

Laboratory Data Consultants, Inc.

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Northgate Environmental Management, Inc.

December 30, 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 December 7, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 24524:

SDG #

G0J130426, G0J140638, G0J150566 G0J170404, G0J200489, G0J230497 G0J270514, G0K130496 Dioxins/Dibenzofurans

Fraction

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

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

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

Sincerely.

Erlindá T. Rauto

Operations Manager/Senior Chemist

Attachment 1

DL 12/07/10

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LDC #: <u>24524</u> SDG #: <u>G0J130426, G0J140638, G0J150566, G0J170404</u> G0J200489, <u>G0J230497, G0J270514, G0K130496</u> Page: 1 of 1 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?	х			
II. EDD Qualifier Population		· 第十年	過數	
Were all qualifiers from the validation report populated into the EDD?	Х			
III. EDD Lab Anomalies				
Were EDD anomalies identified?		Х		
If yes, were they corrected or documented for the client?			х	See EDD_discrepancy_ form_LDC24524_122910.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:

September 2, 2010

LDC Report Date:

December 23, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J130426

Sample Identification

SSAN7-04-4BPC SSAN7-04-5BPC**

^{**}Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0287481-MB	9/27/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.089 pg/g 0.31 pg/g 0.065 pg/g 0.070 pg/g 0.043 pg/g 0.15 pg/g 0.070 pg/g 0.36 pg/g	All samples in SDG G0J130426

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

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.

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
SSAN7-04-4BPC SSAN7-04-5BPC**	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J130426	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 G0J130426	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J130426

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	2,3,7,8-TCDF	J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	All compounds reported below the PQL.	J (all detects)	Α	Project Quantitation Limit (sp)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	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 G0J130426

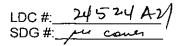
No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0J130426

No Sample Data Qualified in this SDG

SDG	#: 24524A21 #: G0J130426 ratory: Test America	. VA 		_	PLĚT		ESS	derson WORKSHEET	•	Date: / 2//b Page: _/of Reviewer:/ 2nd Reviewer:/
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	Validation	<u>Area</u>		<u> </u>	 			Comn	<u>nents</u>	
<u> </u>	Technical holding times				Sam	pling d	ates:	9/2/10		
II.	HRGC/HRMS instrument pe	<u>∍rform</u> a	ance check	<u>A</u>	 			•		
III.	Initial calibration			<u>Δ</u>	 					
IV.				A	 				· · · · · · · · · · · · · · · · · · ·	
V.	Blanks			SW	—	1.	,	1 /		
VI.		plicate	s	N	C	lien		specified		
VII.	Laboratory control samples			A		LC	>	V		
VIII.	. Regional quality assurance a	and qu	ality control	N	<u> </u>					
IX.	Internal standards		· · · · · · · · · · · · · · · · · · ·	· <u>\</u>	—					
X.	Target compound identificati	ions		Δ,	Not	review	ed for	Stage 2B validation.		
XI.	Compound quantitation and	CRQL	<u>.s</u>	SW	Not	review	ed for	Stage 2B validation.		
XII.	System performance			Δ	Not	review	ed for	Stage 2B validation.		
XIII.	Overall assessment of data			A						
XIV.	. Field duplicates			N			_			
XV.	Field blanks			N						
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet ted Samples: ** Indicates samp		R = Rin: FB = Fie	ield blank		cted		D = Duplicate TB = Trip blank EB = Equipment blan	nk	
1 +	SSAN7-04-4BPC	11	02871	481-MI	3	21			31	
2 .	SSAN7-04-5BPC**	12				22			32	
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Notes:_				
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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.				
III. GC/MS Instrument performance check	<u> </u>			· · · · · · · · · · · · · · · · · · ·
Was PFK exact mass 380.9760 verified?		<u> </u>		
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?	/			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?			<u> </u>	
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?			i	
IV: Continuing calibration	1	· ·		
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?			<u> </u>	<u> </u>
V. Blänks				
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?				
VI Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				-
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	ar di cipitar di	a y Algera e		Street Mary Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of Control of
VII Laboratory control samples		加速等等	TWE! 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?	2			

LDC #: 24524 A2

VALIDATION FINDINGS CHECKLIST

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

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VALIDATION FINDINGS WORKSHEET

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

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

Notes:

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VALIDATION FINDINGS WORKSHEET

2nd Reviewer: 0 Reviewer:

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

Were all samples associated with a method blank?

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

ンヤ Associated samples: Blank analysis date: 10/15/10 Was the method blank contaminated? Blank extraction date: 9/27/10 Conc. units: Y N/A AN AY

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Sample Identification]		 														
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Blank ID	0287481-MB	0.089	0.3/	0.065	0.070	0.043	0.15	0.070	0.36									
bunod																		
Compound		I	e e	H	X	7	9	d	Q									

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 # 2452442/

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

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

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) JK detects (k) U 1/10det **Associated Samples** ₹ Ì All compounds reported below PQL All compounds reported as EMPC Pamale Finding Sample ID Der 3 ¥ Date

Comments: _See sample calculation verification worksheet for recalculations

LDC#: 24524 AZ/ SDG#: ALE 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 = (A₄)(C₈)/(A₁₈)(C₄) average RRF = sum of the RRFs/number of standards %RSD = 100 * (S/X)

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated.
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (52) std)	RRF (Cシラ std)	%RSD	%RSD
Ψ-	147	48/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	04770	0716170	1.04206	902401	5.67386	719.5
<u> </u>		2/6/	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.10 069	1.10069	1.15267	192311	82626.5	5.729
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.16239	1.16239	1.31722	1-31722	12 sh5.6	1/s.6
<u> </u>			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.00001	1.0000/	1-12727	1-12727	4. 82825	828-h
			OCDE (13C, OCDD)	1.39942	1.39944 1.39942	1.51113	1. 51113	7.02964	4.009
7	DBW	1/20/10	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.057	1001	1.02	70.1	3.32	3.32
			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	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDE (3C.OCDD)			,			
3			2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)						
		,	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						

Comments: Refer to 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#. 24524A21 SDG#:

Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: 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 = $(A_{\nu})(C_{\nu})/(A_{\nu})(C_{\nu})$

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A_x = Area of compound, A_x = Concentration of compound, C_x = Where:

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	0 %	0 %
	eev 13:5B	01/51/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.96170	1.01702	1.01702	8:5	R55
		, 	2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.10069	8117 1.1	81221.1	6.9	6.9
		,	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.16239	1.1787	1.1782	6.0	6.0
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.0090.1	1.13967	1.13967	7.5	7.5
			CODE (13C.OCDD)	1.39942	1.4972	111911	7.0	2.4
2	lev 8:4/	C/22/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.050	1.15	1.12	0.6	9.0
		•	2;3;7;8-TCDD (1°C-2,3;7,8-7CDD)					
		-	1,2,3,6,7,8-HxCDD(13C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,2,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			CONF (196-ORD)					
က		21/2401	ew 10:44 10/2/1/0 2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.05	ho-1	1.04	1.8	8-1
			2378.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-HpedD (13C-1,2,4,6,7,8,-HpCDD)					
			oco r (2 ocop)					

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 #: 443 4474 // SDG #: 444 1001

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Reviewer: C2

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)

028748,

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	Sp	lke	Spiked	Sample	1.08	v.	I CSD	a	(CS// CSD	CSD
Compound	Add	Added,	Concentration	tration	Paccent Recovery	Vievooe	Percent Recovery	Vievose	OPR	۵
	108	l csn	108	1 CSD	Ranortad	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.02	AV	6.61	40	97	47				
1,2,3,7,8-PeCDD	001		0.46		þb	46				\
1,2,3,4,7,8-HxCDD	001		98.3	-	86	86				
1,2,3,4,7,8,9-HpCDF	001		701		107	70/				
OCDF	Cos	7	187	1	16	44	NA	\		
							`			
									,	
				,						

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

1 303.9016		21 25	Flemental Composition	Amelican					
	46		IIONISO INC.	Analyte	Descriptor	Accurate Mass ^(a)	Ion ID	Elemental Composition	Analyte
205 00	0.00	Σ,	C ₁₂ H ₄ **Cl ₂ O	TCDF	4	407 7818		30	
7969.606	78	M+2	C12H4**C13**C10	TCDF		409 7788	Z+Z	C ₁₂ H ² Cl ₃ ClO	HPCDF
#D'CLO	2	≅	O, 10, 1, 1, 10, 1, 1	TCDF (S)		444 9000	1VI+4	C12H~C13O	FECOF
317,9389	68	M+2		TCDF (S)		417.0250	Σ	1,C1,2H3,C1,O	HpCDF (S)
319.8965	-	Σ	C,H,*CI,O,	TCDD (3)		419,8220	M+2	13C12H35CII	HocoF
321.8936	98	M+2	C.H.*CI.*C10.	מחסד		423.7767	M+2	C,H ³⁵ Cl ₂ ³⁷ ClO,	HpCDD
331,9368	89	Σ	13C H &CI O	100 F		425.7737	M+4	C.,H*Cl.*7Cl.O.	ייין דו מייט קו
333,9338	38	M+2	137 1 37	(8)		435.8169	M+2	13C H ₃₅ Cl 37ClO	
375.8364	- 79	M +		(S)		437.8140	M+4	13C H ³⁵ C 37C O	
[354.9792]	1282	20		HXCDPE		479.7165	M+4	C 137 050	HPCDD (S)
•	•		5	AH.		[430.9728]	LOCK	0,F; 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,	NCDPE
†									<u> </u>
2 339,8597	26	M+2	CH.ªCI.VCIO	7.00-2					i
341,8567	. 29	M+4	O.H. 201.70	מינים ש	so.	441.7428	M+2	C, *CI, *CIO	OCHE
351,9000	00	M+2	13 C 1 3 C 1 C 1 C 1 C 1 C 1 C 1 C 1 C 1	בים בים		443.7399	M+4		300
353.8970	02	M+4	12 12 12 12 12 12 12 12 12 12 12 12 12 1	Pecur (S)		457.7377			יים כי מינים כי
355,8546	16		(12.13 (13 (12)) C II 35C 37CIO	recur (s)		459,7348			מיניס כ
357.8516	9			Pecup		469.7780			ממסט מיי ממסט
367,8949	6		12 13 02 02 13 02 02 13 13 13 13 13 13 13 13 13 13 13 13 13	Pecup		471.7750		150 350 350 0	(s) 0000 0000 0000
369 8919				Pecdo (s)		513,6775			(s)
400 2024		₩+4		Pecdo (S)	-	[422 9278]	1 20		DCDPE
181.804	± 3	M+2	(C ₁₂ H ₃ *Cl ₈ *ClO	HDCDPE			3	C ₁₀ F ₁₇	PFK
[354.9792]	92]	LOCK	n n	PFK			•		
					<u> </u>			-	
3 373.8208	ğ	M+2	CIOR IS						
375,8178			C12 12 C13 C1C	TXCDF					
383.8639	 _		12 12 13 OF OF OF OF OF OF OF OF OF OF OF OF OF	TXCDF 1.00F			-		
385,8610	_	0	13, 12, 13, 13, 13, 13, 13, 13, 13, 13, 13, 13	HXCDF (S)					
389.8156	 9			1XCD1 (S)		_	_		
391.8127			C. H. & C. S. C. C.	HXCDD					
401.8559	<u>.</u>		13C. H. 3C. 3C.	מיייים מיייים			_		
403,8529	<u> </u>	M+4	19C. H. 3C. 17C. O	(8)			,		
445.7555	٠.	M+4	C,2H,36Cl,30Cl,0	OCDPF OCDPF	_			_	
430.9728		Lock	C,F ₁ ,	PFK					
							<u> </u>		

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 ¹³C = 13.003355 F = 18,9984

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

S = internal/recovery standard

LDC #:	2	45	- 2	YA	2]
SDG #:					

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:_	/_of_	
Reviewer:	F	ħ
2nd reviewer:	D	<u> </u>

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

/	/ Y	N	N/A
1	Y/	Ν	N/A

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

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

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

(A_)(RRF)(V_s)(%S)

A_x = Area of the characteristic ion (EICP) for the compound to be measured

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard

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

V_o = 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. #2 2, 3, 7, 8-7010

Conc. = (33337,744) (2000) 289(4749) (1.10/) (10.43) (0.01/8

= 2.2 pg/g

			·		
#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
	д э о	3, 7, 8-TCPF = 1102412544 559 457648(
 	#2 2,	D, 1, 8 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 -	/ >		_
		= 1102412544	1000)		
		559 457648(1.056) (10.43) (0.918)	
		= 390	12/9		
			10/		
			<u> </u>		
					<u> </u>
		11-71-87-74-77-47-1-42-7-1-1			
					
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			<u></u>	<u> </u>	

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

October 12, 2010

LDC Report Date:

December 20, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J140638

Sample Identification

SSAL2-04-1_01_BPC

SSAL2-04-2 01 BPC

SSAL2-04-3 01 BPC

SSAL2-04-4 01 BPC**

SSAL2-05-1_01_BPC

SSAL2-05-2 01 BPC

SSAL2-05-3 01 BPC

SSAL2-05-4_01_BPC

SSAL2-04-3 01 BPCMS

SSAL2-04-3 01 BPCMSD

^{**}Indicates sample underwent Stage 4 review

Introduction

This data review covers 10 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0288451-MB	10/16/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.060 pg/g 0.12 pg/g 0.53 pg/g 0.026 pg/g 0.19 pg/g 0.17 pg/g 0.12 pg/g	All samples in SDG G0J140638

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

Sample	Compound	Reported Concentration	Modified Final Concentration
SSAL2-04-3_01_BPC	1,2,3,7,8,9-HxCDD	0.21 pg/g	0.21U pg/g
	OCDD	1.8 pg/g	1.8U pg/g
SSAL2-05-2_01_BPC	1,2,3,7,8,9-HxCDD	0.13 pg/g	0.13U pg/g
	OCDD	0.85 pg/g	0.85U pg/g

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	O(D // imita)		_	
SSAL2-04-2_01_BPC	13C-OCDD	%R (Limits) 35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A or P
SSAL2-04-3_01_BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	24 (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)	Р
SSAL2-04-4_01_BPC**	¹³ C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-05-1_01_BPC	¹³ C-OCDD	36 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-05-2_01_BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	30 (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
SSAL2-05-3_01_BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	29 (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)	Р
SSAL2-05-4_01_BPC	¹³ C-OCDD	28 (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
SSAL2-04-1_01_BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
SSAL2-04-4_01_BPC**	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
SSAL2-05-3_01_BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	2,3,4,7,8-HxCDF 2,3,4,6,7,8-HpCDF 2,3,4,7,8,9-HpCDF		J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	Р

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J140638	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 G0J140638	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	Α .

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J140638

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J140638	SSAL2-04-2_01_BPC SSAL2-04-4_01_BPC** SSAL2-05-1_01_BPC SSAL2-05-4_01_BPC	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J140638	SSAL2-04-3_01_BPC SSAL2-05-2_01_BPC SSAL2-05-3_01_BPC	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)
G0J140638	SSAL2-04-1_01_BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0J140638	SSAL2-04-4_01_BPC**	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0J140638	SSAL2-05-3_01_BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0J140638	SSAL2-04-1_01_BPC SSAL2-04-2_01_BPC SSAL2-04-3_01_BPC SSAL2-04-4_01_BPC** SSAL2-05-1_01_BPC SSAL2-05-2_01_BPC SSAL2-05-3_01_BPC SSAL2-05-4_01_BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J140638	SSAL2-04-1_01_BPC SSAL2-04-2_01_BPC SSAL2-04-3_01_BPC SSAL2-04-4_01_BPC** SSAL2-05-1_01_BPC SSAL2-05-2_01_BPC SSAL2-05-3_01_BPC SSAL2-05-4_01_BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0J140638

\$DG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J140638	SSAL2-04-3_01_BPC	1,2,3,7,8,9-HxCDD OCDD	0.21U pg/g 1.8U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J140638	SSAL2-05-2_01_BPC	1,2,3,7,8,9-HxCDD OCDD	0.13U pg/g 0.85U pg/g	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0J140638

No Sample Data Qualified in this SDG

Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #: 24524B21 SDG #: G0J140638 Stage 2B/4 Laboratory: Test America

Date:	12/16/10
Page:_	<u>/</u> of
Reviewer:	<u> </u>
2nd Reviewer:	4

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

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

	Validation Area		Comments
<u> </u>	Technical holding times	Δ	Sampling dates: 10/12/10
11.	HRGC/HRMS Instrument performance check	Д	,
<u>B</u> I.	Initial calibration	A	
IV.	Routine calibration/I CV	A	
V.	Blanks	SW	·
VI.	Matrix spike/Matrix spike duplicates	لىرى	
VII.	Laboratory control samples	A	ies
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	سی	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

<u>50/L</u> 0288451 1 SSAL2-04-1_01_BPC 11 21 31 32 SSAL2-04-2_01_BPC 12 22 3 SSAL2-04-3_01_BPC 13 23 33 SSAL2-04-4_01_BPC** 14 24 34 5 SSAL2-05-1_01_BPC 15 25 35 6 SSAL2-05-2_01_BPC 16 26 36 SSAL2-05-3_01_BPC 17 27 37 8 SSAL2-05-4_01_BPC 18 28 38 9 SSAL2-04-3_01_BPCMS 19 39 SSAL2-04-3_01_BPCMSD 20 30 40

Note	s:				

LDC#: 2454B2/ SDG#: 14 cones

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
िनechnical holding times	<u> </u>	<u>.</u>		
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check	·	·		
Was PFK exact mass 380 9760 verified?		<u> </u>		
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)?			<u> </u>	
Was the mass resolution adequately check with PFK?			ļ	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
fill: Initial calibration	, -	,	т	
Was the initial calibration performed at 5 concentration levels?			<u> </u>	
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?			<u> </u>	
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?				
V. Blanks		9.34.34 <u>8</u>		
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?				
VI. Matrix spike/Matrix spike duplicates			lite son When	
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?				
VillaLaboratory control samples		MESSA MEGIS	可能的	
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 #: 24 5 24 B2 | SDG #: 14 comes

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: 2 of 2

VIII: Regional Quality Assurance and Quality Control	i i		_	
Were performance evaluation (PE) samples performed?			1	
Were the performance evaluation (PE) samples within the acceptance limits?				
IX_Internal standards			34数(<u>新文</u>)	
Were internal standard recoveries within the 40-135% criteria?	X	1		
Was the minimum S/N ratio of all internal standard peaks ≥ 10?		<u>ł</u>		
X: Target compound identification	· .		· ·	
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?				
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?		ļ,	<u> </u>	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	Warm	/	ļ	
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	/		ļ	
Does the maximum intensity of each specified characteristic ion coincide within ±2 seconds (includes labeled standards)?	/			
For PCDF identification, was any signal (S/N \geq 2.5, at \pm seconds RT) detected in the corresponding PCDPE channel?	/	}		
Was an acceptable lock mass recorded and monitored?		<u> </u>		
XI: Compound quantitation/CRQLs		v J	<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?				
XII System performances:		22) 33 34 24		
System performance was found to be acceptable.		-		
XIII Overall assessment of data		de in Care		
Overall assessment of data was found to be acceptable.	/	-		
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.			<u> </u>	
Target compounds were detected in the field duplicates.				
XVdField blanks 2014 2014 2014 2014				
Field blanks were identified in this SDG.			F	
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC# 2452482

VALIDATION FINDINGS WORKSHEET

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

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

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

Blank analysis date: 10 25 10 Y/N N/A Was the method blank contaminated?

Blank extraction date: 10 10 10

Conc. units: 👝 🏳 🗸

Y N N/A VN ₹

Associated samples:

Page:

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:																	
									i			•					
- Jacobi Sam	Sample Identification																
	2															-	
	9	h/&1.0	ì	h/58.0											·		
	3	10.21/y	1	1.8/4	-											•	
	MB 5X	0.30	0.40	2.65	0.13	0.95	0.85	0.60									
Rlank ID	X2 8M 12 4 82 50	0.000	0.12	6.53	0.026	0.19	0.17	0.12									
) OI		בב	U_	P		ø	Q.	প									

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

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

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

MS/MSD. Soil / Water.

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

#	Date		Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		01+10	Р	651-PT) Et1	(bel-6L) (H	(3	mo gual cessin
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LDC# 245 24B2/

VALIDATION FINDINGS WORKSHEET

Internal Standards

*	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	40-135%)	Que	Qualifications
\vdash		7	I	35 (40-135)	dl rnic	OWAL G, G
					(4		
\vdash)			
		3	I) <i>γ γ</i>			8,5
\vdash			Э	68	(90
\vdash							
		ц	I) ६६			ଷ୍ଟ
)			
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			6) 6E	(9
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		7	I) hor			
			6) 68)		•
		•)	(
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					(
					(
)	(

	Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
Ā	13C-2.3.7.8-TCDF		Ϋ́	¹³ C-1.2.3.4-TCDD	
æ	¹³ C-2 3 7 8-TCDD		-	¹³ C-1 2 3 7 8 9-HxCDD	
C			Σ		
4	13C-12378-PeCDD		z		
Щ	13C-123678-HxCDF		q		
ц	¹³ С-123678-HxCDD		Ω		
ď	=		С		
月	13C-1234678-HnCDD		œ		
	13c Ochu		ŀ		

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VALIDATION FINDINGS WORKSHEET

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Reviewer: 2nd Reviewer:

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".

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

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

*	Date	Lab ID/Reference	Internal Standard	% Re	% Recovery (Limit: 40-135%)	Qualifications
		<u>a_</u>	#	30	Se1-0h)) NO grad MS
			Н	ر د ر	1)	
			(5)	35)	
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		10	+1	36	361-0h)	We apout MSV
			I	61)	
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)	(
		Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
∢	¹³ C-2.3.7.8-TCDF	;DF		K. 13C.	¹³ C-1.2.3.4-TCDD	
æ	13C-2 3 7 8-TCDD	ממנ			¹³ C-1 2 3 7 8 9-HxCDD	
d	13C-12378-PeCDE	PecDF		M		
٩	13C-12378-PeCDD	PeCDD		Z		
Ч	13C-1,2,3,6,7,8-HxCDE	3-HxCDF		q		
ш	13C-1,2,3,6,7,8-HxCDD	3-HxCDD		А		
d	13C-12.3.4.6.7.8-HpCDE	7,8-HpCDF		q		And the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of t
<u>크</u>	13C-1,2,3,4,6,7,8-HpCDD	7,8-НрСDD		<u>a</u>		
-	130 000			 1-		

LDC# 24624B27

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".

X N N/A

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

	Qualifications	J/A detects (sp)			K detecte ///	(i) connocio	J/Pdet (e)		(0)			
	Associated Samples	All			All		1	4	7			
	Finding	All compounds reported below PQL			All compounds reported as EMPC		x'd cal lange	1	1			
1	Sample 10						H, O, OS	Ø, Ø	HK 8, P, B			
	# Date											

Comments: See sample calculation verification worksheet for recalculations

SDG#: MS 24 B 2/

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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

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

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

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

								The second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second secon	
			-	Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
		Calibration		Average	Average	RRF	RRF		
*	Standard ID	Date	Compound (Reference Internal Standard)	RRF (initial)	RRF (initial)	(dsか std)	(d.s.3 std)	%RSD	%RSD
_	1CA7	CIPZIL	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.05%	1.052	1.07	1:02	3.32	3.32
	DBM	<u>}</u> -	2,2,7,8,TCDD (13C,2,3,7,8,TCDD)						
			1,2,3,6,7,8-HXCDD (13C-4,2,3,6,7,8-HXCDD)						
		<u>, </u>	1,2,3,4,6,7,8-HeCDD (13C-1,2,4,6,7,8,-HpCDD)						
		,	OCDE PAC OCODI						
7	KAL	7/21/10	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	0.995	ه نطور	0.9849	6-9849	3.69	3.68
		-	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	0.983	0.983	0.968	0.968	3.24	3.24
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	१।७३	1.163	1.1014	1.1014	11.5	2.17
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.042	1.072	1.0735	1.0435	7.6	7-19-2
			OCDE (13C OCDD)	1.570	1.30	1.3500	<u>્ર</u> ાજફ-}	1.98	×e
က			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)		_				
			2,3,7,8-TCDD (1°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 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#: 24524/82/

VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of Reviewer:

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

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

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

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

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

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

	,				Reported	Recatculated	Reported	Recalculated
**:	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
-	aev 5:18	Medal	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.050	71.1	1-12	6.1	(.9
	Dow.		2,3,7,8-TCDD ("C-2,3,7,8-TCDD)					
		•	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
	eer 19:03	0/2/01	17,3,4,6,7,8-HpCDD ("3C-1,2,4,6,7,8,-HpCDD)	1.050		<u>+</u> =:	ک ^د هر	گ ^ر گ
	pork		ACDE (13C, OCDD)					
2	test 4:24	01/22/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	566.0	h6.0	6.94	9.5	<i>S</i> :6
		`	2,3,7,8-TCDD ('3C-2,3,7,8-TCDD)	6 %L.0	10.0	hb.o	(· ,	(÷
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	691.1	12.1	1.22	<i>۲</i> ٠ ×	8.4
		, ,	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.072	801	Xo.		o o
			OCDE (3C-OCDD)	1.370	1.39	1.30	ブ :	<u>.</u>
3			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)		•			•
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)					
		·	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			OCDF (13C-OCDD)					

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.

/ nahr chr the south SDG#: LDC#:

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:

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

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

MS/MSD samples:

0/+

		Sample		Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	Renorted	Recalculated
Added Concentration		Concen (tration 2	Concentration (1924)	Concentration	Percent	Percent Recovery	Percent	Percent Recovery	RPD	RPD
0.00 USW SW	MSD) Dr		SW SW	O MSD	Reported	Rocalc	Benorted	Bossle		
21.1 21.2 0.093		6.093		622	22.9	113	5	100	χol	4.0	-2: -1:
105 106 0.15		0.15		021	125	114	hli	11.8	<i>\$</i> !	۶. ۶.	3.8
51.0	4 0.15	0.15		105	011	100	pol	hal	hal	4.7	4.7
7.7	7.7	7.7		951	157	113	오네	7-	141	/× 0	48-0
211 212 42		23		11ع	284	601	60	11/2	ا <u>د</u> ا	4.7	4.7
			اڙ			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,					

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# 443 47 101/ SDG# 444 4044

VALIDATION FINDINGS WURNSHEET Laboratory Control Sample Results Verification

Reviewer: C2

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

3

0284年5

TCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	28.	ika	Solked S	amole	SUI	8	USDI	ď	(CS/ICSD	csp
Compound	A (2)	Added (20)	Concentration (22 (4)	tration (2)	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	۵
) so i	្រំ (1.CSD	$\bigcap_{\mathbf{I}} \mathbf{s}_{\mathbf{S}\mathbf{I}}$	['0 csp	Raportad	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	ت. مح	女 2	v. 0	47	0"	110				
1,2,3,7,8-PeCDD	001		118		118	118				
1,2,3,4,7,8-HxCDD	0 oJ		111		11	111				
1,2,3,4,7,8,9-HpCDF	001	-	621		621	123				
OCDF	002	>	724	>	41	117	42			
						٠				

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

ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

, , ,			_				_					_		-			_	_					_	_			_			_
	Anaiyte	НрСОР	HPCDF	HpCDF (S)	HPODF	HPCDD	Hoch (s)			<u>:</u> :	2000	OCDF	OCDD	ocpp	ocpp (s)	ocpp (s)	DCDPE	PFK .							,				-	
	Elemental Composition	C ₁₂ H ³⁶ Cl ₆ 37ClO	O ² ID ₁ ² ID ₂ ID ₂ ² ID ₂	1.C. H.C.)O	C. T. C. S. C.O.	C.H**CL**CLO	(3C,H ³⁶ Cl, 3ClO,	¹¹C,'H**CI,*7CI,O,	C ₁₂ H³*Cl,3*Cl ₂ Ō ¯ C ₉ F ₁₇		C. 38CL 37CIO	C, 20, 30;0		C ₁₂ **C1 ₁ **C1 ₂ O ₂		-														
-	Ol uol	M+2	₩ + 7	Z .	¥ ± ₹	M+4	M+2	M+4	M+4 LOCK X		M+2							Š										,		
According to the control (k)	Accurate Mass	407.7818	417 8250	419 8220	423.7767	425,7737	435,8169	437.8140	479.7165 [430.9728]		441.7428	443.7399	457.7377	459.7348	459.7780 471 7750	519 677E	10.070	0/36:321												
Descriptor	Indiana.	4									ທ																			
Analyte		TCDF	TCDF (S)	TCDF (S)	TCDD	1CDD 7055 (3)	1CDD (8)	1000 (s)	PFK 1		PeCDF	Pecur Pecur	Pectre (s)	PacDD (3)	PecDD	Pecob (S)	PeCDD (S)	HpCDPE	PFK		HXCDF	HXCDF	HXCDF (S)	HxCDF (S)	HXCDD	HXCDD	HXCDD (S)	HXCDD (S)	PFK	
Elemental Composition	O TOROL O	C12H,**C13*C10	O,12**H,20*	LacizH, acijarcio		12 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	120, H. 350, 970, 0	C,H, aC 37C O	ر الجاري الجاري			1,5,1,3,0,3,0,5,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0	0.10%, 10%, 11%, 10%, 10%, 10%, 10%, 10%,	C,H,*Cl,*ClO,	C12H3*C13*C12O2	13C12H3*C13*C1O3	13C ₁₂ H ₃ 3Cl ₃ O ₂	C ₁₂ H ₃ 2ClO	C, F ₁₃		C ₁₂ H ₂ ³¹ ClO	G ₁₂ H ₂ **Cl ₄ *Cl ₂ O	O,U,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	12C12H2 30C10	C ₁ H ₂ *C ₃ *C O ₂		130 12 12 12 12 12 12 12 12 12 12 12 12 12	C,H, #C1,**C1,O	C,F,	
Ol nol	2	M+2	Σ,	M+2	M + 2	. ≥	M+2	M+2	Fock		M+2	M+2	M+4	M+2	M+4	M+2	M+4	M+2	Look			4				M++		M+4	Lock	
Accurate mass ^(a)	303,9016	305.8987	315,9419	310 906	321,8936	331.9368	333.9338	375,8364	[354.9792]	330 BE07	341.8567	351.9000	353,8970	355,8546	357,8516	357.8848	369,8919	409.7974	[354.9792]		3/3,6208	3/5,8178	900,0009	380 8456	391.8127	401,8559	403,8529	445.7555	[430.9728]	
Descriptor	-					-	-			0	1						-			Į,	°									

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 $^{10}C = 13.003355$ F = 18.9984

O = 15.994915 $^{36}Cl = 34.968853$ $^{57}Cl = 36.965903$

S = internal/recovery standard

LDC #:_	24	1521B21	,
		cover	

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:_	/_of_	
Reviewer:	卢	7
2nd reviewer:	\mathscr{A}	

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

Y/N N/A Y N N/A Were all reported results recalculated and verified for all level IV samples?

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

Concentration = (A)(I)(DF)

(A_k)(RRF)(V_a)(%S)

A_x = Area of the characteristic ion (EICP) for the compound to be measured

A_k = Area of the characteristic ion (EICP) for the specific internal standard

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

V_o = 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. #4 . 2,3,7,8-TCDD

Conc. = (222/240) (2000) ()
75454600) (0.99) (0)

= 6.12 pg/g

r - 7			<u> </u>	1	1
#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
		#4 2, 3, 7, 8- TCDF			
\vdash		77,70			<u> </u>
			-		
		2780300 (2000	1	= 177 p	\$ 19
		2780300 (2000 30758800 (1.056) ((10.0)(0.97)	1	¥ '()
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1					
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	•				
\vdash					
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				·	
			 		
1 1				<u> </u>	1

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

October 13, 2010

LDC Report Date:

December 20, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J150566

Sample Identification

SSAP3-05-1_01_BPC

SSAP3-05-2_01_BPC

SSAP3-05-3_01_BPC

SSAP3-05-4_01_BPC

SSAP3-05-5_01_BPC**

SSAP3-05-2_01_BPCMS

SSAP3-05-2 01 BPCMSD

^{**}Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
10/28/10	¹³ C-OCDD	45.9	SSAP3-05-2_01_BPCMS SSAP3-05-2_01_BPCMSD 0292315-MB	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0292315-MB	0292315-MB 10/19/10 1,2,3,4,6,7,8-F		0.13 pg/g	All samples in SDG
	OCDD		0.45 pg/g	G0J150566

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
SSAP3-05-2_01_BPC	OCDD	1.2 pg/g	1.2U pg/g
SSAP3-05-3_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.12 pg/g 1.8 pg/g	0.12U pg/g 1.8U pg/g
SSAP3-05-4_01_BPC	1,2,3,4,6,7,8-HpCDD	0.56 pg/g	0.56U pg/g
SSAP3-05-5_01_BPC**	1,2,3,4,6,7,8-HpCDD	0.49 pg/g	0.49U pg/g

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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:

		<u></u>			
Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAP3-05-1_01_BPC	¹³ C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAP3-05-2_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 21 (40-135) 25 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAP3-05-3_01_BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	28 (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)	Р
SSAP3-05-4_01_BPC	¹³ C-1,2,3,6,7,8-HxCDD ¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,6,7,8-HxCDF ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 22 (40-135) 12 (40-135) 33 (40-135) 20 (40-135)	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAP3-05-5_01_BPC**	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	27 (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)	Р

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAP3-05-5_01_BPC**	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 G0J150566	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 G0J150566	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J150566

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J150566	SSAP3-05-1_01_BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J150566	SSAP3-05-2_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0J150566	SSAP3-05-3_01_BPC SSAP3-05-5_01_BPC**	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)
G0J150566	SSAP3-05-4_01_BPC	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 OCDF 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	J (all detects) UJ (all non-detects)	P .	Internal standards (%R) (i)
G0J150566	SSAP3-05-5_01_BPC**	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (no 2 nd column confirmation) (o)
G0J150566	SSAP3-05-1_01_BPC SSAP3-05-2_01_BPC SSAP3-05-3_01_BPC SSAP3-05-4_01_BPC SSAP3-05-5_01_BPC***	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J150566	SSAP3-05-1_01_BPC SSAP3-05-2_01_BPC SSAP3-05-3_01_BPC SSAP3-05-4_01_BPC SSAP3-05-5_01_BPC**	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0J150566

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J150566	SSAP3-05-2_01_BPC	OCDD	1.2U pg/g	А	bl
G0J150566	SSAP3-05-3_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.12U pg/g 1.8U pg/g	A	bl
G0J150566	SSAP3-05-4_01_BPC	1,2,3,4,6,7,8-HpCDD	0.56U pg/g	А	bl
G0J150566	SSAP3-05-5_01_BPC**	1,2,3,4,6,7,8-HpCDD	0.49U pg/g	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0J150566

No Sample Data Qualified in this SDG

SDG _abc	#: <u>24524C21</u> 6 #: <u>G0J150566</u> oratory: <u>Test America</u>		ALIDATIO	St	PLETE tage 2l	NES : 3/4	S WORKSHEET		Date: /2// Page: _/of_ Reviewer:
WET	HOD: HRGC/HRMS Diox	kins/D)ibenzofurar	ıs (EPA S\	W 846 N	/lethod	1 8290)		
fhe : /alid	samples listed below were ation findings worksheets	e revi i.	ewed for ea	ch of the fo	ollowing	valida	ation areas. Validatio	on find	lings are noted in attached
	Validation	Area	3				Comm	ents	
.1				Δ	Samplin	dates	10/10/		
11.		erform	ance check	Ā		d ami	·		
III.				A					
IV.				SW	1				
V.	Blanks			SW					
VI.	Matrix spike/Matrix spike du	uplicate	es	SW					
VII.	-			A	LC	ク			
VIII			uality control	N					
IX.				SW					
X.	Target compound identificat	tions		Δ	Not revi	ewed fo	or Stage 2B validation.		
XI.			_S	SVAV			or Stage 2B validation.		
XII.				Δ			or Stage 2B validation.		····
XIII.		-		A					
XIV.	. Field duplicates			N					
XV.	. Field blanks			Ν					·
lote: 'alida	A = Acceptable N = Not provided/applicable SW = See worksheet ted Samples: ** Indicates samp		R = Rins FB = Fie	eld blank	s detected		D = Duplicate TB = Trip blank EB = Equipment blank	k	
1	SSAP3-05-1_01_BPC	11	02922	315	21			31	
2	SSAP3-05-2_01_BPC	12	1		22			32	
3	SSAP3-05-3_01_BPC	13			23		<u> </u>	33	
4	SSAP3-05-4_01_BPC	14			24	<u> </u>		34	
5	SSAP3-05-5_01_BPC**	15			25			35	
6	SSAP3-05-2_01_BPCMS	16			26			36	
7	SSAP3-05-2_01_BPCMSD	17			27			37	
8		18			28			38	
9	·	19			29			39	
10 l	,	20	1		20			40	

Notes:	_	 	
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LDC #: 24524C2/ SDG #: pu comes

VALIDATION FINDINGS CHECKLIST

Page:_	 _of	2-
Reviewer:	 17	
2nd Reviewer:	0	

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

Method: Dioxins/Dibenzofurans (EPA SW 846 Method 8290) 	Ι	1	
Validation Area	Yes	No	NA	Findings/Comments
L.Technical holding times				
All technical holding times were met.			<u> </u>	
Cooler temperature criteria was met.				·
III. GC/MS Instrument performance check		<i>-</i>		
Was PFK exact mass 380.9760 verified?	_			
Were the retention time windows established for all homologues?	_		ļ .	
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		,2,		
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?	hoc	/		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?			<u> </u>	
V. Blanks	12.15.11 12. 16.18	1. 1		
Was a method blank associated with every sample in this SDG?	<u>-</u>			
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			je o 1600. I	
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?				
VIIs Laboratory control samples		mi-iri Mitot		
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#: 24524C2 | SDG#: 14 comm

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
Reviewer: 2nd Reviewer:

				
VIII. Regional Quality Assurance and Quality Control	 	·	1	
Were performance evaluation (PE) samples performed?	<u> </u>		-	
Were the performance evaluation (PE) samples within the acceptance limits?	<u> </u>	2044 140		
IX: Internal standards	(193) T) } -		
Were internal standard recoveries within the 40-135% criteria?	1	[v		
Was the minimum S/N ratio of all internal standard peaks ≥ 10?		<u> </u>		
X: Target compound identification	` ·			
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	/			·
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	/			
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	/			
Did compound spectra contain all characteristic ions listed in the table attached?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	مهميا	/		
Was the signal to noise ratio for each target compound and labeled standard > 2.5?		-	 	
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?			ļ <u>.</u>	
For PCDF identification, was any signal (S/N \geq 2.5, at \pm seconds RT) detected in the corresponding PCDPE channel?	/			
Was an acceptable lock mass recorded and monitored?				
XI: Compound quantitation/CRQLs		1.		
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	_			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XII Systemiperormance:				
System performance was found to be acceptable.		-		,
XIII Goverall assessment of data :				
Overall assessment of data was found to be acceptable.				
XIV. Fieldiduplicates 1.7. 20 (a)				
Field duplicate pairs were identified in this SDG.			-	
Target compounds were detected in the field duplicates.				
XV.Fieldblanks				
Field blanks were identified in this SDG.			-	V
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC# 2452402/

VALIDATION FINDINGS WORKSHEET Routine Calibration

Reviewer:_

2nd Reviewer: 4

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

N N N	N/A W.	as a routine calibra ere all percent diffe d all routine calibrat	Was a routine calibration was performed at the beginning and end of each 'Were all percent differences (%D) of RRFs < 20% for unlabeled compound: Did all routine calibration standards meet the Ion Abundance Ratio criteria?	the beginning and er < 20% for unlabeled er lon Abundance Rat	$\frac{1}{2}$ N N/A Was a routine calibration was performed at the beginning and end of each 12 hour period? Were all percent differences (%D) of RRFs \leq 20% for unlabeled compounds and \leq 30% for labeled? Y/N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?	d? for labeled?	(a)	
) #	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Finding lon Abundance Ratio	Associated Samples	Qualifications	
	01/8401	cen.(closing	13C-0CDD		35)	BH-S/8260	J/41/P que	X
	11.22		0		\	6, 7	, /6/	0
	22:24							-
				•				
							:	
		PCDDs	Selected ions (m/z)	Ion Abundance Ratio	PCDFs	Selected ions (m/z)	(m/z) Ion Abundance Ratio	ltio
	Tetra-		M/M+2	0.65-0.89	Tetra-	M/M+2	0.65-0.89	
	Penta-		M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78	
	Hexa-		M+2/M+4	1.05-1.43	Hexa-	M+2/M+4	1.05-1.43	
	Hexa-13C-HxCDF (IS) only	DF (IS) only	M/M+2	0.43-0.59	Hexa-13C-HxCDF (IS) only	nly M/M+2	0.43-0.59	
	Hepta- ¹³ C-Hp	Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51	Hepta-13C-HpCDF (IS) only	only M/M+2	0.37-0.51	
	Hepta-		M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20	
	Octa-		M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02	

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VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Oc 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". Y N/A

Were all samples associated with a method blank?

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

Was the method blank contaminated? $\frac{Y/N N/A}{B'ank extraction date: \frac{Io}{19}/10}$

Conc. units:

Y N N/A

Blank analysis date: 10/28/10

Associated samples:

Sample Identification 49/4 0.50/4 1.8/1 0.12/4 2 <u>ن</u> بع X 0.65 9.25 OH 5/82600 Blank ID 0.45 Compound O

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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LDC#
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Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

Page: of 2nd Reviewer: N 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 a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water. N N/A

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

17									
اۃ	Date	MS/MSD ID	Compound	MS %R (Limits)	nits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		6+7	3	رم ر	£h1-08)	(()	7	M 942 Les 11
			H	78 ((79-13)7	()	()	A	7 /
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LDC# 2452402/

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: of Z

Reviewer: F

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 M N/A

Are all internal standard peaks > 10?

Y M N/A

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

ৰ্থ মু হ অ K, L, M, NP, E) 9 প্ প্ ø **Check Standard Used** ल, ष J a В Qualifications 3 Recovery Standards 40-135 % Recovery (Limit: 40-135%) 13C-1237,89-HXCDD 13C-1.2.3.4-TCDD ر ح 20 20 77 44 4 35 3 39 K d 4 ۵ Check Standard Used Internal Standard HIG vস ± H|P|Ц ড Lab ID/Reference Internal Standards ٦ 3 13C-123678-HxCDD ¹³C-123,6,7,8-HxCDE ¹³C-1,2,3,7,8-PeCDD ¹³C-1 2 3 7 8-PeCDF 13C-2 3 7 8-TCDD ¹³C-2,3,7,8-TCDF Date

d

LDC# 2452462

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: 20f 2

Reviewer: F

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

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

3	Date	Lab ID/Reference	Internal Standard	% F	% Recovery (Limit: 40-135%)	Qualifications	cations
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)	(
		Internal Standards	Check Standard Used		Recovery Standards	Check	Check Standard Used
⋖	13C-2,3,7,8-TCDF	DF		K. 13C	¹³ C-1.2.3.4-TCDD		
۳	13C-2,3,7,8-TCDD	da		_	¹³ C-1 2 3 7 8 9-HxCDD		
c	13C-12,3,7,8-PeCDE	PECDE		M			
4	13C-12378-PeCDD	PecDD		Z	990000		
щ	13C-1,2,3,6,7,8-HxCDE	-HxCDF		q			
щ	12-12-3,6,7,8-HxCDD	-HxCDD		а			
ď	13C-12,3,4,6,7,8-HpCDE	,8-HpCDF		q			
=	13C-1:2,3,4,6,7,8-HpCDD	8-НрСDD		<u>R</u>			
_	מחייר ילו			- -		_	•

LDC# 24524021

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLS

Page: 6f / Reviewer: FT 2nd Reviewer: 0

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

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

Y N NA

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)				JK detects (K)	me (p)	+				
	Associated Samples	All			ΔI		5	7				
	Finding	All compounds reported below PQL			All compounds reported as EMPC		no seems column	down firmation was performer	1			
l l	Sample ID						#					
	# Date											

Comments: _See sample calculation verification worksheet for recalculations

LDC #: 2 45 246 2/ SDG #: 410 court

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:

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

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

 $A_{is} = Area \ of \ associated internal standard <math>G_{is} = Concentration \ of internal standard s, X = Mean of the RRFs$

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (& と ら std)	RRF (25,3 std)	%RSD	%RSD
-	7001	04/11/6	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1860	186.0	1.05	1.05	8://	8-//
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.032	1.032	901	1.06	8.01	10%
			1,2,3,6,7,8-HXCDD (13C-1,2,3,6,7,8-HXCDD)	////	161.1	-X	\x'	12.	/3.7
,			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.134	1./34	1.26	7.7	ره. ک	12.3
			OCDE (19C-OCDD)	8/1/2	3.11.8	2:36	2-36	6.51	15.3
7	1696	01/14/01	2,3,7,8-TCDF (¹C-2,3,7,8-TCDF)	1.0/573	1.01573	1.02991	166 20:1	5,51093	8,015.8
	•	•	2,3,7,8-TCDD (¹3C-2,3,7,8-TCDD)	1.10816	7/801.1	1.16359	1.16359	0£968.h	4.8%E
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	16951.1	1681-1	19/621	1.23467	/1/eh.h	11/ch.h
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCDD)	1.04588	SWho-1	1.16792	1.16792	8.8336/	8.8336
			OCDE (13C. OCDD)	1890S·1	18905-1	1.63/8	88/cs 9-1	8.83207	8.8320
က	ICAL	0/108/01	10/30//O 2,3,7,8-TCDF (°C-2,3,7,8-TCDF)	1.060	1.060	800./	200./	3.59	3.59
	pons	,	-2-3-7-8-TCDD("C-2;37,8-1CDD)						
			1,2,3,6,7,8-HxCDD (13C-4-2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8+46CDD ("3C-1,2,4,6,7,8,-HpCDD)						
			o est ("c-ocpo)						

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.

245 yez SDG#: LDC #.

Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: Page: 2nd Reviewer:

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

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

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

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF A_x = Area of compound, A_x = Concentration of compound, C_x = Concentration of compound, C_x Where:

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

					Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	a%
7	C1/86/01 ro:h1 NOD	01/24/01	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0.984	16.0	/2.0	7.2	7.7
			2,3,7,8-TCDD (¹³C-2,3,7,8-TCDD)	1.05%	16.0	0.3/	9.//	9//
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.14.1	1.2/	/×/	5:6	5.6
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.134	1.15	5/./	/ :/	/-/
			OCDE ("C-OCDD)	2.//8	7. 92	1.92	9.4	9.4
2	00:22 NOB	01/8/11	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.01573	1.01097	1.01077	5.0	0.5
		,	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.10816	1.15153	65/5/1	3.9	3.9
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.15691	1. 19924	14661.1	3.7	3.7
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.04888	1.09233	1.09233	1.4	1.4
			OCDE (13C-OCDD)	1.5068/	1.49523	1.49523	8.0	S-0
က	aes 19:2/	01/4/11	2,3,7,8-TCDF (1 ³ C-2,3,7,8-TCDF)	1.060	56.0	26.0	1.01	1.01
	0872	~	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

1,241541 ter cons LDC#: SDG #:7

Matrix Spike/Matrix Spike Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

/of/	67	
Page:	Reviewer:	2nd Reviewer:

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

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

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

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

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

MS/MSD samples:

6+7

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

Concentration (P3/9)
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an

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.

ノナントナン SDG#:_ LDC#:

Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

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 = Spiked sample concentration SA = Spike added

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

CD - 518660

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

Spike Spiked Sample Added Concentration	Spiked Sample Concentration				103		TCSD	D.	LCS/I CSD	CSD
Compound	(63	(9)	(63)	0	Percent Recovery	ecovery	Percent Recovery	scovery	RPD	0
	o , sol	l CSD	0 / 801	uso 1	Reported	Recalc	Reported	Racalc	Reported	Recalculated
2,3,7,8-TCDD	0.02	カツ	0.81	MM	76	90				\
1,2,3,7,8-PeCDD	001		7.66		66	26				
1,2,3,4,7,8-HxCDD	001		33.6		126	94				
1,2,3,4,7,8,9-HpCDF	00/	/	201		101	101				
OCDF	200	7	811	ĵ	68	63	NN	\		
							\			
			,							
						·				

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

lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	⊩								
	Accurate mass.	Ol nol	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(s)	on to	Element	
-	303,9016	Σ	O H SCI	1004			ai no:	CIGILIBRIAL COMPOSITION	Analyte
	305.8987	M+2	C.H.*CI.**C10	1001 1001	4	407.7818	M+2	C.,H**Cl,37ClO	HACOR
	315,9419	2		7001		409.7788	M+4	C.H**Cl.*7Cl O	7004
	317,9389	M+2	130, H 80, 970,0	1CDr (8)		417.8250	Σ	12C.H.3CI.O	TOOGL (S)
	319,8965	Σ		(8) AUST		419.8220	M+2	13C, H*CL, 37CIO	(S) Hoods (S)
	321.8936	M+2	C.H. &C. 97C10	2007		423.7767	M+2	C, H C, JOO	T CO
	331,9368	≅	13C H 3C O	TOUD		425.7737	M+4		מיני מיני
	333,9338	Z+2	19C H 35C 37CIO	1CDD (S)		435,8169	M+2	13C H 3C 13C 1	
	375,8364	M+2	- (12.14 CI3. CIO.2	(s) aaa		437.8140	M+4	13C. H3C. H3C. 13C. I	
	[354.9792]	LOCK	(S) (S) (S) (S) (S) (S) (S) (S) (S) (S)	TXCOFT		479.7165	M+4	C.Hachach	(s) DOOD
				<u>-</u>		[430.9728]	LOCK.	O.F.17	T XI
								-	<u>.</u>
2	339,8597	M+2		100					
	341.8567	M+4	C. H &C. 170	recur Propri	ιΩ	441.7428	M+2	G. 3601.37010	7,400
	351,9000	M+2	12.13 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2	Pecur		443.7399		C12 C1 3C1 O	1000
	353,8970	M+4		PeCDF (S)		457.7377			OCD-
	355 8546	: C		PeCDF (S)		459.7348			0000
===	357 8516	Z+7	Cr.H. Colo.	PecDD		469.7780			OCDD
	367 8040	M+1	C124, C1, VC1, O2	PecDD		471 7750			OCDD (S)
	000000	M+2	13C12H3#CI(37CIO2	Pecdo (S)	_	513 677E		_	OCDD (s)
	400.0919	M+4	12C12H128C137C12O2	Pecdo (S)		[422 6278]		<u> </u>	DCDPE
	409.7974	M+2	C ₁ ,H, ³ Cl ³ /ClO	Hoode		[444.34/0]	FOCK	C ₀ F ₁ ,	PFK
	[354.9792]	LOCK	C. F. C.	PEK TOTAL	_			= 2	
					•			_	
е	373,8208	M+2	H 3000	100	-}- 				
-	375.8178		C T 30 0 0	TXCO					
-	383.8639		12 12 C2 C3 C3 C3 C3 C3 C3 C3 C3 C3 C3 C3 C3 C3	HXCDF					
-	385,8610		130 L 30 200	HXCDF (S)					
	389.8156			HXCDF (S)					
_	391,8127			HXCDD					
	401.8559		12 C C C C C C C C C C C C C C C C C C C	HXCDD					
,	403.8529			HxCDD (S)					
	445.7555		1,522,522,522,522,522,522,522,522,522,52	HxCDD (s)					-
	[430,9728]		0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m 0.0 m	OCDPE					
	•		-1-	PFK -		•			

The following nuclidic masses were used:

ø

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 #:	24	524cz]
		cover

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:	/_of/
Reviewer:	FT
2nd reviewer:	8)

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

/ Y /N N/A Y/ N N/A Were all reported results recalculated and verified for all level IV samples?

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

Concentration = (A)(I,)(DF)
(A_k)(RRF)(V_o)(%S)

A_x = Area of the characteristic ion (EICP) for the compound to be measured

A_k = Area of the characteristic ion (EICP) for the specific internal standard

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

V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).

RRF = Relative Response Factor (average) from the initial

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. #4 , OCDF

Conc. = (/33 44.8 \(4000)()()

28 9 58/. 66) (1.50 7)(10.19)(0.95)

13 pg/g

			<u> </u>		<u> </u>
			Reported	Calculated	
		0	Concentration	Concentration	0121
#	Sample ID 2	2,7 X Compound	()	()	Qualification
	#4 T		erro)		
		361446304(1.0	56) (10.19) (0.954)	
				<u> </u>	
			= 0.84 pg	Vg	
				<u> </u>	
				· · · · · · · · · · · · · · · · · · ·	
				,	

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

October 14, 2010

LDC Report Date:

December 20, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J170404

Sample Identification

SSAL4-04-2_01_BPC SSAL4-04-3_01_BPC

SSAL4-04-4 01 BPC**

^{**}Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
10/28/10	¹³ C-OCDD	45.9	All samples in SDG G0J170404	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0292315-MB	10/19/10	1,2,3,4,6,7,8-HpCDD OCDD	0.13 pg/g 0.45 pg/g	All samples in SDG G0J170404

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

Sample	Compound	Reported Concentration	Modified Final Concentration	
SSAL4-04-3_01_BPC	OCDD	1.1 pg/g	1.1U pg/g	
SSAL4-04-4_01_BPC**	1,2,3,4,6,7,8-HpCDD	0.56 pg/g	0.56U pg/g	

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAL4-04-2_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 29 (40-135) 30 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAL4-04-4_01_BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,6,7,8-HxCDF ¹³ C-1,2,3,4,6,7,8-HpCDF	16 (40-135) 9.9 (40-135) 35 (40-135) 14 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 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 1,2,3,4,6,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAL4-04-4_01_BPC**	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 G0J170404	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 G0J170404	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J170404

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J170404	SSAL4-04-2_01_BPC SSAL4-04-3_01_BPC SSAL4-04-4_01_BPC**	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Continuing calibration (%D) (c)
G0J170404	SSAL4-04-2_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J170404	SSAL4-04-4_01_BPC**	1,2,3,4,6,7,8-HpCDD OCDD OCDF 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	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J170404	SSAL4-04-4_01_BPC**	2,3,7,8-TCDF	None	P	Project Quantitation Limit (no 2 rd column confirmation) (o)
G0J170404	SSAL4-04-2_01_BPC SSAL4-04-3_01_BPC SSAL4-04-4_01_BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J170404	SSAL4-04-2_01_BPC SSAL4-04-3_01_BPC SSAL4-04-4_01_BPC**	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 G0J170404

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J170404	SSAL4-04-3_01_BPC	OCDD	1.1U pg/g	A	bl
G0J170404	SSAL4-04-4_01_BPC**	1,2,3,4,6,7,8-HpCDD	0.56U pg/g	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0J170404

No Sample Data Qualified in this SDG

Tronox Northgate Henderson VALIDATION COMPLETENESS WORKSHEET

LDC #:__24524D21 SDG #: G0J170404 Stage 2B/4 Laboratory: Test America

Date:	12/16/1
Page:_	
Reviewer:	£2
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
l.	Technical holding times	Δ	Sampling dates: 10 14 10
11.	HRGC/HRMS Instrument performance check	Δ	
111.	Initial calibration	Δ	
IV.	Routine calibration/l €V	5 W	
V.	Blanks	ريسا	
VI.	Matrix spike/Matrix spike duplicates	SW	SSAP3 -05-2_U1_B8C MS/D
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SIA	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	s W	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	Ν	
XV.	Field blanks	\mathcal{N}	

Note: A = Acceptable

N = Not provided/applicable

SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank EB = Equipment blank

Validated Samples: ** Indicates sample underwent Stage 4 validation

	SOIL	····			
1	SSAL4-04-2_01_BPC	11	0292315-MB	21	31
2	SSAL4-04-3_01_BPC	12	,	22	32
3	SSAL4-04-4_01_BPC**	13		23	33
4		14		24	34
5		15		25	35
6		16		26	36
7		17		27	37
8		18		28	38
9		19		29	39
10		20		30	40

Notes:	

LDC #: 2452402) SDG #: pu cones

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
ITechnical holding times	7 - ""		····	
All technical holding times were met.				
Cooler temperature criteria was met.	_			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers < 25% ?				
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?				
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Inittal calibration	· · · · · · · · · · · · · · · · · · ·			
Was the initial calibration performed at 5 concentration levels?	<u></u>			
Were all percent relative standard deviations (%RSD) \leq 20% for unlabeled standards and \leq 30% for labeled standards?				
Did all calibration standards meet the Ion Abundance Ratio criteria?				
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?				
and internal standard ≥ 10? IV: Continuing calibration	1	· /*		
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	3 145 2000 m	i aj didig Barasa	SELE Service	
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			, de soj t Vojeroza	
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?	red contacts		_	belief v 2. ** ve v
VII Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC#: 2452402/ SDG#: ucom

VALIDATION FINDINGS CHECKLIST

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

				
VIII. Regional Quality Assurance and Quality Control		· · · · ·		
Were performance evaluation (PE) samples performed?	ļ	<u> </u>	_	
Were the performance evaluation (PE) samples within the acceptance limits?				
IX: Internal standards			か(数) 数19 2 1	
Were internal standard recoveries within the 40-135% criteria?	١.		1	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?	<u> -</u>	<u>t</u>	<u>L</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?		ļ 		
Did compound spectra contain all characteristic ions listed in the table attached?	/			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	Area.		ļ	
Was the signal to noise ratio for each target compound and labeled standard ≥ 2.5?				_
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?				
For PCDF identification, was any signal (S/N \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	ja ja		1 14 - 12	
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	_			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XII System performance				
System performance was found to be acceptable.		-		,
XIII-Overall assessment ordata:				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XV:Fieldiblanks				
Field blanks were identified in this SDG.			_	AND MADE TO THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PARTY OF THE PA
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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LDC#:

VALIDATION FINDINGS WORKSHEET Routine Calibration

2nd Reviewer: Reviewer._

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

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

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

Y (N N/A)

Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled?

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Ratio		
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						Table 1		
*	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications	
	0/84/01	eev (clasing)	136-0000)	A1/	7400 d/5n/s	6,0
	hire	0		•				
				-				
								Ī
						•		
		PCDDs Se	Selected ions (m/z)	lon Abundance Ratio	PCDFs	Selected ions (m/z)	/z) Ion Abundance Ratio	Ratio
	Tetra-		M/M+2	0.65-0.89	Tetra-	M/M+2		
	Penta-		M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78	
	Hexa-		M+2/M+4	1.05-1.43	Hexa-	M+2/M+4	1.05-1.43	
	Hexa-13C-Hx	Hexa- ¹³ C-HxCDF (IS) only	M/M+2	0.43-0.59	Hexa- ¹³ C-HxCDF (IS) only	M/M+2	0.43-0.59	
	Hepta-13C-H	Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51	Hepta-13C-HpCDF (IS) only	y M/M+2	0.37-0.51	
	Hepta-		M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20	,
	Octa-		M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02	

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VALIDATION FINDINGS WORKSHEET

2nd Reviewer: Reviewer:

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

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? If $\frac{10}{19}$ Blank analysis Blank extraction date: 10 Y N N/A X X/V

Blank analysis date:

Associated samples:

		7	- -	_	,		1		;											_	
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ample Identifica																					
S	3	b/25.0	ļ																		
	2	1	1.1/4	,		•				_											
								:													
	МВ	0.65	×.×																		
Blank ID	2182620	0./3	0.45																	;	
		F	6																		
	Blank ID	Blank ID	Blank ID 2 3 3 0.05 - 0.55/4	Blank ID 2 3 3 0.05 0.45 0.45 0.50/4 - 0.50/4 -	Blank ID	Blank ID	Blank ID Sample Identification 0.2 3	Blank ID Sample Identification Sample Identification	Blank ID 0.0923/5/4/8 0.05 0.45 0.45 0.45 0.45 0.1/4 0.50/4 1.1/4 0.50/4	8 Blank ID Sample Identification 8 0.09 2 3/5	8 Blank ID Sample Identification 8 0.0 9 2.3	81ank ID Sample Identification 0.09	Blank ID Sample Identification 0.243 0.65 0.52/4 - 0.52/4 0.45 2.35 1.1/4 0.52/4 - 1.1/4 0.52/4	Bilank ID Sample Identification Sample Identification	8 Jank ID Sample Identification Sample Identification 0.09	Blank ID Sample Identification Sample Identification 0.09 a 2/5	Blank ID Sample Identification C2 3	Blank ID Sample Identification Sample Identification	8 Blank ID 0.03 0.05 - 0.55 /y 0.45 2.35	8 Blank ID Sample Identification 0.13 0.05 - 0.55 \/ 0.45 2.3 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1.1 \/ 1	Blank D Sample Identification 0.73 0.05 0.45 3.75 1.1/4 - 0.45 3.75 1.1/4 - 2.50/4 - 1.1/4 - 2.50/4 - 3.75 1.1/4 3.75 1.1/4 3.75 1.1/4 4.1/4 - 5.50/4 - 6.75 3.75 1.1/4 - 2.50/4 - 3.75 1.1/4 5.50/4 - 6.75 3.75 1.1/4 - 5.50/4 - 6.75 3.75 1.1/4 - 6.75 - 7.50/4 - 8.25/4 - 9.75 - 1.1/4 - 1.1/4 - 1.1/4 - 1.1/4 - 1.1/4 - 1.1/4 - 1.1/4 - </td

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".

2 drz shz LDC #:

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

2nd Reviewer:. Reviewer: FT Page:

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

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

Not applicable questions are identified as "N/A".

Not applicable questions are identified as "N/A".

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

Note the MS/MSD analyzed every 20 samples of each matrix?

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

	<u></u>	ī	ī		T	$\overline{}$	T		IT	T	ī	T	<u> </u>	T	_	_	_	.			1	Г	_
Qualifications	1-16 cm	//								10.1													
Associated Samples	J. 24																						
RPD (Limits)	()	()	()	()	^	()	()	,	((]()) (()	^	^	î	(((^	^	(î
RPD (
MSD ' %R (Limits)	()	()	()	()	()	()	()	()	<u> </u>	()	()	()	()	()	(()	()	()	()	()	()	()	(
		7															. :			-	-		
MS (Limits)	EH-08)	78 (79-137	, , , ,	(()	(()	,	(((()	()	()	()	()	(()	()	()	()	()	(
MS %R (Limits)	72,	78																					
Compound	Ē	H	-							1			:						,		-		
DI DE	55AP3-05-2	OLBRASID																					
Date																							
*																							

LDC # 2452402/

VALIDATION FINDINGS WORKSHEET

Reviewer: F7

Page:

Internal Standards

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

Y N/A

Are all internal standard peaks > 10?

Was the S/N ratio all internal standard peaks > 10? METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

*	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135%)	t: 40-135%)	Qualifi	Qualifications
		1	H		33	(40-135)	d1 54/5	quel F
			Ī		29	^		60
			87		30	3	7	9.6
						()		
		3	Н		9/	(SE/-ah)	9/64/E	F
			I		6.6	(/		6,0
		•	Ħ		35	()	7	K. L. M. N
			6	i i	14	,	7	0.0
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			7 1.7 207	į		()		
		,				()		
						()		
		Internal Standards	Check Standard Used		Re	Recovery Standards	Chec	Check Standard Used
4	¹³ C-2.3.7.8-TCDF)F		됨	13C-1.2.3.4-TCDD			
ď	13C-2.3.7.8-TCDD	dc		_	13C-123789-HxCDD	CDD		
d	13C-12378-PeCDF	ACDF		Σ				
٩	13C-1,2,3,7,8-PeCDD	аСDD		z				
щ	13C-12,3,6,7,8-HXCDE	HXCDE		d				
ч	13C-1,2,3,6,7,8-HxCDD	НХСПП		۵				
d	13C-123467.8-HpCDE	8-HpCDF		<u>d</u>	****			
=	3C-1,2,3,4,6,7,8-HpCDD	8-НрСПП	:	<u>R</u>				
- =	130,000			_			_	

LDC#: 24 52402/

Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: _ Reviewer:

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

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

Y M 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).

	i		_	7	 	~~~	 			==	 	_	
	Qualifications	J/A detects (sp)				(k)		(4) 0/ 000	+ +				
	Associated Samples	All				All		6					
	Finding	All compounds reported below PQL		-		All compounds reported as EMPC		no mal column	confirmation was	rustine			
	compel Sampte to							H					
	Date												
L	*												

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24524D2) SDG#: 2450001

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification



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

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

 $\label{eq:RRF} RRF = (A_{\rm s})(C_{\rm ts})/(A_{\rm ts})(C_{\rm s})$ average RRF = sum of the RRFs/number of standards %RSD = 100 * (S/X)

 $A_x = Area$ of compound, $A_s = Area$ $C_x = Concentration of compound, <math>G_s = Con$ S = Standard deviation of the RRFs, <math>X = Mean

A_{is} = Area of associated internal standard
G_{is} = Concentration of internal standard
X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (Can std)	RRF (~ \$ \$std)	%RSD	%RSD
-	eat 14:23	CH24fes	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.784	6860	7.05	1.05		8 //
	LCAL	9/14/10	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8.	8.01
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1611	1/4/-1	1.2	12/	/3./	12.7
			1,2,3,4,6,7,8-HpCDD (*3C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	75.1	126	72.3	1.5
			OCDE (13C. OCDD)	2.118	3.118	2.36	2.36	15-3	5:3
7			2,3,7,8-TCDF (13C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (1°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)						
ო			2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)						
		•	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)						
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF ("C-OCDD)						

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

24524021 SDG#: LDC#

Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: Page: 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 = $(A_{\lambda})(C_{\mu})/(A_{\lambda})(C_{\lambda})$

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF $A_x = Area$ of compound, $A_{ss} = C_{ss} = Concentration of compound, <math>C_{ss} = C_{ss}$ Where:

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

					Reported	Recalcutated	Reported	Recalcutated
#±	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q %	Q%
-	aeV 14:22	CH24/01	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	4860	16.0	16.0	7.7	7.2
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	16.0	0.9/	11.6	7://
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	161.1	/٣/	/7./	9:5	25
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1./34	5/./	51.1	1.1	/ '/
			OCDE (13C-OCDD)	3.11.8	16./	76:1	7.6	2.6
2			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)					
			OCDE (¹3C-OCDD)					
ť			2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (¹³ C-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#_ 0/3 1771/ SDG#_ 44_ const

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Reviewer: C2

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 Wh

Where: SSC = Spiked sample concentration SA ** Spike added

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

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 02923/5-105

	<u> </u>	<u></u>	- 1			— т			 		
CS/I CSD	RPD	Recalculated					,				
1831	æ	Reported									
ď	Secovery	Recalc								•	
ICSD	Percent Recovery	Reported					NA	\			
CS	Recovery	Recalc	96	66	16	Lol	68				
21	Percent Recovery	Reported	90	66	h6	107	68				
ample	ncentration (09/97	l csn	NA	*			7				
Spiked S	Concentration (09 / 4)	/ N	/ק.0	2.66	23.6	201	7.18				
ike	Added (129/97)	/ /	44	1			3				
S	A SC	108	D. Oc	001	001	001	200			,	
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	ocpf				-

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	of Accurate mass(8)	Ol not	ū						
	╢┈	a lipr	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	fon ID	Elemental Composition	Andrea
_	303,9016	Σ	O.H.ªCI.O	1005				Housedings manager	Analyte
	305.8987	M+2	C;H,*Ci,*C10	TCDF	4	407.7818	M+2	C ₁₂ H ³⁵ CI _k 37CIO	HDCDF
	315.9419	≊	0.0%.H.30.0	1000		409.7788	M+4	C,H2CL17CLO	T C C
	317,9389	M+2	13C, H. 43Cl, 27ClO	TCDE (8)		417.8250	Σ	¹³ C,H ³ CI,O	HUCUE (8)
	319.8965	Σ	0.08.H.Q	(S) COT		419.8220	M+2	13C12H**C1,37CIO	HDCDF
	321.8936	M+2	O.H. 301.3010	200		423.7767	M+2	C,H ³⁵ Cl, ³⁷ ClO,	H
	331.9368	≥	13C H 3C O	1007		425.7737	M+4	C.H.*C.J.	2004
<u>-</u>	333,9338	V+2	13: 4 0.402 130 H 3501 37010	1000 (S)		435,8169	M+2	13C. H ³² C. 37CIO	
	375,8364	M+2	C H 801 370	1CDD (S)		437.8140	M+4	13CH ³² C37C.I	(s)
	[354,9792]	, X		HXCDPE		479.7165	M+4	C.Haclaci	Mpcou (s)
	-			٧		[430.9728]	Lock	C ₂ F ₁ , C ₂ F ₁	PFK
(•		:	-
	339,8597	M+2	C, H, *C *C O	Pache					
	341.8567	M+4	C,D, H, acli, acl, o	Pecne	n	441./428		C ₁₂ **CI ₂ **CIO	OCDF
	351.9000	M+2	1"C,H, "C1,0"C10	Pache (a)		446.7689		C;2*C ,**C ,*O	OCDF
 -	353.8970	M+4	1.0 H, 3.0 L, 3.0 L, 0	Pecine (9)		457,7377		C ₁₂ **Cl, **ClŌ,	ocpo
<u> </u>	355,8546	M+2	C,H, scl, sclo,	Pecino (v)		459,7348		C,3"C ,3"C ,0,	OCDD
	357.8516	M+4	C,H,**Cl,**Cl,O,	Pacho		469.7780			OCDD (S)
_	367.8949	M+2	13C, H, 35CJ, 37CjO,	Pecho (s)		06//.1/4			OCDD (S)
	369.8919	M+4	13C, H, 35Cl, 37Cl, O,	Pechn (S)		513.6775			DCDPE
	409.7974	M+2	C.H. "Cl. "ClO	Harring (a)		[422.9278]	LOCK	ر برور برور	PFK
	[354.9792]	LOCK		H Y Y		_		-	
					•	_			
e	373.8208	M+2	C H 3001 37010	100					
==	375.8178		C.H.*CI9CIO	יאכטאר דמסאדו					
	383,8639		12: 2 04 02 13: H 3C: I	יייייייייייייייייייייייייייייייייייייי	-				
	385.8610		13C, H. 3CI, 37CIO	(8) LL (1)					
	389.8156	M+2	C.H.*CI.*CIO	(s) 40041					
	391,8127		C,H,*C,*C,O,	2000					
	401.8559		¹³ C ₁₂ H, ³³ Cl ₁ 3, ³ ClO ₃	HXCDD (S)			•	_	
	403,8529		"C,"H, "CI, o',	HXCDD (S)					
	445.7555		C ₁₂ H ₂ **Cl ₆ **Cl ₂ O *	OCDPE					
	[430.8728]	Lock	C,F1,	PFK					

The following nuclidic masses were used:

®

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

O = 15.934915 $^{36}Cl = 34.968853$ $^{37}Cl = 36.965903$

S = internal/recovery standard

LDC #: 24	5 24 D2)
SDG #: Ju	

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

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

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

/	Y	N	N/A
	Υ/	N	N/A

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

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

Conce	entratio	$n = (A_{\bullet})(I_{\bullet})(DF)$ $(A_{\bullet})(RRF)(V_{\bullet})(%S)$	Example:
A _x	=	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D. #3 . 2, 3, 7, 8
A _k	=	Area of the characteristic ion (EICP) for the specific internal standard	100/2
l _x	R	Amount of internal standard added in nanograms (ng)	Conc. = (364868) (2000) (268591000 0.98) (10.0
V.	=	Volume or weight of sample extract in milfiliters (ml) or grams (g).	70.0
RRF	=	Relative Response Factor (average) from the initial calibration	= 0.30 pg/g
Df	=	Dilution Factor.	, , ,
%S	=	Percent solids, applicable to soil and solid matrices only.	

#	Sample ID	Сотроили	Reported Concentration ()	Calculated Concentration ()	Qualification
	V				

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

September 27, 2010

LDC Report Date:

December 23, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J200489

Sample Identification

SSAN6-08-1BPC

Introduction

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

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

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

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
0298269-MB	10/25/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.12 pg/g 0.32 pg/g 0.088 pg/g 0.13 pg/g	All samples in SDG G0J200489

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-08-1BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD	38 (40-135) 22 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	Р

X. Target Compound Identifications

Raw data were not reviewed for this SDG.

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN6-08-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J200489	All compounds reported below the PQL.	J (all detects)	А

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J200489	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 G0J200489

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J200489	SSAN6-08-1BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF	J (all detects) UJ (all non-detects)	P	Internal standards (%R)
G0J200489	SSAN6-08-1BPC	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0J200489	SSAN6-08-1BPC	All compounds reported below the PQL.	J (all detects)	А	Project Quantitation Limit (sp)
G0J200489	SSAN6-08-1BPC	All compounds reported as EMPC	JK (all detects)	А	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0J200489

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0J200489

No Sample Data Qualified in this SDG

	: 24524E21 : G0J200489		N COMF		enderson SS WORKS	HEET	Date: /2//7
	atory: <u>Test America</u>	_		nage 2D			Reviewer: #7
	OD: HRGC/HRMS Diox	kins/Dibenzofurar	ns (EPA S\	W 846 Meth	nod 8290)		Page: _/of _/ Reviewer:
The sa validat	amples listed below were ion findings worksheets	e reviewed for ea	ach of the f	ollowing val	idation areas. \	Validation findin	ngs are noted in attached
	Validation	Area				Comments	
1.	Technical holding times		Δ.	Sampling da	tes: 9/27	1/10	
II.	HRGC/HRMS Instrument po	erformance check	Δ		1	<u>, </u>	
101.	Initial calibration		A				
IV.	Routine calibration/I CV →		Д				
V	Blanks		SW			·	
VI.	Matrix spike/Matrix spike du	plicates	S #/	55A P ?	5-03-9-0	1-BPMS/12	(no Ass. sample
VII.	Laboratory control samples		Α	les			, ,
VIII.	Regional quality assurance	and quality control	N				
IX.	Internal standards		SW			·	
X.	Target compound identification	tions	N				
XI.	Compound quantitation and	CRQLs	N				
XII.	System performance	•	SW				,
XIII.	Overall assessment of data		A				
XIV.	Field duplicates		N				
XV.	Field blanks		N/				
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet	R = Rin	lo compound nsate ield blank	s detected	D = Duplicat TB = Trip bl EB = Equipr	ank	
/alidate	d Samples: くりし	·				·	
1 5	SSAN6-08-1BPC	11		21		31	
2		12		22		32	
3	,	13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:_

VALIDATION FINDINGS WORKSHEET

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 1613B)

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	а. осрғ	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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3
#

VALIDATION FINDINGS WORKSHEET

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?

Was the method blank contaminated? $\frac{Y/N \text{ N/A}}{\text{Was the method blan}}$ Was the method blandark extraction date: $\frac{IO/3\mathcal{E}}{IO}$ A N N/A N/A

Blank analysis date: $10/{
m W}/10$

Associated samples:

Sample Identification 0.65 0.44 ٥٥ و 0298269 MP Blank ID 0.12 0.32 0.08% 51.0 Compound φ Ц D Ø Conc. units: ַ

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

LDC# 24 Sty Per/

VALIDATION FINDINGS WORKSHEET

Internal Standards

2nd Reviewer:

Reviewer: FT

Ø

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

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

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

タネア Qualifications 861-04 % Recovery (Limit: 40-135%) 72 W X Internal Standard Was the S/N ratio all internal standard peaks > 10? Lab ID/Reference Date Y N N/A

	Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
⋖	¹³ C-2.3.7.8-TCDF		¥	¹³ C-1,2,3,4-TCDD	
ш	13C-2.3.7 8-TCDD		_	¹³ C-1 2 3 7 8 9-HxCDD	
C			M		
O	13C-12378-PeCDD		Z		
ш			0		
Ľ	_		<u>а</u>		
G	_		G		
I	_		R	,	
-	יויים ייני				

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). Y N N/A

Qualifications	J/A detects (sp)		JK detects (k)	1/Per (C)			
Associated Samples	All		All				
Finding	All compounds reported below PQL		All compounds reported as EMPC	xid and Range			
Compod Sample ID.				F G, H, T, J, K, L, M,	\document{\document}{2}		
# Date							

Comments: See sample calculation verification worksheet for recalculations

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

October 22, 2010

LDC Report Date:

December 19, 2010

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J230497

Sample Identification

SSAP3-03-1_01_BPC

SSAP3-03-5_01_BPC

SSAP3-03-9_01_BPC

SSAP3-04-1_01_BPC

SSAP3-04-1_01_BPC_FD

SSAP3-04-5_01_BPC

SSAP3-04-9_01_BPC

SA47-1_01_BPC

SA47-2_01_BPC

SA47-3_01_BPC SA47-4_01_BPC

SA47-4 01 BPC FD

SA47-5 01 BPC

SA47-6_01_BPC

SA47-7_01_BPC

SA47-8_01_BPC**

EB-10222010-RZC

SSAP3-03-9_01_BPCMS

SSAP3-03-9_01_BPCMSD

^{**}Indicates sample underwent Stage 4 review

Introduction

This data review covers 18 soil samples and one water sample listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8290 for Polychlorinated Dioxins/Dibenzofurans.

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
10/28/10	¹³ C-OCDD	45.9	0298269-MB	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0298247-MB	10/25/10	OCDD 1,2,3,4,6,7,8-HpCDF	12 pg/L 2.7 pg/L	All water samples in SDG G0J230497
0298269-MB	10/25/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.12 pg/g 0.32 pg/g 0.088 pg/g 0.13 pg/g	All soil samples in SDG G0J230497

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-10222010-RZC	2010-RZC OCDD 1,2,3,4,6,7,8-HpCDF		8.1U pg/L 3.3U pg/L
SSAP3-03-1_01_BPC	1,2,3,4,6,7,8-HpCDD	0.59 pg/g	0.59U pg/g
SSAP3-03-9_01_BPC	SSAP3-03-9_01_BPC 1,2,3,4,6,7,8-HpCDF		0.39U pg/g
SSAP3-04-5_01_BPC		0.17 pg/g 0.1 0.29 pg/g 0.29	
SSAP3-04-9_01_BPC	OCDD	0.32 pg/g	0.32U pg/g
SA47-2_01_BPC	OCDD	0.83 pg/g	0,83U pg/g
SA47-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.24 pg/g 0.79 pg/g	0.24U pg/g 0.79U pg/g
SA47-4_01_BPC_FD	OCDD	1.3 pg/g	1.3U pg/g
SA47-5_01_BPC	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.25 pg/g 0.27 pg/g 0.48 pg/g	0.25U pg/g 0.27U pg/g 0.48U pg/g
SA47-6_01_BPC	1,2,3,4,6,7,8-HpCDD	0.32 pg/g	0.32U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
SA47-7_01_BPC	OCDD	0.40 pg/g	0.40U pg/g
SA47-8_01_BPC**	1,2,3,4,6,7,8-HpCDF	0.22 pg/g	0.22U pg/g

Sample EB-10222010-RZC was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-10222010-RZC	10/22/10	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	8.1 pg/L 3.3 pg/L 10 pg/L	All soil samples in SDG G0J230497

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

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS percent recovery (%R) was not within QC limits for one compound, the MSD and 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 with the following exceptions:

LCS ID_	Compound	%R (Limits)	Associated Samples	Flag	A or P
0298247-LCS	1,2,3,6,7,8-HxCDF 1,2,3,4,7,8,9-HpCDF	134 (76-133) 131 (83-130)	All water samples in SDG G0J230497	J+ (all detects) J+ (all detects)	Р

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
SSAP3-03-1_01_BPC	¹³ C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAP3-03-5_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 18 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Ф
SSAP3-03-9_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 20 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAP3-04-1_01_BPC	¹³ C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAP3-04-1_01_BPC_FD	¹³ C-1,2,3,7,8-PeCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 27 (40-135) 37 (40-135)	1,2,3,7,8-PeCDD 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
SSAP3-04-9_01_BPC	¹³ C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA47-1_01_BPC	¹³ C-OCDD	32 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA47-2_01_BPC	^{t3} C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA47-3_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 14 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SA47-4_01_BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 36 (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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SA47-4_01_BPC_FD	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 23 (40-135) 33 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SA47-6_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 17 (40-135) 39 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SA47-7_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 18 (40-135) 39 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SA47-8_01_BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 16 (40-135) 36 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-5_01_BPC SSAP3-04-9_01_BPC SA47-2_01_BPC SA47-5_01_BPC SA47-5_01_BPC SA47-8_01_BPC**	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J230497	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 G0J230497	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 SSAP3-04-1_01_BPC and SSAP3-04-1_01_BPC_FD and samples SA47-4_01_BPC and SA47-4_01_BPC_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentration (pg/g)					
Compound	SSAP3-04-1_01_BPC	SSAP3-04- 1_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDD	0.52U	0.16	_	0.36 (≤0.52)	-	-
1,2,3,7,8-PeCDD	2.6U	0.36	-	2.24 (≤2.6)	-	-
1,2,3,4,7,8-HxCDD	2.6U	0.26	-	2.34 (≤2.6)	-	-
1,2,3,6,7,8-HxCDD	0.30	0.36	-	0.06 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	0.37	0.70	-	0.33 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	0.77	2.1	-	1.33 (≤2.6)	-	-
OCDD	3.6	5.0	-	1.4 (≤5.2)	-	-

	Concentra	ation (pg/g)		-		
Compound	SSAP3-04-1_01_BPC	SSAP3-04- 1_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
2,3,7,8-TCDF	1.8	3.3	-	1.5 (≤0.52)	<u>-</u>	-
1,2,3,7,8-PeCDF	1.5	3.5	-	2 (≤2.6)	-	-
2,3,4,7,8-PeCDF	0.77	2.0	-	1.23 (≤2.6)	-	-
1,2,3,4,7,8-HxCDF	2.3	7.3	-	5 (≤2.6)	J (all detects)	Α.
1,2,3,6,7,8-HxCDF	1.8	5.1	-	3.3 (≤2.6)	J (all detects)	А
2,3,4,6,7,8-HxCDF	0.51	1.3	-	0.79 (≤2.6)		-
1,2,3,7,8,9-HxCDF	0.37	0.99	-	0.62 (≤2.6)	_	-
1,2,3,4,6,7,8-HpCDF	6.7	25	-	18.3 (≤2.6)	J (all detects)	Α
1,2,3,4,7,8,9-HpCDF	3.1	12	-	8.9 (≤2.6)	J (all detects)	А
OCDF	24	130	_	106 (≤5.2)	J (all detects)	Α

	Concentr	ation (pg/g)				
Compound			RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,6,7,8-HxCDD	2.6U	0.15	_	2.45 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	2.6U	0.27	-	2.33 (≤2.6)		-
1,2,3,4,6,7,8-HpCDD	0.24	2.4	-	2.16 (≤2.6)	-	-
OCDD	0.79	1.3	-	0.51 (≤5.2)	-	-
2,3,7,8-TCDF	0.51	0.69		0.18 (≤0.52)	•	-
1,2,3,7,8-PeCDF	0.41	0.55	-	0.14 (≤2.6)	-	-
2,3,4,7,8-PeCDF	0.26	0.21	-	0.05 (≤2.6)	٠.	-
1,2,3,4,7,8-HxCDF	0.71	0.63	-	0.08 (≤2.6)	-	-
1,2,3,6,7,8-HxCDF	0.37	0.29	-	0.08 (≤2.6)	· -	-
2,3,4,6,7,8-HxCDF	0.22	0.096	-	0.124 (≤2.6)	-	-

	Concentr					
Compound	SA47-4_01_BPC	SA47-4_01_BPC_FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,7,8,9-HxCDF	2.6U	0.15	-	2.45 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDF	1,4	1.2	-	0.2 (≤2.6)	-	-
1,2,3,4,7,8,9-HpCDF	0.62	0.45	-	0.17 (≤2.6)	-	-
OCDF	3.9	2.8	-	1.1 (≤5.2)	-	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J230497

r			,		
SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J230497	EB-10222010-RZC	1,2,3,6,7,8-HxCDF 1,2,3,4,7,8,9-HpCDF	J+ (all detects) J+ (all detects)	Р	Laboratory control samples (%R) (I)
G0J230497	SSAP3-03-1_01_BPC SSAP3-04-1_01_BPC SSAP3-04-9_01_BPC SA47-1_01_BPC SA47-2_01_BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J230497	SSAP3-04-1_01_BPC_FD	1,2,3,7,8-PeCDD 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)
G0J230497	SA47-4_01_BPC	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)
G0J230497	SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SA47-3_01_BPC SA47-4_01_BPC_FD SA47-6_01_BPC SA47-7_01_BPC SA47-8_01_BPC**	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J230497	SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-5_01_BPC SSAP3-04-9_01_BPC SA47-2_01_BPC SA47-5_01_BPC SA47-7_01_BPC SA47-8_01_BPC**	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (no 2 rd column confirmation) (o)
G0J230497	SSAP3-03-1_01_BPC SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD SSAP3-04-5_01_BPC SSAP3-04-9_01_BPC SA47-1_01_BPC SA47-1_01_BPC SA47-2_01_BPC SA47-3_01_BPC SA47-4_01_BPC SA47-4_01_BPC_FD SA47-5_01_BPC SA47-6_01_BPC SA47-7_01_BPC SA47-7_01_BPC SA47-7_01_BPC SA47-8_01_BPC SA47-8_01_BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J230497	SSAP3-03-1_01_BPC SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD SSAP3-04-5_01_BPC SSAP3-04-9_01_BPC SA47-1_01_BPC SA47-2_01_BPC SA47-2_01_BPC SA47-4_01_BPC_FD SA47-4_01_BPC_FD SA47-5_01_BPC SA47-6_01_BPC SA47-6_01_BPC SA47-6_01_BPC SA47-8_01_BPC SA47-8_01_BPC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0J230497	SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0J230497

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J230497	EB-10222010-RZC	OCDD 1,2,3,4,6,7,8-HpCDF	8.1U pg/L 3.3U pg/L	Α	ы
G0J230497	SSAP3-03-1_01_BPC	1,2,3,4,6,7,8-HpCDD	0.59U pg/g	Α	Ы
G0J230497	SSAP3-03-9_01_BPC	1,2,3,4,6,7,8-HpCDF	0.39U pg/g	Α	bl
G0J230497	SSAP3-04-5_01_BPC	1,2,3,4,6,7,8-HpCDF OCDF	0.17U pg/g 0.29U pg/g	А	bl
G0J230497	SSAP3-04-9_01_BPC	OCDD	0.32U pg/g	Α	∙bl
G0J230497	SA47-2_01_BPC	OCDD	0.83U pg/g	А	bl
G0J230497	SA47-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.24U pg/g 0.79U pg/g	А	ы
G0J230497	SA47-4_01_BPC_FD	OCDD	1.3U pg/g	Α	bl
G0J230497	SA47-5_01_BPC	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.25U pg/g 0.27U pg/g 0.48U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J230497	SA47-6_01_BPC	1,2,3,4,6,7,8-HpCDD	0.32U pg/g	Α	bl
G0J230497	SA47-7_01_BPC	OCDD	0.40U pg/g	Α	bl
G0J230497	SA47-8_01_BPC**	1,2,3,4,6,7,8-HpCDF	0.22U pg/g	A	bi

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0J230497

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

LDC #: 24524F21	VALIDATION COMPLETENESS W
SDG #: <u>G0J230497</u>	Stage 2B/4
aboratory: Test America	· •

Date	12/17/10
Page:	<u>/of_/</u>
Reviewers	_7
2nd Reviewer:	70

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
ı.	Technical holding times	A-	Sampling dates: /0/22//U
11.	HRGC/HRMS Instrument performance check	A	
111.	Initial calibration	Δ	
IV.	Routine calibration/ICV	SW	
V.	Blanks	Δ	
VI.	Matrix spike/Matrix spike duplicates	5 W	
VII.	Laboratory control samples	SW	Les
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	ےس	
Х.	Target compound identifications	Λ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	5M/	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	رسي	D=4,5 11,12
XV.	Field blanks	SW	EB=17

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

·	50/L						
1	SSAP3-03-1_01_BPC ^	11	SA47-4_01_BPC -	21 /	0298269	31	
2	SSAP3-03-5_01_BPC	12	SA47-4_01_BPC_FD -	22 2	0298247	32	
3	SSAP3-03-9_01_BPC	13	SA47-5_01_BPC -	23		33	
4	SSAP3-04-1_01_BPC	14	SA47-6_01_BPC	24		34	
5	SSAP3-04-1_01_BPC_FD .	15	SA47-7_01_BPC -	25		35	
6	SSAP3-04-5_01_BPC -	16	SA47-8_01_BPC** -	26		36	
7	SSAP3-04-9_01_BPC	17 7	EB-10222010-RZC (J)	27		37	
8	SA47-1_01_BPC -	18	SSAP3-03-9_01_BPCMS .	28		38	
9	SA47-2_01_BPC -	19	SSAP3-03-9_01_BPCMSD	29		39	
10	SA47-3_01_BPC /	20		30		40	

Notes:	· · · · · · · · · · · · · · · · · · ·	
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LDC #: 24524 F2 | SDG #: ru cones

VALIDATION FINDINGS CHECKLIST

Page: _/_ of _2 Reviewer: _____2 2nd Reviewer: _____2

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	_			
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?				
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers < 25% ?	_			
Is the static resolving power at least 10,000 (10% valley definition)?			<u> </u>	
Was the mass resolution adequately check with PFK?	_			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	_			
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	m valle La a			
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?				
Vt. Matrix spike/Matrix spike duplicates			re in i Grand	
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.				
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VII Laboratory control samples				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?		-		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC #: 245 24F2] SDG #: 14 comes

VALIDATION FINDINGS CHECKLIST

Page:_	_2_of_	2_
Reviewer:		_
2nd Reviewer:	9	

VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				
IX: Internal standards			Hindo Strik	
Were internal standard recoveries within the 40-135% criteria?	1.	_		
Was the minimum S/N ratio of all internal standard peaks > 10?				
X: Target compound identification	• .	***		
For 2,3,7,8 substituted congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?				
For 2,3,7,8 substituted congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?				
For non-2,3,7,8 substituted congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?				
Did compound spectra contain all characteristic ions listed in the table attached?	/_		ļ	
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	مععا	/		
Was the signal to noise ratio for each target compound and labeled standard ≥ 2.5?				
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?		_		
For PCDF identification, was any signal (S/N \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	<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?	_			
XII System performance			unità Com	
System performance was found to be acceptable.				
XIII: Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV:sFieldiduplicates				
Field duplicate pairs were identified in this SDG.				The state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the state of the s
Target compounds were detected in the field duplicates.				
XV:Field blanks				
Field blanks were identified in this SDG.		n . Serie Medicality	**********	enement on the commonwealth and advice the second was professed from the first of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the second of the
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC# 24524/F2/

VALIDATION FINDINGS WORKSHEET Routine Calibration

lease 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 routine calibration was performed at the beginning and end of each 12 hour period? Were all percent differences (%D) of RRFs \leq 20% for unlabeled compounds and \leq 30% for labeled?

Did all routine calibration standards meet the Ion Abundance Ratio criteria?

N/A N/A



2nd Reviewer: Reviewer: FT

#	Date	Standard ID	Сотропп	Finding %D (Limit: <30.0%)	Finding lon Abundance Ratio	Associated Samples	Qualifications	ns
	C1 22 01	cen (closing)	136-0CDD	145.9	(435)	0298269-MB	3/43/P auch	G, Q
						-	-	,
1								
11								
		PCDDs	Selected ions (m/z)	Ion Abundance Ratio	PCDFs	Selected ions (m/z)	n/z) Ion Abundance Ratio	nce Ratio
	Tetra-		M/M+2	0.65-0.89	Tetra-	M/M+2	0.65-0.89	.89
	Penta-		M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78	1.78
	Hexa-		M+2/M+4	1.05-1.43	Неха-	M+2/M+4	1.05-1.43	1.43
	Hexa-13C-H;	Hexa- ¹³ C-HxCDF (IS) only	M/M+2	0.43-0.59	Hexa-13C-HxCDF (IS) only	nly M/M+2	0.43-0.59	.59
	Hepta-13C-F	Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51	Hepta- ¹³ C-HpCDF (IS) only	only M/M+2	0.37-0.51	.51
	Hepta-		M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20	.20
	Octa-		M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02	.02

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VALIDATION FINDINGS WORKSHEET

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

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

Was a method blank performed for each matrix and whenever a sample extraction was performed? Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

| N | N/A | Was a method blank performed for each matrix and whenever a sample extraction was perform |
| Y | N/A | Was the method blank contaminated? | 10/29/10 |
| Blank analysis date: 10/26/10 | Associated s

Blank analysis date: 10/29/10

Associated samples:_

() ()

Sample Identification 2 8.1/4 છ. છ 1-MB 029824 Blank ID 4 Compound Conc. units: Ф D

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".

(10C# 24524 F2)

VALIDATION FINDINGS WORKSHEET

L of 2 2nd Reviewer: Reviewer:__

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

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? Blank extraction date: 10 25 10 V/N N/A

Blank analysis date: 10 246 10

Associated samples:

(bd)

Conc. units: 29 9			+							
Compound	Blank ID	XS			Š	Sample Identification	ation			
	OZAKZCAL MB	- MB		3	و	٦	٩	H	12	13
IJ.	21.0	09.0	0.59 M					0.24 /u		
B	78.0	ا.6	1			n/22.0	0.83 H	1/61.0	1.3/4	0.25/M
P	0.08%	hh.a	ſ	10.39 N	10/L1.0			-		0.27 /u
ঠা	0.13	0. ام	,	-	n/62.0					0.48/4
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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

Reviewer: FT 2nd Reviewer:

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Rease 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?

Blank analysis date: でしょし Y N N/A Was the method blank contaminated? Blank extraction date: 10/25/10 Blank analysi N N N X

Associated samples: All 801L >

Sample Identification 0.27/4 2.40 M 2/25.0 0.60 ダメ ۵ ف ه 029826-MB 440 Blank ID 0.088 0.13 0.32 Compound Conc. units: ಬ್ರಾಗ್ನ J প্

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 FIELD BIANKS

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

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XSX																
XSX Slios 11A	on			1												
N	Sample Identification															
Associated Samples:	San															
Associa																
Pala- Ier: EB																
s SDG? ole units: Rinsate / Oth				į												
identified in thi sociated samp) Field Blank / I	Blank ID	17	8.	3.3	<i>0</i> 1											
Y N/A Were field blanks identified in this SDG? Brank units: Pa L Associated sample units: Pa L Sampling date: Dat L Field Blank / Rinsate / Other:	Compound		G	Ġ	Ø	-										CRQL
Sar Ba						<u> </u>	<u> </u>		<u> </u>	<u>L</u>			<u> </u>	<u> </u>		<u>L</u>

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

LDC# 24524F2)

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

Page: _of 7 2nd Reviewer: \mathcal{G} Reviewer: FT

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

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

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

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

	_		_	_			_			, -		-	_		_		_	_	-	_	_	,	_
Qualifications	mo gual Les in																						
Associated Samples	3		•														,						
RPD (Limits)	33 (3)	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
MSD %R (Limits)	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
MS %R (Limits)	(Chl-08) X9	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	(
Compound	មា																						
MS/MSD ID	61471				•														-				
Date																							
#																							

LDC# 24524F2

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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

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

Reviewer: FT 2nd Reviewer:

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· performed? nits?	Associated Samples	AII Water	->																								
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or whenever a san rcent difference (RE	LCSD %R (Limits)	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	(()	()	()	()	()	()	()	()	()	(
ples for each matrix %R) and relative pe	LCS %R (Limits)	134 (76-133	131 (83-130	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
every 20 sam t recoveries (Compound	نـ	۵																								
Was a LCS required? Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	Lab ID/Reference	827-LH28620																									
Y N NIA Y N NIA	# Date																										

LDC# 3452 1/2 2/

VALIDATION FINDINGS WORKSHEET Internal Standards

Reviewer: FT 2nd Reviewer:

Page:

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

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

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

*	Date	Lab ID/Reference	Internal Standard	% Re	% Recovery (Limit: 40-135%)		Qualifications	ກຣ
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))		
		Internal Standards	Check Standard Used		Recovery Standards	,	Check Sta	Check Standard Used
┪	13C-2.3.7.8-TCDF	u		K. 13C-1	¹³ C-1.2.3.4-TCDD			
ᆆ	13C-2 3 7 8-TCDD	0		Щ	¹³ C-1 2 3 7 8 9-HxCDD			
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LDC# 24524 F2/

VALIDATION FINDINGS WORKSHEET Internal Standards

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Reviewer: FT 2nd Reviewer: S

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

X MAN/A
Are all internal standard recoveries were within the 40-135% criteria?

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

*	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	t: 40-135%)		Qualifications
		0	I	77	(361-04)	1/41/P	De Car
	-					-	
	ı	Çl	H	38			ìL.
			工	দী			8.2
			6	3)	(8,6
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		1)		27			ଷ୍ଟ
			b	うを)		9
				Managara da			
		12	Н	14)		ı.
			Н	42	(ଷ ଷ
			り	८६	(9.0
		14	H	Le	(<u> </u>
			Н	11	()		(4 , &
			6	39	(9.9
)		
		15	H	78	(U_
				81	(প ড
			G	ક્રવ	(/)	4	0,0
		Internal Standards	Check Standard Used	Re	Recovery Standards		Check Standard Used
_₹	13C-2.3.7.8-TCDE	DE		K. 13.4-TCDD			
œ	13C-2.3.7.8-TCDD	da		1 13C-123789-HxCDD	CDD		
٥	¹³ C-12,3,7,8-PeCDF	RCDF		M			
<u>d</u>	13.7.8-PeCDD	РЕСПО	groups and de d	Z			
щ	13C-1 2 3 6 7 8-HXCDE	HYCDE					

13C-1234678-HpCDD

¹³C-1,2,3,4,6,7,8-HpCDE ¹³C-1 2 3 6 7 8-HxCDD

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LDC# 24524F2/

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: 3 3

Reviewer: FT 2nd Reviewer:

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

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

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

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

*	Date	Lab ID/Reference	Internal Standard		% Recovery (Limit: 40-135%)	t: 40-135%)	Qualifications	ons
		ما	+		74	(541-04)	JIMJ 1P	and F
			Ы		b)(-		0 G B
			J		36	->	->	9
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						()		
		81			5	(1)	Aug a con Aug Aug	
		Ы	<u>T</u>		(<	\ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \rightarrow \ \rightarrow \ \rightarrow \ \rightarrow \rightarrow \ \rightarrow \rightarrow \ \rightarrow \rightarrow \rightarrow \ \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rightarrow \rig	AN Cyrred MA	Q PM
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	·					(
						()		
		Internal Standards	Check Standard Used		Re	Recovery Standards	Check St	Check Standard Used
₫	¹³ C-2.3.7.8-TCDF	DF		¥	13C-1.2.3.4-TCDD			
α	13C-2 3 7 8-TCDD	OD			_	CDD		
٥	井	PCDF		M			-	

þ

¹³C-1234678-HnCDE 13C-1,2,3,4,6,7,8-HpCDD

¹³C-123678-HxCDD ¹³C-1,2,3,6,7,8-HxCDE ¹³C-1,2,3,7,8-PeGDD

LDC# 24524F2/

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLS

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".

K N N/A

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

ı	ochusos			
Date	Sample ID.	Finding	Associated Samples	Qualifications
		All compounds reported below PQL	All	Waterie (cn)
				(de) capan Lu
		All compounds reported as EMPC	All	VI 17 17 17 17 1
				JA detects (K)
Ì	#	no dud column confirmation	23679 12 15	(0) 2/00 11 0/1
		was perferment	+	

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24524F21

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

Page:_	$l_{of} 2$	_
Reviewer:	FX /	
nd Reviewer:	$\neg \cup -$	

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

<u>Y</u>	Ņ	NA
$\overline{\mathbf{v}}$	V	NA

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

fd

	Concentrat	ion (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	4	5	≤50	Difference	Limits	(Parent Only)
A	0.52U	0.16		0.36	≤0.52	
В	2,6U	0.36		2.24	≤2.6	
С	2.6U	0.26		2.34	≤2.6	·
D	0.30	0.36		0.06	≤2.6	
E	0.37	0.70		0.33	≤2.6	
F	. 0.77	2.1	,	1.33	≤2.6	
G	3.6	5,0		1.4	≤5.2	
н	1.8	3.3		1.5	≤0.52	
1	1.5	3.5		2	≤2.6	
J	0.77	2,0		1,23	≤2 .6	
К	2.3	7.3		5	≤2.6	JAST
L	1.8	5.1		3.3	≤2.6	J/A Det
М	0.51	1.3		0.79	≤2.6	
N	0.37	0.99		0.62	≤2.6	
0	6.7	25		18.3	≤2.6	J/A dut
Р	3.1	12		8.9	≤2.6	V
Q.	24	130		106	≤5.2	V

V:\FIELD DUPLICATES\24524F21.wpd

LDC#: 23906B4

VALIDATION FINDINGS WORKSHEET Field Duplicates

Page: 2of 2nd Reviewer:

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

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

	Concentra	tion (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	11	12	≤50	Difference	Limits	(Parent Only)
D	2.6U	0.15		2.45	≤2.6	
E	2.6U	0.27		2,33	≤2.6	
F	0.24	2.4		2.16	≤2.6	
G	0.79	1.3		0.51	≤5.2	
Н	0.51	0.69		0.18	≤0.52	
1	0.41	0.55		0.14	≤2.6	
J	0.26	0.21		0,05	≤2.6	
К	0.71	0.63		0.08	≤2.6	
L	0.37	0.29		0.08	≤2.6	,
м	0.22	0.096		0.124	≤2.6	
N	2.6U	0.15		2.45	≤2.6	
0	1.4	1,2		0.2	≤2.6	
P	0.62	0.45		0.17	≤2.6	
Q.	3.9	2.8		1.1	≤5. 2	

V:\FIELD DUPLICATES\24524F21.wpd

1DC#: 4457472/ SDG#: 445 cooust

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

Page: of Reviewer: 27

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

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

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

 $A_x = Area$ of compound, $A_x = C_x = Concentration of compound, <math>C_x = S = Standard$ deviation of the RRFs, X = I

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (ひろう std)	RRF (Cシう std)	%RSD	%RSD
+	1,000,1	01/82/01	2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)	121.1 -52-41	0211	4151.1	1.1574	かけれる	494
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.053	1.053	-120 120	+2T1.	02.k	4.20
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	081.1	1.180	1,2326	1.23%	2-14	7-19
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.104	hol.)	1.155	1531-1	3.85	3.85
			OCDE (3C.OCDD)	1.681	1.68	1.7419	1.7419	66.5	465
2	1CAL	0 4 6	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	6.984	09×4	201	50:	8:11	8.11
		•	2,3,7,8-TCDD (°C-2,3,7,8-TCDD)	1.032	1.032	1.06	20-1	10.8	8-01
		•	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1141	1.441	52.1	1.%	12.7	12.7
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	1-26	-2	5.51	12.3
			OCDE (13C. OCDD)	2.118	21118	7.36	2.36	15.3	5.3
3			2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)						
			2,3,7,8-TCDD (°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 (¹³ C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						

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

LDC#: SDG#: 100 co

VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

Page: of Reviewer: 67

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

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

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

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

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

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

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
1	cey 7:11	01/01/11	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	1.120	1.06	1.06	0.3	5.0
		•	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.053	1.17	1.17	10.X	X.01
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	08//	121	127	4٠٠	4,7
		 -	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	401.1	1.14	7.17	3.5	3.5
			OCDE (13C-OCDD)	1.68/	1.98	26.1	8.1€1	X-1-1
2	22:41 Nan	U/24/01	2,3,7,8-TCDF (12C-2,3,7,8-TCDF)	486.0	16.0	0.91	7.4	7.7
		,	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	1.032	0.9]	6.9	9:	=
		· · · · · ·	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.14/	1.2.1	1.2]	7.5	5.6
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.134	5 -1	\ <u>\</u>		
			OCDE (12C-OCDD)	a.118	.92	1.97	٩٠٠	9.4
ဗ			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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LDC#	SDG#:

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates Results Verification

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Page:	Reviewer:	2nd Reviewer:

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

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

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

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

RPD = 1 MSR - MSDR I * 2/(MSR + MSDR)

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

MS/MSD samples:

mples: |\$ + P

	Sp	Spike	Sample	Spiked	Spiked Sample	Matrix	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Compound	Adde (PA 9	ded م)	Concentration (PA 9)	Concentration (()	tration اح)	Percent Recovery	Recovery	Percent Recovery	Recovery	RPD	RPD
	Or MS	U MSD	0, o,	Or SW) MSD	Reported	Recalc	Reported	Recalc		
2,3,7,8-TCDD	0.22	22.0	ΔN	20. G	21.)	94	hb	29	9	2.6	7.8
1,2,3,7,8-PeCDD	110	110	47	107	110	47	97	0 01	gal	3.C	2.4
1,2,3,4,7,8-HxCDD	01	OII	da	6.10	110	28	1ડ્ર	1 00	100	د	8
1,2,3,4,7,8,9-HpCDF	واا	011	ηη	7 8	145	021	02	132	(32	8.9	8.0
OCDF	m	220	02	에 10	269	112	7	12.21	[12	6.3	8.7
		:									

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#._ LDC#:

Laboratory Control Sample Results Verification VALIDATION FINDINGS WORKSHEET

Reviewer: 2nd Reviewer:

Page: ___or___

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

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

% Recovery = 100 * SSC/SA

Where: SSC = Spiked sample concentration SA = Spike added

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

02 98269-10>

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

[]		 1							 	 		
CS/LCSD	٥	Recalculated										
1/831	RPD	Reported										
	tecovery	Racalc										
usoı	Percent Recovery	Reported					× 2	\			•	
8	lecovery	Recalc	. 101	99	68	ላጸ	14					
SUL	Percent Recovery	Reported	107	bb	16 ×	ባኔ	(11					
ample	ration %	I CSD	Λ)				_	
Spiked S	Concentration (ペーツ)	101	21.3	app.0	89.3	18. co	m					
ike	Added (Os Or)	l Csn	Δ	-			\rightarrow					
S	A A	1.08	20.02	001	001	O 01	0 02					
	Compound		2,3,7,8-TCDD	Q	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF					

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

ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	If Acciliate mass(1)	<u> </u>							
	╢	Cit up:	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	Gl nol	Flomental Companier	
_	303,9016	Σ	C.H.3CI.O	1001				Lieliieliidi Composition	Analyte
<u> </u>	305.8987	M+2	C;H,**Cl,**C10	 	4	407.7818	M+2	C,H ³⁵ Cl, 37ClO	HoCDF
	315,9419	×	C.O.HO.	20.1		409.7788	M+4	C,H"C "C ,O	H C
	317.9389	M+2	1.2. H.30[37CIO	(8)	_	417.8250	Σ	13C,H3CLO	HPCDE (8)
	319.8965	Σ	, C,	(5)		419,8220	M+2	1°C,H°CI,*7CIO	HPCDF (9)
	321.8936	M+2	C.H.*CL**C10.	1001		423,7767	M+2	C,Hackarolo,	HOOD
	331.9368	2	13C. H. 8C.I.O.	1007		425.7737	M+4	C.Haclarol	
	333,9338	Ø+2	13. 4 0.402 13. H 35. 37.00	(8) 0001		435.8169	M+2	13C H 2C 13C C	
	375.8364	1 0 ± 1 × 1	C T 80 300	(s) day		437.8140	M+4	13C H3C 3C C	
	[354.9792]	: X	<u> </u>	HXCDPE		479.7165	. + M	C Harring 200	HPCUU (S)
==			<u> </u>	Υ Υ		[430.9728]	LOCK	C.F.; C. C.2.	NOUPE PEK
,									
N	339,8597	M+2	C.H.*Cl.arClO	שיטים					_
	341.8567	M+4			က	441,7428	M+2	C, 30, 370, 0	2000
	351,9000	M+2	13 L 32 L 32 L 32 L 32 L 32 L 32 L 32 L	בים בים		443.7399	M+4	0.08.0	200
	353.8970	M+4	0 10 4 10 5 13 0 10 10 10 10 10 10 10 10 10 10 10 10 1	recor (s)		457.7377			700
	355.8546			recur (s)		459,7348		_	מממ
	357.8516			Pecdo		469,7780	-	120 25 25 25 25 25 25 25 25 25 25 25 25 25	0000
	367,8949			PecDD	_	471.7750		_	OCDD (S)
	369.8919			PecdD (S)		513.6775		,,	OCDD (s)
	409.7974			PecdD (s)		[422,9278]	_	C. C. C. C. C. C. C. C. C. C. C. C. C. C	DCDPE
	[354 0700]			HOOPE		-			PFK
	[28/8/60]	- Yoo -	r. 	PFK					
တ	373.8208	M+2	C. H. acl acid	100	+				
-	375.8178	M+4	C.H. C. S.C.						
	383,8639	≅	13C.H.3CIO	באר בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בייניים בי					
	385.8610	M+2	130, H. 30, 130, 100, 1	(s) LOOK!					
	389.8156		C H 35C 37C -	(8)					
	391,8127		C.H.*C.*7C.C.	בייייייייייייייייייייייייייייייייייייי	-				
	401,8559		OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF TH	מאנים האינים	_				
	403.8529		ac, H, acl, acl, o	(e)					
	445.7555		C,H,*C1,*C1,O	OCUPE OCUPE	-				-
	[430.9728]	Lock	C,F, ,	PFK					

The following nuclidic masses were used:

®

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

O = 15.994915 $^{35}Cl = 34.968853$ $^{37}Cl = 36.965903$

S = internal/recovery standard

LDC #:_			
SDG #:	u	cover	

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

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Reviewer:	F	2_
2nd reviewer:	2	

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?

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

Example:

Concentration = (A_)(I_)(DF) (A,)(RRF)(V,)(%S) Area of the characteristic ion (EICP) for the compound to be measured Area of the characteristic ion (EICP) for the specific internal standard Amount of internal standard added in nanograms (ng)· Volume or weight of sample extract in milliliters (ml) or grams (g). RRF Relative Response Factor (average) from the initial calibration Df Dilution Factor. Percent solids, applicable to soil and solid matrices %S

Sample I.D. #16 . 3, 3, 7, X - tc0 F

Conc. = (225023) (2000) ()
1/2484040) (10.32) (1.12) (0.929

= 0.37 palg

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

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

October 26, 2010

LDC Report Date:

December 19, 2010

Matrix:

Soil/Water

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0J270514

Sample Identification

SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC-FD SA183-4-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSA04-1-01-BPC RSAO4-2-01-BPC RSAO4-3-01-BPC RSAO4-4-01-BPC RSAO4-5-01-BPC RSAO4-6-01-BPC RSAO4-7-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD SSAN6-05-1-01-BPC SSAN6-05-2-01-BPC

SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC EB-10262010-RZC SA183-1-01-BPCMS SA183-1-01-BPCMSD SSAN6-05-3-01-BPCMS SSAN6-05-3-01-BPCMSD

^{**}Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
11/9/10	¹³ C-1,2,3,4,7,8-HxCDF	41	EB-10262010-RZC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC SSAN6-05-3-01-BPCMS SSAN6-05-3-01-BPCMSD 0301424-MB 03014440MB	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) UJ (all non-detects)	P

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
0301420-MB	10/28/10	OCDD 1,2,3,4,6,7,8-HpCDF	0.27 pg/g 0.060 pg/g	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC-FD SA183-4-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-6-01-BPC SA183-8-01-BPC** RSA04-1-01-BPC RSA04-2-01-BPC RSA04-3-01-BPC RSA04-5-01-BPC RSA04-6-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC-FD SSAN6-05-1-01-BPC

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
SA183-2-01-BPC	OCDD	0.87 pg/g	0.87U pg/g
	1,2,3,4,6,7,8-HpCDF	0.099 pg/g	0.099U pg/g
SA183-3-01-BPC	OCDD	1.0 pg/g	1.0U pg/g
SA183-4-01-BPC OCDD		0.46 pg/g	0.46U pg/g
1,2,3,4,6,7,8-HpCDF		0.12 pg/g	0.12U pg/g

Sample	Compound	Reported Concentration	Modified Final Concentration
SA183-5-01-BPC	OCDD	0.80 pg/g	0.80U pg/g
SA183-6-01-BPC OCDD 1,2,3,4,6,7,8-HpCDF		0.41 pg/g 0.14 pg/g	0.41U pg/g 0.14U pg/g
SA183-7-01-BPC	1,2,3,4,6,7,8-HpCDF	0.20 pg/g	0.20U pg/g
SA183-8-01-BPC**	OCDD	0.79 pg/g	0.79U pg/g
RSAO4-2-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	1.0 pg/g 0.24 pg/g	1.0U pg/g 0.24U pg/g
RSAO4-3-01-BPC	1,2,3,4,6,7,8-HpCDF	0.25 pg/g	0.25U pg/g

Sample EB-10262010-RZC was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Equipment Blank ID	Sampling Date	Compound	Concentration	Associated Samples
EB-10262010-RZC	10/26/10	OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	5.6 pg/L 2.8 pg/L 3.4 pg/L 2.4 pg/L 5.9 pg/L 4.1 pg/L 9.4 pg/L	All soil samples in SDG G0J270514

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

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Although the MS/MSD percent recoveries (%R) 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
SA183-1-01-BPC	¹³ C-OCDD	30 (40-135)	OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA183-2-01-BPC	¹³ C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-3-01-BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	33 (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)	Р
SA183-3-01-BPC-FD	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 19 (40-135) 32 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SA183-4-01-BPC	¹³ C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA183-5-01-BPC	¹³ C-OCDD	23 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA183-6-01-BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 25 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SA183-7-01-BPC	¹³ C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SA183-8-01-BPC**	¹³ C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
RSAO4-2-01-BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 25 (40-135) 35 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

		-			
Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
RSAO4-3-01-BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	26 (40-135) 36 (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
RSAO4-4-01-BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	37 (40-135) 23 (40-135) 35 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
RSAO4-5-01-BPC	¹³ C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
RSAO4-6-01-BPC	¹³ C-OCDD	24 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
RSAO4-7-01-BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 22 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
RSAO4-8-01-BPC**	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 22 (40-135) 28 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
RSAO4-8-01-BPC-FD	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	39 (40-135) 28 (40-135) 33 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р
SSAN6-05-1-01-BPC	¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	26 (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
SSAN6-05-2-01-BPC	¹³ C-OCDD	37 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р
SSAN6-05-3-01-BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 19 (40-135) 30 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-05-4-01-BPC	13C-OCDD	38 (40-135)	OCDD	J (all detects)	Р
			OCDF	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
SSAN6-05-1-01-BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAN6-05-3-01-BPC	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
SA183-8-01-BPC** RSAO4-4-01-BPC RSAO4-6-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD EB-10262010-RZC	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 G0J270514	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 G0J270514	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 SA183-3-01-BPC and SA183-3-01-BPC-FD and samples RSAO4-8-01-BPC** and RSAO4-8-01-BPC-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

	Concentr					
Compound	SA183-3-01-BPC	SA183-3-01-BPC-FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,6,7,8-HxCDD	0.19	2.6U	-	2.41 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	0.18	2.6U	-	2.42 (≤2.6)	-	
1,2,3,4,6,7,8-HpCDD	0.40	0.40	-	0 (≤2.6)	-	-
OCDD	1.0	1.8		0.8 (≤5.3)	_	-
2,3,7,8-TCDF	0.31	0.60	-	0.29 (≤0.53)	-	-
1,2,3,7,8-PeCDF	0.47	0.71	-	0.24 (≤2.6)	-	-
2,3,4,7,8-PeCDF	2.6U	0.31	-	2.29 (≤2.6)	-	-
1,2,3,4,7,8-HxCDF	0.88	1.1	-	0.22 (≤2.6)	-	-
1,2,3,6,7,8-HxCDF	0.65	0.95	-	0.3 (≤2.6)	-	
2,3,4,6,7,8-HxCDF	0.29	0.19	-	0.1 (≤2.6)	-	-
1,2,3,7,8,9-HxCDF	0.32	0.21	-	0.11 (≤2.6)	-	-

	Concentr						
Compound	SA183-3-01-BPC	SA183-3-01-BPC-FD	RPD (Limits)	Difference (Limits)	Flags	A or P	
1,2,3,4,6,7,8-HpCDF	1.9	3.0	-	1.1 (≤2.6)	-	-	
1,2,3,4,7,8,9-HpCDF	1.1	1.6	-	0.5 (≤2.6)	-	-	
OCDF	4.1	6.3	-	2.2 (≤5.3)	-	-	

	Concentra					
Compound	RSAO4-8-01-BPC**	RSAO4-8-01-BPC-FD	RPD (Limits)	Difference (Limits)	Flags	A or P
1,2,3,4,6,7,8-HpCDD	0.54	0.64	-	0.1 (≤2.6)	-	-
OCDD	5.1	5.9	-	0.8 (≤5.2)	-	-
2,3,7,8-TCDF	0.14	0.17	•	0.03 (≤0.52)	-	
1,2,3,4,7,8-HxCDF	0.15	0.23	-	0.08 (≤2.6)	-	-
1,2,3,6,7,8-HxCDF	0.13	0.14	-	0.01 (≤2.6)	-	-
2,3,4,6,7,8-HxCDF	2,6U	0.069	-	2.531 (≤2.6)	-	-
1,2,3,7,8,9-HxCDF	2.6U	0.076		2.524 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDF	0.66	0.90	-	0.24 (≤2.6)	-	-
1,2,3,4,7,8,9-HpCDF	0.20	0.44	-	0.24 (≤2.6)	-	
OCDF	1.2	1.6	_	0.4 (≤5.2)	•	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0J270514

				l .	
SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J270514	EB-10262010-RZC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) UJ (all non-detects)	Р	Routine calibration (%D) (c)
G0J270514	SA183-1-01-BPC SA183-2-01-BPC SA183-4-01-BPC SA183-5-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSAO4-5-01-BPC RSAO4-6-01-BPC SSAN6-05-2-01-BPC SSAN6-05-4-01-BPC	OCDD	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J270514	SA183-3-01-BPC RSA04-3-01-BPC SSAN6-05-1-01-BPC	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)
G0J270514	SA183-3-01-BPC-FD SA183-6-01-BPC RSA04-2-01-BPC RSA04-4-01-BPC RSA04-7-01-BPC RSA04-8-01-BPC** RSA04-8-01-BPC-FD SSAN6-05-3-01-BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0J270514	SSAN6-05-1-01-BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P .	Project Quantitation Limit (exceeded range) (e)
G0J270514	SSAN6-05-3-01-BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0J270514	SA183-8-01-BPC** RSA04-4-01-BPC RSA04-6-01-BPC RSA04-8-01-BPC** RSA04-8-01-BPC-FD EB-10262010-RZC	2,3,7,8-TCDF	None	Р	Project Quantitation Limit (no 2 nd column confirmation) (o)

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J270514	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC SA183-4-01-BPC SA183-5-01-BPC SA183-5-01-BPC SA183-7-01-BPC SA183-8-01-BPC SA183-8-01-BPC RSA04-1-01-BPC RSA04-3-01-BPC RSA04-3-01-BPC RSA04-5-01-BPC RSA04-6-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC SAN6-05-1-01-BPC SSAN6-05-1-01-BPC SSAN6-05-3-01-BPC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC SBAN6-05-4-01-BPC SBAN6-05-4-01-BPC SSAN6-05-4-01-BPC SBAN6-05-4-01-BPC SBAN6-05-4-01-BPC SBAN6-05-4-01-BPC SBAN6-05-4-01-BPC SBAN6-05-4-01-BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J270514	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC SA183-4-01-BPC SA183-5-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSA04-1-01-BPC RSA04-2-01-BPC RSA04-3-01-BPC RSA04-5-01-BPC RSA04-5-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC RSA04-8-01-BPC SSAN6-05-1-01-BPC SSAN6-05-2-01-BPC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC EB-10262010-RZC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)		Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0J270514

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J270514	SA183-2-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	0.87U pg/g 0.099U pg/g	Α	bl
G0J270514	SA183-3-01-BPC	OCDD	1.0U pg/g	Α	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0J270514	SA183-4-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	0.46U pg/g 0.12U pg/g	А	bl
G0J270514	SA183-5-01-BPC	OCDD	0.80U pg/g	А	þI
G0J270514	SA183-6-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	0.41U pg/g 0.14U pg/g	А	bl
G0J270514	SA183-7-01-BPC	1,2,3,4,6,7,8-HpCDF	0.20U pg/g	А	bl
G0J270514	SA183-8-01-BPC**	OCDD	0.79U pg/g	А	bl
G0J270514	RSAO4-2-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	1.0U pg/g 0.24U pg/g	A	Ы
G0J270514	RSAO4-3-01-BPC	1,2,3,4,6,7,8-HpCDF	0.25U pg/g	Α	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0J270514

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET LDC #: 24524G21 SDG #: G0J270514 Stage 2B/4 Laboratory: Test America

Date:_	12/17/10
Page:_	<u>/</u> of_/_ ′
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METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

	Validation Area		Comments
<u>l.</u>	Technical holding times	Δ	Sampling dates: 10/2 6/10
IJ.	HRGC/HRMS Instrument performance check	Δ	
ıiı.	Initial calibration	Δ	
IV.	Routine calibration/I CV	یسی	
V	Blanks	Δ	
VI.	Matrix spike/Matrix spike duplicates	يسى	
VII.	Laboratory control samples	A	Les
VIII.	Regional quality assurance and quality control	N	·
IX.	Internal standards	sω	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	ىسى	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	4	
XIV.	Field duplicates	رسى	D=3,4 17+18
XV.	Field blanks	ςw	EB= 23

Note: A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

FB = Field blank

D = Duplicate

TB = Trip blank

EB = Equipment blank

Validated Samples: ** Indicates sample underwent Stage 4 validation

	SOIL + water	4		
1 (SA183-1-01-BPC	11	RSAO4-2-01-BPC	21 Z SSAN6-05-3-01-BPC 31 \ 0 3 0142U
2	SA183-2-01-BPC	12	RSAO4-3-01-BPC	22 2 SSAN6-05-4-01-BPC 32 2 030 1-24
3	SA183-3-01-BPC	13	RSAO4-4-01-BPC	23 EB-10262010-RZC W 33 3 030 1440
4	SA183-3-01-BPC-FD	14	RSAO4-5-01-BPC	24 / SA183-1-01-BPCMS 34
5	SA183-4-01-BPC	15	RSAO4-6-01-BPC	25 SA183-1-01-BPCMSD 35
6	SA183-5-01-BPC	16	RSAO4-7-01-BPC	26 1 SSAN6-05-3-01-BPCMS 36
7	SA183-6-01-BPC	17	RSAO4-8-01-BPC**	27 1 SSAN6-05-3-01-BPCMSD 37
8	SA183-7-01-BPC	18	RSAO4-8-01-BPC-FD	28 38
9	SA183-8-01-BPC**	19	SSAN6-05-1-01-BPC	29 39
10	RSAO4-1-01-BPC	20	SSAN6-05-2-01-BPC	30 40

Notes:			

LDC #: 245 24 G2 \ SDG #: 10 cones

VALIDATION FINDINGS CHECKLIST

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

Wethod: Dioxins/Dibenzorurans (EPA 5VV 846 Method 8290 Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	_			
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?				
Were the retention time windows established for all homologues?	_			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	/			
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				T
Was the initial calibration performed at 5 concentration levels?	_		<u> </u>	
Were all percent relative standard deviations (%RSD) \leq 20% for unlabeled standards and \leq 30% for labeled standards?	_			
Did all calibration standards meet the Ion Abundance Ratio criteria?	_			
Was the signal to noise ratio for each target compound \geq 2.5 and for each recovery and internal standard \geq 10?		<u></u>		
IV: Continuing calibration	•		.'	
Was a routine calibration performed at the beginning and end of each 12 hour period?				
Were all percent differences (%D) \leq 20% for unlabeled standards and \leq 30% for labeled standards?	ut	~	-	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				
Was a method blank associated with every sample in this SDG?	-			
Was a method blank performed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		-		
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?	A STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STATE OF THE STA			
VIIa Eaboratory control samples #				
Was an LCS analyzed for this SDG?				
Was an LCS analyzed per extraction batch?	-			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC #: 24524G2/ SDG #: ________

VALIDATION FINDINGS CHECKLIST

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Reviewer:	F	_
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VIII Regional Quality Assurance and Quality Control	<u> </u>	: 		
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?	11901 - 11			
IX: Internal standards				
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	· ·	.	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?		_	1	
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?		<u></u>		·
XII.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 12.				
System performance was found to be acceptable.	/			
XIII overali assessment of data	1.27 5.34			
Overall assessment of data was found to be acceptable.				
XIV Field duplicates 2.44				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
We prediction to the second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second se				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.	100			
	.===			

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

Notes:

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VALIDATION FINDINGS WORKSHEET Routine Calibration

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".

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

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

(v)

<u>Y</u>	N/A	Did all routine calibration standards meet the Ion Abundance Ratio criteria?	on standards meet 1	the Ion Abundance Ka	tio criteria?		
*	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Finding lon Abundance Ratio	Associated Samples	Qualifications
	01/9/11	cer (chosing)	113-1,23,4,7,8-			0301424-MB,	1/42/P. OURL
	<i>ઇ</i> નેઃ¢	ò	Hx CDF	=		+/2,20,25,12	· -
						all water (Oso, yas	
				`			
		PCDDs S	Selected ions (m/z)		PCDFs	Selected ions (m/z)	iz) Ion Abundance Ratio
	Tetra-		M/M+2	0.65-0.89	Tetra-	M/M+2	0.65-0.89
	Penta-		M+2/M+4	1.32-1.78	Penta-	M+2/M+4	1.32-1.78
	Неха-		M+2/M+4	1.05-1.43	Hexa-	M+2/M+4	1.05-1.43
	Hexa-13C-l	Hexa-13C-HxCDF (IS) only	M/M+2	0.43-0.59	Hexa-13C-HxCDF (IS) only	y M/M+2	0.43-0.59
	Hepta-13C-	Hepta-13C-HpCDF (IS) only	M/M+2	0.37-0.51	Hepta-13C-HpCDF (IS) only	ly M/M+2	0.37-0.51
	Hepta-		M+2/M+4	0.88-1.20	Hepta-	M+2/M+4	0.88-1.20
	Octa-		M+2/M+4	0.76-1.02	Octa-	M+2/M+4	0.76-1.02

LDC# 2452462

VALIDATION FINDINGS WORKSHEET

2nd Reviewer: 🖰 Reviewer.

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

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

Were all samples associated with a method blank?

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

Was the method blank contaminated? Y M N/A

Blank analysis date: 사이에 10 Blank extraction date: 10 28 10

02 4 Associated samples:

0.24/4 1.0/4 Q 0-20/11 0.41/4 0.14/4 Sample Identification 0.80/4 0.46/4 6.0 0.8874 1.0/11 4 3 0.099/4 0.25 4 0.87/M 6 1.35 9.5 6 1.35 \ \ 030 W20-MB Blank ID 0.000 0.060 0.27 0.27 Conc. units: Rala Compound P J ρ 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# 2452492/

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 N/A Were field blanks identified in this SDG?

Blank units: (20) Associated sample units

Sampling date: 10/2 6 10

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

All 801 | S/S/ Associated Samples:

			S.a	Sample Identification	ation		
						-	
K 5.4							
7.7		:					
1·h d	,						
4.6							
						,	
						٠	
CROL							

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

LDC# 245 249 2/

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

2nd Reviewer: Page: 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 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. A N/A

/ 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? N/A

Qualifications	no grand resu	1							mo a mad lesting														
Associated Samples									12														
RPD (Limits)	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()	()
MSD %R (Limits)	were water age	()	()	()	()	()	()	()	()	()	()	()	()	()	()	(()	, ()	()	()	()	()	()
MS %R (Limits)	(Runs () mar	P () 1/2)	()	()	()	()	()	()	()	()	()	(.)	()	(()	()	()	()	()	()	()	()
Compound	Some	%																					
CI CSW/SW									12, 25				-					-		•			
Date																		·					
#																							

100#. 2457/92/

VALIDATION FINDINGS WORKSHEET

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2nd Reviewer: Reviewer: FT

Internal Standards

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

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

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

*	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
			T	30 (40-135)	Just Prancy G. Q
)	
		7) 36	
				(•
		3	H	33 (G, Q
			6	39 (0,10
				()	
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			Ξ.	()	প্র
			Ğ	() 78	90
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		ى	H) OE	9'9
				()	
		9	H	25 ()	(4, k)
				()	
		·	1) %E	L
			H	٠)	G. B
			5	54 ()	Ø, P
				(1
				()	
		Internal Standards	Check Standard Used	Recovery Standards	Check Standard Used
A.	13C-2.3.7.8-TCDF	:DF		K. ¹³ C-1.2.3.4-TCDD	
В	13C-2.3.7.8-TCDD	OC		1 13C-123789-HxCDD	
d	13C-12378-PeCDE	PACDF		W	
4	¹³ C-12378-PeCDD	PACDD		Z	
ц	13C-123678-HxCDE	L-HXCDF		d	
u	¹³ C-1,2,3,6,7,8-HxCDD	HXCDD		α	
ď	13C-1,2,3,4,6,7,8-HpCDE	, 8-HpCDE	:	a	
뒥	13C-1,2,3,4,6,7,8-HpCDD	,8-HpCDD		В	
4	13C OCDD			1	

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VALIDATION FINDINGS WORKSHEET

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Internal Standards

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

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

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

N N/A

å	Date	Lab ID/Reference	Internal Standard	% Recover	% Recovery (Limit: 40-135%)		Qualifications
		8	H	27	(40-135)	J MJ P	great G, Q
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			+	38	()		11.
			H	7,	()		6,0
		i	G	35	()		9.0
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		7)	H	26)		<u>۾</u> ھ
			6	36	()		9
					()		
		(5	1	18			u_
			Ţ	23)		ক্
			6	35	()		9,9
	ĺ				()	-	
		7-1	H	30	()		۵,6
					()		
		15	Н	42	(>	G, &
					()		
					()		
		Internal Standards	Check Standard Used		Recovery Standards		Check Standard Used
¹³ C-2.3.7.8-TCDF	.8-T	ODF		K, 13C-1.2.3.4-TCDD	TCDD		
13C-2 3 Z 8-TCDD	7.8-TC	and			13C-1 2 3 7 8 9-HxCDD		
¹³ C-1 2 3 7 8-PeCDF	7 8-	PeCDF		M			
13C-12378-PeCDD	7 8-	PeCDD		Z			
130-123	67	¹³ C-1 2 3 6 7 8-HxCDF		q			
13C-123	6.7	¹³ C-1,2,3,6,7,8-HxCDD		O.		-	
130-123	46	¹³ C-1,2,3,4,6,7,8-HpCDF		О			
130-123	46	¹³ C-1,2,3,4,6,7,8-HpCDD		В			
13C OCDD	d						

126 #3/2 #201

VALIDATION FINDINGS WORKSHEET Internal Standards

Reviewer: FT 2nd Reviewer: Q

Page: Sof 2

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

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

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

*	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	0-135%)	Qualifications	
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)	^		
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			H) 22	(
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		-	H	2%	(
			5) کو	(
)	(
		61	[26)	9.6	d
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		ρZ	Н	37 (\uparrow	(a) (b)	X
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))		
))		
		Internal Standards	Check Standard Used	Reco	Recovery Standards	Check Standard Used	pes
Ą	13C-2,3.7,8-TCDF)DF		K. 13C-1,2,3,4-TCDD			
<u>a</u>	13C-2.3.7.8-TCDD	מת:		1 13C-1,2,3,7,8,9-HxCDD	0		
c	¹³ C-12378-PeCDF	PACDE		M			
4	13C-12378-PeCDD	PeCDD		Z			
ц	13C-123678-HxCDE	3-HxCDF		q			
и	13C-1,2,3,6,7,8-HxCDD	з-НхСDD		α			
Q	13C-1,2,3,4,6,7,8-HpCDE	7,8-HpCDF		q			
크	13C-1,2,3,4,6,7,8-HpCDD	Z,8-HpCDD		В			
_	J 13C OCDD			-			

/26/73/27/65/

VALIDATION FINDINGS WORKSHEET

Page: Yof Y Reviewer FT

2nd Reviewer:

Internal Standards

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

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

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

- -	200	אמט נווכ סיוא ומנוס מוו וווכווומו פנמוזממות סכמוט - וסי	חוממות הכמות – וס:			
**	Date	Lab ID/Reference	Internal Standard	% Recovery (% Recovery (Limit: 40-135%)	Qualifications
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		Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
Ä	13C-2,3,7,8-TCDF	CDF		K. 13C-1.2.3.4-TCDD	das	
М	13C-2 3 7 8-TCDD	ano		1 13C-123789-HxCDD	9-HxCDD	
ပ	13C-12378-PeCDE	PeCDF		M		
٩	13C-1,2,3,7,8-PeCDD	PeCDD		Z		
Щ	13C-12367,8-HxCDE	8-HxCDE		q		
Щ	13C-1,2,3,6,7,8-HxCDD	8-HxCDD		Д		
G	13C-1234678-HpCDF	7 8-HpCDF				

¹³C-1234678-HpCDD

100 # 2452492/

Compound Quantitation and Reported CRQLs VALIDATION FINDINGS WORKSHEET

Reviewer: FT 2nd Reviewer: Q Page: Lof

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

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

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

Qualifications	JPad (e)		(6)							
Associated Samples	19		2							
Finding	xld cal Range)	7							
Comport	, P. R.		Ø, Ø	•	-					
# Date										

Comments: See sample calculation verification worksheet for recalculations

LDC# 2452492

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

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

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

Y N N/A

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

		_	 	$\overline{}$	·			_			 _	_	 ==		_
o de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de la companya de l	Qualifications	J/A detects (sp)					JK detects (k)	(2)	1						
Associated Samples		All					All	9, 13, 15, 17	1,7 2,7						
Finding	All compounds reported below DO	TO LANCISC TO THE COLUMN TO THE	,			V	All compounds reported as EMPC	no and column	1 ×						
compol Sample ID								1,							,
Date															
#						•							 Ţ		

Comments: _See sample calculation verification worksheet for recalculations

LDC#: 24524G21

VALIDATION FINDINGS WORKSHEET Field Duplicates

Reviewer 2nd Reviewer:

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

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

	Concentrat	ion (pg/g)	%RPD ≤50	(pg/g)	(pg/g)	Qualifications
Compound	3	4		Difference	Limits	(Parent Only)
D	0.19	2.6U		2.41	≤2.6	
E	0.18	2.6U		2,42	≤2,6	,
F	0.40	0.40		o	≤2.6	
G	1.0	1.8		0.8	≤5.3	
Н	0.31	0.60		0.29	≤ 0,53	
1	0.47	0.71		0.24	≤2.6	
J	2.6U	0.31		2.29	≤2.6	
к	0.88	1.1		0.22	≤2.6	
L	0,65	0.95		0.3	≤2.6	
м	0.29	0.19		0.1	≤2.6	
N	0.32	0.21		0,11	≤2.6	
0	1.9	3.0		1.1	≤2.6	
Р	1.1	1.6		0.5	≤2.6	
Q.	4.1	6.3		2,2	≤5.3	

245462/

LDC#: 23906B4

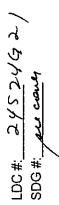
VALIDATION FINDINGS WORKSHEET Field Duplicates

2nd Reviewer:

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

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

	Concentra	tion (pg/g)	%RPD	(pg/g)	(pg/g)	Qualifications
Compound	17	18	≤50	Difference	Limits	(Parent Only)
F	0.54	0.64		0.1	≤2.6	
G	5.1	5.9		0.8	≤ 5.2	
Н	0.14	0.17		0.03	≤0.52	
К	0.15	0.23		0.08	≤2.6	
L	0.13	0.14		0.01	≤2.6	
М	2.6U	0.069		2.531	≤2.6	
N	2.6U	0.076		2.524	≤2.6	
0	0.66	0.90		0.24	≤2.6	
Р	0.20	0.44		0.24	≤2.6	
Q	1.2	1.6		0.4	≤5.2	



VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

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

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

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

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

 A_{ic} = Area of associated internal standard C_{ic} = Concentration of internal standard s, X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (cふう std)	RRF (Cシ うstd)	%RSD	%RSD
-	1471	012/1	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	0 -9915	sbb.o	०.जाक्षान	0.7849	39.5	3.68
		<u>-</u>	2,3,7,8-TCDD ('3C-2,3,7,8-TCDD)	6.993	686.0	0.968	1896.0	3.20	3.24
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	८१।	1.163	4101.1	1.1014	5.17	5.17
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8,-HpCD <u>D</u>)	1.093	1.093	SELOI	1.0135	9 D. P	6.49
			OCDE (13C. OCDD)	0.530	1.370	0055-1	1-3500	1.9×	36.
2			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 (1 ² C-1,2,4,6,7,8,-HpCDD)						
			OCDE (3C.OCDD)						
က	•		2,3,7,8,TCDF ("3C-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 ('3C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						

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

botheshe SDG#: LDC#

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_x = Area of compound, A_x = Concentration of compound, C_x = Concentration of compound, C_x Where:

 $A_{is} = Area$ 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%
-	01/01/11 X5:81 NOD	01/01/11	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0,995	0.94	pbo	6.5	5.3
		· •	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	0.983	1.04	1.04	۲.۶	4.5
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	(ना५३	1.16	<u>:</u>	0	0
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	1.093	80.1	1.0 X	2.0	50
			OCDE (13C-OCDD)	1.370	1-2	1.7	11.7	17:
2	aed 20:20	01/6/11	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	•	0.96	25.0	3.7	3.7
		· -	2,3,7,8-TCDD (13C-2,3,7,8-TCDD)		10.1	1.01	2.6	3.6
		 -	1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)		1.09	1.09	0.9	0.9
		 ,	1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)		00-1	1.06	0.0	6.0
			OCDE (13C-OCDD)	7	1.72	1.22	10.01	6.01
က	aev 9:12	5	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)		० व ३	0.93	6.3	6-3
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)		1.01	1.0]	2.7	2.7
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)		1.0-1	1.07	7.7	7-7
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)		1.06	90-1	0. X	Ŋ-0
			OCDF (13C-OCDD)	/	1.21	1.2.1		

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

Mons w se cons SDG#: LDC#:

Matrix Spike/Matrix Spike Duplicates Results Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer: < Reviewer:_

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

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

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

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

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

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

MS/MSD samples:

7

Spike	-	Sample		Spiked	Spiked Sample	Matrix	Matrix Snike	Matrix Spik	Matrix Spike Duplicate	Reported	Recalculated
Concentration (PR a)	Concentration (P. P. P.)	- ,	_	Concer (P.	Concentration	Percent	Percent Recovery	Percent	Percent Recovery	RPD	Ca
		0,0		F							
MS MSD MS		WS	MS		MSD	Reported	Recalc	Reported	Recalc		1
8.Pl QU 2.00 2.15	22		19.8		186	97	16	٩	ام	ن	7.9
105 UN 201 FOI	CAN		05		98.2	Хb	9 k	و	2	\$-9 %	6X
H-18 102 0.054 87.4	8 0.050	8	4.1%		21.5	18	الا	10	1.0	20	B
411 . 2.2 . 401 [0]	7.7	,	444		720	911	911	三	의	ا- م	6
احدا ١٥١ احمد الالا	10 ما	۲.	الحرا		Q	38	38	2	87	18.0	ø.×
51	5	5	<u>b</u>		8						
							<u> </u>				
,											

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.

V:\Validation Worksheets\Dioxin90\MSDCLC90.21

SDG #: 41 conf

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Reviewer:

Page: ___ot__

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 V

Where: SSC = Spiked sample concentration SA = Spike added

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

501-02/1050

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

		bat		· <u> </u>						1 11		-
csn	RPD	Recalculated	\									
(CS/I CSD	Ŗ	Reported			<u> </u>							
QX	ecovery	Racalc										
uso i	Percent Recovery	Reported					NA	\				
S	tecovery	Recalc	43	Lb	EL	01	92					
SUI	Percent Recovery	Reported	26	Llo	٤L	101	٩L					
amole	nation) 1 CSD	NA	-			 →					
Spiked	Concentration) () (18.4	97.3	75.6	(0)	131					
[ke	Added (Pa a)	10 LCSD	D.V				_>					
aS	Adr	108	20	0 0	Cal	0 04	0.02					-
	Compound		2,3,7,8-TCDD	1,2,3,7,8-PeCDD	1,2,3,4,7,8-HxCDD	1,2,3,4,7,8,9-HpCDF	OCDF					

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

ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Acciliate mass(1)								
	- -	מי ווסו	Elemental Composition	Analyte	Descriptor	Accurate Mass ⁽⁴⁾	Ci noj	Elemental Composition	Ampleto
-	303,9016	Σ	O.H. SOLO	1001					Апајуте
	305.8987	M+2	C.H 3C1 37710	7001	4	407.7818	M+2	C. H ³⁶ Cl. 37ClO	1
	315,9419	. N	12 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	- CD-		409.7788	M+4	12: 08 00 Tago 32: 0	ייוסקר ו
-	317.9389	2		ICDF (S)		417.8250		12 12 02 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05 12 05	Hpcu-
	319 8965	7 2		TCDF (S)		419,8220	C-M	12/ C-12 C-13	HPCDF (S)
	321 8036	<u> </u>	2, T. T. T. T. T. T. T. T. T. T. T. T. T.	TCDD		423.7767	7 - M		HpcDF
	0000000	N+Z	J C ₁₂ H, 301,37010,	TCDD		40E 7707	7 + IAI	CIO. "CIO."	Heco
	531.9368	≥	_ '0' O;;'H''30;	TODD (e)		450.7757	M+4	C ₁₂ H ²³ Cl ²³ Cl ₃ O ₃	Hood
-	333.9338	M+2	13C, H, 35C, 13/CiO	(0)		435,8169	M+2	13C, Harcio	(a) (d) (d) (d) (d) (d) (d) (d) (d) (d) (d
	375.8364	M+2	C H 300 37010	(8) 000 (1)		437.8140	M+4	2-1-2-3-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-	(e) and 1
	[354.9792]	X S S	() (s) () (s) (s) (s) (s) (s) (s) (s) (s	TXCDF		479.7165	M+4	C H ₃₅ Cl 37Cl O	(S) DOOD!
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						_		`	2
2	339,8597	M+2	C, H, "C , "C O	Pache	ı				
	341.8567	M+4	C. H, "CI, "CI, O	100 d	n	441.7428		C _{1,35} C _{1,37} CIO	OCDE
	351,9000	M+2	1 "C. H. "C. "C. 10	13/ 14/10		443.7399	M+4	C,3*C ,3*C ,O	OCDE
	353,8970	M+4	13C, H, 3Cl, 3Cl, O	(a) 1000 (b) 1000 (c)		457.7377	M+2	O. S. C. S. C. C.	1000
	355.8546	. M+2	C.H.*C.3.C.O.	(5)		459.7348	M+4	C	
 -	357,8516	M+4	C.H.**C.**C.O.	ממטפ	-	469.7780	M+2		1000
	367,8949	M+2	13. 13. 13. 13. 13. 13. 13. 13. 13. 13.	ביים ביים		471.7750			(8) 4400
	369.8919	M+4	19C H 2C 17C O	recon (s)		513,6775		C*C	(6) 2220
	409.7974	M+v	C T 3 0 0 0	recup (s)		[422.9278]	LOCK		יים ביים ביים ביים ביים ביים ביים ביים
	[354.9792]	Y SO		HPCDPE	,				
	•	}	2, 20	XX					
n ==	373,8208	M+2	C. H. "CI. "CIO	בייטטים					
	375.8178	4	C,H, acl, acl, o	HXCDE					
	383,8639	Σ	190, H. 201.0						
	385.8610	M+2	13C. H. 3CL 37ClO	UKCDE (8)					
	389.8156			(s) LOSE	•				
	391,8127		0, H. 30, 30, 10	ממאני					
	401.8559		13C, H, 3CI, 3CIO,	HXCDD (8)					
	403.8529	M+4	13C, H, 33CJ, 37CJ, O,	HVCDD (8)					
	445.7555		C ₁₂ H ₂ ² C1 ₈ ² C1 ₂ ²						
	[430,9728]	Lock Lock	C,F ₁ ,	PFK			••		

(a) The following nuclidic masses were used:

H = 1.007625 C = 12.000000 $^{13}C = 13.003355$ F = 18.9984

O = 15.994915 $^{36}Cl = 34.968853$ $^{57}Cl = 36.965903$

S = internal/recovery standard

LDC #:	24	5246	2/
SDG #:	_		•

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:_	/_of	
Reviewer:	Z	7
2nd reviewer:		

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

<u>/Y</u>	N.	N/A
Y.	N	N/A

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

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

Concentration = $(A_s)(I_s)(DF)$ $(A_s)(RRF)(V_s)(\%S)$

A_x = Area of the characteristic ion (EICP) for the compound to be measured

A_{is} = Area of the characteristic ion (EICP) for the specific internal standard

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

V_o = 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.

Examp	ole:
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Sample I.D. 9 , OCDF

Conc. = (80737)(4000)(36142600)(1.37)(10.05)(0.932

0.701919

	-	111	Conce	oorted entration	Calculated Concentration	
#	Sample ID	Compound	()	()	Qualification
	·					
			-			
		·				
		· · · · · · · · · · · · · · · · · · ·				
					 	
-						
		· · · · · · · · · · · · · · · · · · ·				

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:

Tronox LLC Facility, PCS Additional Sampling,

Henderson, Nevada

Collection Date:

November 12, 2010

LDC Report Date:

December 20, 2010

Matrix:

Soil

Parameters:

Dioxins/Dibenzofurans

Validation Level:

Stage 2B & 4

Laboratory:

TestAmerica, Inc.

Sample Delivery Group (SDG): G0K130496

Sample Identification

SSAN6-08-2.0_01_BPC SSAN6-08-3.0_01_BPC SSAN6-08-4.0 01 BPC**

^{**}Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0319266-MB	11/15/10	OCDD	0.36 pg/g	All samples in SDG G0K130496

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

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.

Vil. 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
SSAN6-08-2.0_01_BPC	¹³ C-1,2,3,4,6,7,8-HpCDD ¹³ C-OCDD ¹³ C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 23 (40-135) 32 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

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
SSAN6-08-2.0_01_BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	Р
SSAN6-08-3.0_01_BPC	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 G0K130496	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 G0K130496	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	А

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0K130496

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0K130496	SSAN6-08-2.0_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	Р	Internal standards (%R) (i)
G0K130496	SSAN6-08-2.0_01_BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	Р	Project Quantitation Limit (exceeded range) (e)
G0K130496	SSAN6-08-3.0_01_BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0K130496	SSAN6-08-2.0_01_BPC SSAN6-08-3.0_01_BPC SSAN6-08-4.0_01_BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0K130496	SSAN6-08-2.0_01_BPC SSAN6-08-3.0_01_BPC SSAN6-08-4.0_01_BPC**	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0K130496

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0K130496

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET LDC #: 24524H21 SDG #:__G0K130496 Stage 2B/4 Laboratory: Test America

Date:	12/16/
Page:_	<u>of</u>
Reviewer:	<u> </u>
2nd Reviewer:	4

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: /// 2//0
l1.	HRGC/HRMS Instrument performance check	4	, , ,
III.	Initial calibration	A	
IV.	Routine calibration/ ICV	A	
V.	Blanks	رسي	,
VI.	Matrix spike/Matrix spike duplicates	M	client specified
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N 、	
lX.	Internal standards	SW	
X.	Target compound identifications	4	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	رسي	Not reviewed for Stage 2B validation.
XII.	System performance	7	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	Ν	·
XV.	Field blanks	N	

Note:

A = Acceptable

N = Not provided/applicable SW = See worksheet

ND = No compounds detected

R = Rinsate

D = Duplicate TB = Trip blank

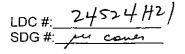
FB = Field blank

EB = Equipment blank

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

	30,0				 	
1	SSAN6-08-2.0_01_BPC	11	0319264	21	31	
2	SSAN6-08-3.0_01_BPC	12		22	 32	
3	SSAN6-08-4.0_01_BPC**	13		23	 33	
4		14		24	34	
5		15		25	 35	
6		16		26	 36	
7		17		27	37	
8		18		28	 38	
9		19		29	39	
10		20		30	40	

Notes:_				



VALIDATION FINDINGS CHECKLIST

	Page:	1	_of_	2
	Reviewer:		F	•
2nd	Reviewer:		O	

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

Validation Area	Yes	No	NA	Findings/Comments
L Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.				
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?		-		
Were the retention time windows established for all homologues?	/	_		
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers \leq 25% ?		-		
Is the static resolving power at least 10,000 (10% valley definition)?				
Was the mass resolution adequately check with PFK?		•		
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?				
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) ≤ 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) \leq 20% for unlabeled standards and \leq 30% for labeled standards?	/	_		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		_		
M 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?	responsibility	3.77% Dec 20		manuer-Alanyda - 19, 00, 10, 10, 10, 10, 10, 10, 10, 10, 10
VIIs Laboratory control samples : 2		Jes.		
Was an LCS analyzed for this SDG?			. <u> </u>	
Was an LCS analyzed per extraction batch?				
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?				

LDC #: 24 524 H2 SDG #: Les comes

VALIDATION FINDINGS CHECKLIST

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

				·
VIII. Regional Quality Assurance and Quality Control	<u></u>			· · · · · · · · · · · · · · · · · · ·
Were performance evaluation (PE) samples performed?			/	7
Were the performance evaluation (PE) samples within the acceptance limits?				
IX::internal:standards			ingini British	
Were internal standard recoveries within the 40-135% criteria?	/		<u> </u>	
Was the minimum S/N ratio of all internal standard peaks ≥ 10?		<u> </u>	<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?	ممريو	_		
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	\angle			
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?	/			
And XII System performance v				
System performance was found to be acceptable.	/			,
XIII Overall assessment of data	4. 19. A 2- 3/2			
Overall assessment of data was found to be acceptable.				
XIV Field duplicales				
Field duplicate pairs were identified in this SDG.			_	
Target compounds were detected in the field duplicates.				
XV:/Field/blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

1/2
524
7
.DC #.

VALIDATION FINDINGS WORKSHEET Rights

Page: __of/______FT Reviewer: ______FT 2nd Reviewer: ________

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

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and

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

Blank extraction date: $\frac{11/5}{10}$ Blank analysis date: $\frac{11/7}{10}$

y′ N N/A

Associated samples: All (75×

Sample Identification - MB 0319266 Blank ID 0.36 Compound Conc. units: D

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 # 24524 Hz)

VALIDATION FINDINGS WORKSHEET Internal Standards

Page: _ot/

Reviewer: FT 2nd Reviewer:

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

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

YN MA

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

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

) #	Date	I sh ID/Reference	Internal Standard		% Pocowood (1 imit: 40.4359/)	Ş	Č	1960
			#	29	- 0h)	1.88/-	4/50/1	F gad F
			7	23)	(1, 1	V 6, &
			5	32				9.0
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)	(
		Internal Standards	Check Standard Used		Recovery Standards	tandards	Ch	Check Standard Used
4	¹³ C-2.3.7.8-TCDF	DP.		K	¹³ C-1.2.3.4-TCDD			
ш	13C-2 3 7 8-TCDD	aac		-	13C-1 2 3 7 8 9-HxCDD			
C	¹³ C-1,2,3,7,8-PeCDE	PecDF		M				
٩	13C-12378-PeCDD	PeCDD		z				
щ	¹³ C-1 2 3 6 7 8-HxCDF	8-HxCDF		d				
ц	¹³ C-1 2 3 6 7 8-HxCDD	8-HxCDD		Δ				
C	¹³ C-1,2,3,4,6,7,8-HpCDE	Z,8-HpCDE		q				
Ξ	13C-1,2,3,4,6,7,8-HpCDD	Z,8-HnCDD		4				
-	13000							

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLS

Page: /of Reviewer: FT 2nd Reviewer:

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

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

Y/N N/A

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).

	Ouslifications	J/A detects (sp)			, c	Jr. detects (K)		711901		(@)				
	Associated Samples	All			ΨV				,	7				
	Finding	All compounds reported below PQL			All compounds reported as EMPC		X'ol col Ross			*				
70.00	Sample 10						H, I. K L, O. P. B	, , , , , , , , , , , , , , , , , , , ,	8					
	# Date													

Comments: See sample calculation verification worksheet for recalculations

24524421 are count SDG#: LDC#:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET



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

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

RRF = $(A_a)(C_{ta})/(A_{ta})(C_a)$ average RRF = sum of the RRFs/number of standards %RSD = 100 * (S/X)

 A_{is} = Area of associated internal standard C_{is} = Concentration of internal standard X = Mean of the RRFs A_x = Area of compound,
C_x = 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 (んろう std)	RRF (da う std)	%RSD	%RSD
	1647	01/04/01	2,3,7,8-TCDF ('3C-2,3,7,8-TCDF)	1.120	0~1.1	hLS1.1	16514	4.94	1994
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.053	1.053	1.1240	ohel.	4.20	4.10
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	08/1	08/-1	1.2326	1.2326	2.74	2.74
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	601-1	1.104	1.1554	65-31-1	13. KZ	3.87
	,		OCDE (13C. OCDD)	1.681	/89:/	1.7419	1.71/9	5.94	15-94
7	1686	0/22/11	2,3,7,8-TCDF (¹3C-2,3,7,8-TCDF)	1.079	6.601	1.0473	1.0473	4.90	1.90
	PBNK		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 (¹³ C-1,2,4,6,7,8,-HpCDD)					,	
			OCDE (3C-OCDD)						
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 (13C-1,2,4,6,7,8,-HpCDD)						
			OCDF (13C-OCDD)						

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

1DC#. 24534#2/ SDG#: 100 com

VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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

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

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

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

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

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

					Reported	Recalculated	Reported	Recaiculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q%
-	cev 00x	C1/ez/,,	2,3,7,8-TCDF (13C-2,3,7,8-TCDF)	6401	1.02	1.02	5.5	5.7
	PB2K	,	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	70:12 Nov	01/61/11	2,3,7,8-TCDF (¹³C-2,3,7,8-TCDF)	1.120	1.07	1.07	4.6	9.6
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)	630.1	811	61.1	77	7.2
			1,2,3,6,7,8-HxCDD (13C-1,2,3,6,7,8-HxCDD)	1.180	51-1	///ک	8.4	2.8
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)	K01.1	611	1.17	5.7	5.7
			OCDE (13C-OCDD)	1891	1.64	1.64	2.2	7.7
3			2,3,7,8-TCDF (*3C-2,3,7,8-TCDF)		,		•	
			2,3,7,8-TCDD (13C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD (13C-1,2,4,6,7,8,-HpCDD)					
			ocpr (1°c-ocpp)					

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

SDG# 445 47 17 4/

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Reviewer: 2

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

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

% Recovery = 100 * SSC/SA \

Where: SSC = Spiked sample concentration SA = Spike added

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

03/9266-10

LCS ID:

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

	S	, s	Spiked S	amole	108	ď	uso I	'n	I CS/I.CSD	csp
Compound	} ₹ €	Added (2)	Concentration	tration	Percent Recovery	ecovery	Percent Recovery	ecovery	RPD	0
	108	l Csn	108	0 1 CSD	Reported	Recato	Reported	Racalc	Reported	Recalculated
2,3,7,8-TCDD	20.02	NΑ	€.61	NA	126	97				
1,2,3,7,8-PeCDD	00/	-	101	V	101	101				
1,2,3,4,7,8-HxCDD	701		1.68		68	68				
1,2,3,4,7,8,9-HpCDF	001		5'08		8	OB				
OCDF	200	7	£ <i>L</i> /	7	28	<i>L8</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

									T				-			-				_		_		_
	Analyte	HPCDF	HpCDF (S)	HPCDF	Нрсрр	HPCDD (S)	NCDPE (3)	:	OCDF	OCDF	0000	OCDD (S)	OCDD (S)	DCDPE	芫									
	Elemental Composition	C;H**Cl;#ClO	12C, H*Cl,O	Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charles of the Charle	C ₁ H*C ₁ "C ₁ O,	12, H 21, 30, 0	C ₁₂ H ³⁶ Cl, ³⁷ Cl ₂ O		C ₁ , ³⁸ Cl, ³⁷ ClO	C ₁₂ *Cl ₂ °Cl ₂ O	C. 2014 CO.	130, 301, 3010,	13C12 32C12O5	C ₁₂ *Cl ₈ *Cl ₂ O	CtoF17									
	lon ID	M+2	Σ¥	M+2	M+4 4+4	M+2 4+2	M+4 LOCK		M+2	M+4	1	₹+5	M+4	M+4	L C S							٠		
141	Accurate Mass	407.7818 409.7788	417.8250 419.8220	423.7767	425.7737 435 8160	437.8140	479.7165 [430.9728]		441.7428	443.7399	459.7348	469.7780	471.7750	513.67/5 [422 q278]										
Docorintor	Descriptor	4							ໝ	,								<u> </u>						
Analyta		TCDF TCDF	TCDF (S)	TCDD	TCDD (S)	TCDD (S)	PFK Tr Tr Tr		PeCDF Pactor	PeCDF (S)	PeCDF (S)	Pecoo	Pecon (S)	PecDD (S)	HpCDPE PFK	-	HXCDE	HXCDF	HXCDF (S)		HXCOD (8)	HXCDD (S)	OCDPE	
Elemental Composition		Cizit 201,0 Cizit 201,0010 100 H 201,0010		C. T. S. C.	19C12H, 35C1, O2	13C ₁₂ H ₄ 3CJ ₃ 7CJO ₂ C H 8CJ 37CJO	0 Fig. 1.5			15 H 30 1 30 10		C174, 2010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010, 1010,	13C, H, 3C, 17C, 13C, 17C, 17C, 17C, 17C, 17C, 17C, 17C, 17	13C ₁₂ H ₃ 3cO ₁ 37CO ₂ O ₂	C,F,33C 37C O C,F,3		C,H,**CI,**CIO	C12H32C137C12O	13C12H, 43CH, 31CHO	C, H, 3Cl, 3'ClO,	12, H, 3CI, 7CIO,	19C12H_33C1_37C12O2	C ₁₂ H ₂ **C ₄ **C ₂ O C ₆ F ₁ ,	<u>.</u>
Ol nol	1	M+ M	M+2	M+2	Σ	01 01 + + E Z	Look	2	M+4	M+2	4+2				M+2 LOCK		M+2	M+4		M+2			LOCK	
Accurate mass ^(a)	303 9016	305.8987 315.9419	317.9389	321.8936	331.9368	375,8364	[354,9792]	339 R597	341.8567	351,9000	355.8546	357.8516	367.8949	369.8919	403.7974 [354.9792]		373,8208	375.8178 383.8639	385.8610	389,8156 391,8127	401.8559	403.8529	[430.9728]	
Descriptor								2				- ·					<u>ო</u>						. <u> </u>	

(a) The following nuclidic masses were used:

H = 1.007825 C = 12.000000 ¹³C = 13.003355 F = 18.9984

0 = 15,994915 $^{35}Cl = 34,968853$ $^{37}Cl = 36,965903$

S = internal/recovery standard

LDC #:_	24	5	2.	442/	,
SDG #:					

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page:	of	
Reviewer:	F	2/
2nd reviewer:_	$ \circ$	

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

METHOD. III	MOC/ IT IN DIOXITS/DIDENZOIGNAMS (LI A ON C	40 Metriod 0200/
Y N N/A Y N N/A	Were all reported results recalculated and Were all recalculated results for detected to	verified for all level IV samples? arget compounds agree within 10.0% of the reported results?
Concentration	(A _*)(RRF)(V _o)(%S)	Example: Sample ID #3 OCDF .
A _x =	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D# 5
A _{is} =	Area of the characteristic ion (EICP) for the specific internal standard	. 142/17/00. 4000
1 _s =	Amount of internal standard added in nanograms	$Conc. = \frac{(180647690(4000))}{(180647690)(4000)}$

Volume or weight of sample extract in milfiliters (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.

Reported Calculated Concentration

Concentration

Concentration

Concentration

Concentration

			Reported Concentration	Calculated Concentration	
#	Sample ID	Compound	()	()	Qualification
	#3	2,3,7,8-7CPF		`	
		= 68124966 /20			
		45/720192 (1.08	(10.13)(0.942)	
		= 29.0pg	19		
			۲		
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