Wg-58	5(50% ST)		
IX internal standards			ģ;∜∫ J	
Were internal standard recoveries within the 40-135% criteria?	١,			
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	<u>_</u>		<u> </u>	
X: Target compound identification	1	1	· 1 -	
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?		·		
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?		-		
Was the signal to noise ratio for each target compound and labeled standard ≥ 2.5?	/			
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?				
XIs:Compound quantitation/CRQLs	e. i		<u>}                                    </u>	
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XIII System periormance	2.2			
System performance was found to be acceptable.				
XIII Overali assessment of data				
Overall assessment of data was found to be acceptable.				·
XIV/sField/duplicales				
Field duplicate pairs were identified in this SDG.		-		
Target compounds were detected in the field duplicates.				
XV Field blanks on the second				
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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42
LDC #:

## VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer: FT

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

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

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?

Blank analysis date: 10/6/10 Y N/A Was the method blank contaminated? Blank extraction date: 92010

Associated samples:

(pd) y ins

Conc. units: va la concount	Blank ID					Sample Identification	tion		
الإ	1263138	0263138 MB SX		7	W	r	و		
	6.063	0.315			D.111/4	0.057/4			
	0,1]	0.55			- 1		0.5% W		
	0.64	3.7	3.1/4	n/₹.€	0.X4/u	1.0/4	0.93/U		
	0.062	0.3)	-		,	~			
	0,058	62.0							
	0.040	0.20				0.16/U	0.10/H		
_	0.0 48	D.24				0.14/11	0.076/4		
	0.14	<i>6</i> 1.0 ∣				- (			
	~61.0	0. <b>6</b> 5				(4.8)m-			,
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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 Matrix Spike/Matrix Spike Duplicates

Page: /of/ Reviewer: FT 2nd Reviewer: Q

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 'N N/A

/ MS/MSD. Soil / Water N N/A Was a MS/MSD analyzed

X/X/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?

# Date		Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
	9+10	person	2 0/(R +)	OBA %	( )	7	m anal Lesin
	•	to de	7	( ) +	( )		
			( )	( )	( )		
			( )	( )	( )		
			( )	( )	( )	÷	
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	•		( )	( )	( )		
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	<b></b>		( )	( )	( )		
			( )	( )	( )		

LDC #. Zy 44 SD2/

## **VALIDATION FINDINGS WORKSHEET** Internal Standards

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

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

X N/N/A Are all internal standard peaks > 10?

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

Reviewer: FT 2nd Reviewer:\_

							1	,
#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	it: 40-135%)		Qualif	Qualifications
			H	77	351-0h)	1/5/	M3/P C	ONAL G, B
			Ġ	7.5		, (	1,1	
					)	,		4.1.
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		'n	T	35	)	)		<i>چ</i> م, ه
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					)	)		
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		-	£	20	)	^	(	
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					)	(-		
		Internal Standards	Check Standard Used		Recovery Standards		Che	Check Standard Used
<	13C-2.3.7.8-TCDF	CDF		K. 13C-1.2.3.4-TCDD				
۳	13C-2,3,7,8-TCDD	con			xCDD			
d	<sup>13</sup> C-1 2 3 7 8-PeCDF	-PeCDF		M				
4	<sup>13</sup> C-12,3,7,8-PeCDD	-PecDD		z				
щ	13C-1,2,3,6,7,8-HxCDF	8-HxCDF		q				
ц	13C-1 2 3,6,7 8-HxCDD	8-HxCDD		В				
d	13C-12.3,4.6,7.8-HpCDE	Z.8-HpCDF		С				
ㅋ	13C-1,2,3,4,6,7,8-HpCDD	7,8-НрСDD		R				
1	130 0000							

## LDC# ZYYYSDZ

## 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 N/A 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	J/A detects (sp)			.IX detects (x)	(1) 202222		(~) 6/ 000	1			
Associated Samples	All			All			9	2			
Finding	All compounds reported below PQL			All compounds reported as EMPC			no Jul column	Įξ	ļ		
Correct Sample 1D							<b>±</b>				
# Date											

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24445D21

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

Page: <u>/</u> of	_ /
Reviewer: <b>,</b> £	7
2nd Reviewer:	_

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

Υ	N	NA
$\overline{Y}$	N	NA

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

( | d

	Concentrati	on (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	2	3	≤50	Difference	Limits	(Parent Only)
A	0.26	0.091		0.169	≤0.55	
В	0.68	0.21		0.47	≤2.8	
С	0.50	0.11		0.39	≤2.8	
D	1.0	0.29		0.71	≤2.8	
E	0.81	0.17		0.64	≤2.8	
F	3.4	0.87		2.53	≤2.8	
G	3.2	0.84		2.36	≤5.5	
н	6.0	1.2		4.8	≤0.55	J/A set
1	11	2.2		8.8	≤2.8	↓
J	6.0	1.1		4.9	≤2.8	1
к	16	3.0		13	≤2.8	V
Ł	13	2,7		10.3	≤2.8	$\downarrow$
М	2.8	0.63		2.17	≤2.8	•
N	2.5	0.62		1.88 4.02	≤2.8	_
o	51	10		41	≤2.8	1/A out
Р	21	4.6		16.4	≤2.8	Ţ
Q .	110	20		90	≤5.5	J

120 SHAHZ SDG#: LDC#:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Reviewer:\_ 2nd Reviewer: Page:

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

 $A_{\rm s} = A rea \ of \ associated \ internal \ standard \\ C_{\rm ls} = Concentration \ of \ internal \ standard \\ X = Mean \ of \ the \ RRFs$ 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 ( ころう std)	RRF ( 6.3 3std)	%RSD	%RSD
L	141	0/14/16	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	186.0	186.0	1.05	1.05	8-11	8-//
			2,3,7,8-TCDD ('3C-2,3,7,8-TCDD)	1.032	1.032	901	1.06	8 -01	<i>\$.01</i>
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	/+/-/	////	1.72	7 12	2.51	/9./
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	1.26	1.56	(2·3	12.3
			OCDE (13C.OCDD)		2///8	236	236	ارد ، ج	75-3
2	perx	01/20/1	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.05%	250.1	701	107	3.32	3.32
			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)						
			oche ("c.ochn)						
ო			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 (19C-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#: THE COUNTY D2/

## VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

\       	F	. 4
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 =  $(A_{\lambda})(C_{a})/(A_{b})(C_{\lambda})$ 

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

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

A<sub>is</sub> = Area of associated internal standard C<sub>is</sub> = Concentration of internal standard

-					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	G%	Q%
-	14:61 NOD	CV 5/01	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	4860	1.03	1.03	5%	4.5
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.032	1.03	1.03	0.1	0.0
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.14	127	7.27	11.6	9-//
		<u> </u>	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.134	/. يې	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	10.2	10.7
			OCDE (3C.OCDD)	211.5	2.0	2.0	5.4	メジ
2	OW DBIL	01/8/e1	┝┷	1.056	1.04	701	7.	<b>ル</b> ン
	21:17	·	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 ( <sup>13</sup> C-1,2,4,6,7,8,-HpCDD)					
			OCDE (3C-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 (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# LYLLY WY SDG# per cons

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

Page: of Reviewer: £7

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

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

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

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

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

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

MS/MSD samples:

4

	Š	Spike	Sample	Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Compound	Ad (	Added	Concentration	Concer ( Pg	Concentration	Percent	Percent Recovery	Percent 6	Percent Recovery	RPD	RPD
	) I	T U MSD	ופוס	רל SW	0 MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	19.8	20,	3, ]	5/2	2.5	671	671	1.7	711	6.9	6.9
1,2,3,7,8-PeCDD	98.7	1 00	8.6	116	113	109	109	101	104	7	7.6
1,2,3,4,7,8-HxCDD	<b>→</b>	٨	5.9	mal	971	= 2	611	6	119	6.	6.1
1,2,3,4,7,8,9-HpCDF	<b>→</b>	7	250	६५३	460	৮১৩	p59	7	7/2	64	7-9
OCDF	198	2	7007	9 x00	2010	0081	1801	184	138	ベス	28

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 #: 64 cons

# VALIDATION FINDINGS WORNSHEET

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:

Where: SSC = Spiked sample concentration SA = Spike added

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

026313B-10>

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

Recalculated

I CS/I CSD

RPO

	Š	Sike	Solked Sample	amole	ן די	CS	USDI	Sn	וט
	<b>Y</b>	Added	Concentration	tration					
Compound	, 87	100 (3)	\\ \frac{2}{2}	( 02 g	Percent Recovery	<b>Зесо</b> уелу	Percent Recovery	Recovery	
	) S3	U CSD	); SOI	U CSD	Renorted	Recalc	Reported	Racalc	Reported
2,3,7,8-TCDD	26.00	3	1.02	ΑN	101	101			
1,2,3,7,8-PeCDD	ده د		111	-	[11]	(1)			
1,2,3,4,7,8-HxCDD	001		201		102	701			
1,2,3,4,7,8,9-HpCDF	0 01		601		109	109			
OCDF	260	<b>→</b>	71		<i>%</i>	<i>X</i> \$			
							-		

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

Accurate mass"	CII UOI	Elemental Composition	Analyte	Descriptor	Accurate Mass(*)	CI ao		
303.9016	W	0.08.7			Acculate Mass	Ion ID	Elemental Composition	Analyte
305.8987	M+2	C,H,*C,*C10	TOPE	4	407.7818	M+2	C <sub>12</sub> H <sup>35</sup> CI <sub>8</sub> 37CIO	HDCDF
315.9419	Σ	130,H,30,LO	TCDE (e)		409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> 7Cl <sub>2</sub> O	HDCDF
317,9389	M+2	13C, H, 3CI, 3CIO	TCDE (8)		417.8250	Σ	13C <sub>12</sub> H3*CI,O	HpCDF (S)
319.8965	Σ	C, H, 50, O,	TCD (3)	-	419.8220	M+2	13C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> 37ClO	HPCDF
321.8936	M+2	C.H. 801.9010.	1007		423.7767	M+2	C <sub>12</sub> H <sup>35</sup> CI <sub>6</sub> 37CIO,	Hocop
331.9368	Σ	13C, H, SC, D	1007		425.7737	M+4	C,H <sup>25</sup> Cl, <sup>27</sup> Cl,O,	Hoch
333,9338	M+2	13C H 35C 37CIO	(8)		435,8169	M+2	13C, H <sup>32</sup> C, 37ClO,	Huchin (s)
375.8364	W+2	C T 35C 37C 2	(8) 000		437.8140	M+4	13C,H**C[*,O	Hacon (9)
354.9792]	Lock	0, 12 T. 0.	TXCOFF PFK		479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> O <sup>2</sup>	
					[450.9728]	Lock	°,F <sub>1</sub> ,	PFK
339,8597	0+W	C H 3C 37C C	L d					į
341.8567	M+4	0.1.1.3.0.1.0.0.0.0.0.0.0.0.0.0.0.0.0.0.	recur	ເດ	441.7428	M+2	C, *CI, *1CIO	OCHE
351,9000	M+2	12.3 (3.0 (2.0 c)	ייט דיניסיים		443,7399	M+4	0,350,300,0	OCDF OCDF
353,8970	M+4	10 10 10 10 10 10 10 10 10 10 10 10 10 1	Pecur (S)		457.7377	M+2	C,3*CI,3*CIÓ,	1000
355,8546	M+2	C.H. 350 37010	recor (s)		459.7348	M+4	C, **Cl, **Cl, O,	0000
357,8516	M+4	C.H. 401 901 0	recon accon		469.7780	M+2	-	OCDD (8)
367.8949	M+2	12. 3 C13 C12. 2 13C. H. 3CI 37CIO			471.7750	M+4	130,3401,3701,03	OCDD (S)
369.8919	M+4	12: 13 C1 C1C2	(S) (C) (C) (C)		513.6775	M+4	C,35C,05	DCDPE (3)
409.7974	· ^+	C H 8C1 9C10	recon (s)		[422.9278]	Lock	, H	7.7.7.7.7.7.7.7.7.7.7.7.7.7.7.7.7.7.7.
354,9792]	X	ο σ.	HPCDYE					<u> </u>
			ፓ ፕ			•		
373,8208	M+0							
375.8178		0,5,12,0,5,0	TXC		_			
383.8639			HXCDF	,				
385 BE10			HXCDF (S)					
200,000		OlOve Joseph Jone	HXCDF (S)				-	
2 !		C <sub>12</sub> H <sub>2</sub> *C <sub>15</sub> **C <sub>102</sub>	HXCDD					
391.812/		C <sub>12</sub> H <sub>2</sub> **Cl <sub>1</sub> **Cl <sub>2</sub> O <sub>2</sub>	HXCDD					
401,8359			HxCDD (S)					
403.8529	M+4		HxCDD (S)	•				•
445.7555			OCDPE					
[430,9728]	YOO,		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 #:_	24445		D2/
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## **VALIDATION FINDINGS WORKSHEET**

Sample Calculation Verification

Page:		
Reviewer:	7	7
2nd reviewer:		a
•		7

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

/	Y	Ŋ	N/A
	Y	Λĺ.	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?

Example:

Concentration = $(A_{\cdot})(I_{\cdot})(DF)$ $(A_{\cdot})(RRF)(V_{\circ})(%S)$			
A <sub>x</sub>	<del>=</del>	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	
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)	
V.	=	Volume or weight of sample extract in milliliters (ml) or grams (g).	
RRF	=	Relative Response Factor (average) from the initial calibration	
Df	=	Dilution Factor.	
%S	=	Percent solids, applicable to soil and solid matrices only.	

Sample I.D.	<u>#7.</u>	., 3, 7, 8-	TCPD
Conc. = (157)	53240)(20 000 (1.03	) ( (10.40) ) (	0.7918
na .	3.1. pg	/2	

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#7 2,3,7,8-7	CDF		
			24552)		
		= 2/5320000 (	20107		
		= 215320000 ( (492645000)(	1.056) (10.	40)(0.9918	/
		= 80 p	<u> </u>	·	
		= 80 p	1/7		
		****			
					,
	<u> </u>				·
	:				