



Laboratory Data Consultants, Inc.

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

December 9, 2010

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

Dear Ms. Arnold,

Enclosed is the final validation report for the fraction listed below. This SDG was received on November 9, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 24493:

SDG #

Fraction

G0I280539

Dioxins/Dibenzofurans

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

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

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

Sincerely,

Erlinda T. Rauto
Operations Manager/Senior Chemist

EDD Stage 2B/4 LDC #24493 (Tronox LLC-Northgate, Henderson NV / Tronox PCS, Additional Sampling)

LDC	SDC#	DATE REC'D	DATE DUE	Dioxins (8290)	W		S		W		S		W		S		W		S		W		S		W		S		W		S		W		S	
					S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	
Matrix: Water/Soil					W	S																														
A	G01280539	11/09/10	12/17/10	0	2																															
A	G01280539	11/09/10	12/17/10																																	
Total	T/LR					0	3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	

EDD CHECKLIST

LDC #: 24493
 SDG #: G0I280539

Page: 1 of 1
 Reviewer: JE
 2nd Reviewer: BC

Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
I. Completeness				
Is there an EDD for the associated Tronox validation report?	X			
II. EDD Qualifier Population				
Were all qualifiers from the validation report populated into the EDD?	X			
III. EDD Lab Anomalies				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			X	See EDD_discrepancy_form_LDC24493_120910.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 27, 2010

LDC Report Date: December 8, 2010

Matrix: Soil

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 2B & 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0I280539

Sample Identification

SSA07-06-0BPC**
SSA08-05-0BPC
SSAN6-08-0.5BPC
SSAN6-08-0.5BPCMS
SSAN6-08-0.5BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and 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
0274292-MB	10/1/10	OCDD	0.51 pg/g	All samples in SDG G0I280539

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
SSAO8-05-0BPC	OCDD	1.0 pg/g	1.0U 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 recovery (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits.

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
SSAO8-05-0BPC	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 G01280539	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 G01280539	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I280539**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I280539	SSAO8-05-0BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0I280539	SSAO7-06-0BPC** SSAO8-05-0BPC SSAN6-08-0.5BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I280539	SSAO7-06-0BPC** SSAO8-05-0BPC SSAN6-08-0.5BPC	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 G0I280539**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I280539	SSAO8-05-0BPC	OCDD	1.0U pg/g	A	bl

**Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I280539**

No Sample Data Qualified in this SDG

LDC #: 24493A21
 SDG #: G01280539
 Laboratory: Test America

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
 Stage 2B/4

Date: 12/8/10
 Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

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

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

Validation Area		Comments	
I.	Technical holding times	Δ	Sampling dates: 9/21/10
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration HEV	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	Δ	
X.	Target compound identifications	Δ	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	Δ	Not reviewed for Stage 2B validation.
XII.	System performance	Δ	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

Soil

1	SSAO7-06-0BPC**	11	0274292'	21		31	
2	SSAO8-05-0BPC	12		22		32	
3	SSAN6-08-03.5BPC	13		23		33	
4	SSAN6-08-03.5BPCMS	14		24		34	
5	SSAN6-08-03.5BPCMSD	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 #: 24493A21
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the retention time windows established for all homologues?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Is the static resolving power at least 10,000 (10% valley definition)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the mass resolution adequately check with PFK?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $< 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
V. Blanks				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank performed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 24493A2¹
 SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

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

VALIDATION FINDINGS WORKSHEET

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

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

Notes:

LDC #: 24493A21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

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

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

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

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		H	no 2nd column confirmation was performed	2	none / p (0)

Comments: See sample calculation verification worksheet for recalculations

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:

$RRF = (A_x)(C_{is}) / (A_{is})(C_x)$
 average RRF = sum of the RRFs/number of standards
 $\%RSD = 100 * (S/X)$
 A_x = Area of compound,
 C_x = Concentration of compound,
 S = Standard deviation of the RRFs,
 A_{is} = Area of associated internal standard
 C_{is} = Concentration of internal standard
 X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (initial)	(%RSD)	Average RRF (initial)	(%RSD)	RRF (initial)	(%RSD)	RRF (initial)	(%RSD)
1	1 CAL	9/14/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.984	1.05	0.984	1.05	1.05	11.8	1.05	11.8
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.06	1.032	1.06	1.06	10.8	1.06	10.8
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.141	1.25	1.141	1.25	1.25	12.7	1.25	12.7
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.134	1.26	1.134	1.26	1.26	12.3	1.26	12.3
			OCDF (¹³ C-OCDF)	2.118	2.36	2.118	2.36	2.36	15.3	2.36	15.3
2	1 CAL	7/26/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.056	1.02	1.056	1.02	1.02	3.32	1.02	3.32
			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 (¹³ C-OCDF)								
3			2,3,7,8-TCDF (¹³ C-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 (¹³ C-OCDF)								

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

LDC #: 24493A21

SDG #: see conts

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

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 = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = $(A_x)(C_{is}) / (A_{is})(C_x)$ RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	CON 5-15	10/7/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	0.924	0.92	6.5	0.92	6.5
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.032	1.02	1.0	1.02	1.0
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.141	1.09	4.8	1.09	4.8
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.137	1.22	7.9	1.22	7.9
			OCDF (¹³ C-OCDF)	2.118	2.20	3.9	2.20	3.9
2	CON - 5-2	10/18/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.06	0.96	9.2	0.96	9.2
			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 (¹³ C-1,2,4,6,7,8-HpCDD)					
			OCDF (¹³ C-OCDF)					
3			2,3,7,8-TCDF (¹³ C-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 (¹³ C-OCDF)					

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

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

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

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

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

RPD = $|MSR - MSDR| * 2 / (MSR + MSDR)$ MSR = Matrix spike percent recovery, MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 4 + 5

Compound	Spike Added		Sample Concentration (ppb)	Spiked Sample Concentration (ppb)		Matrix Spike		Matrix Spike Duplicate		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery			
				Reported	Recalc.	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	20.0	20.4	80	95.1	99.3	75	75	96	96	4.3	4.3
1,2,3,7,8-PeCDD	103	102	280	382	429	97	97	143	143	11	11
1,2,3,4,7,8-HxCDD	103	102	220	292	293	74	74	76	76	0.52	0.52
1,2,3,4,7,8,9-HpCDF	103	102	7700	7780	8280	91	91	576	576	6.2	6.2
OCDF	206	204	51000	51000	55500	0	0	2060	2060	0	0

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

VALIDATION FINDINGS WORKSHEET 1
Laboratory Control Sample Results Verification

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

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

% Recovery = $100 \cdot \text{SSC}/\text{SA}$ Where: SSC = Spiked sample concentration
 SA = Spike added

RPD = $| \text{LCS} - \text{LCSD} | \cdot 2 / (\text{LCS} + \text{LCSD})$ LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0274292 - 102

Compound	Spike Added (ppb)		Spiked Sample Concentration (ppb)		LCS		LCSD		Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalc
2,3,7,8-TCDD	200	NA	14.8	NA	94	94	94	94						
1,2,3,7,8-PeCDD	100		116		116	116	116	116						
1,2,3,4,7,8-HxCDD	100		103		103	103	103	103						
1,2,3,4,7,8,9-HpCDF	100		102		102	102	102	102						
OCDF	200		188		94	94	94	94	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

Descriptor	Accurate mass ^(a)	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass ^(a)	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C ₁₂ H ₇ ³⁵ Cl ₃ O	TCDF	4	407.7818	M+2	C ₁₂ H ₇ ³⁵ Cl ₃ ³⁷ ClO	HpCDF		
	305.8987	M+2	C ₁₂ H ₇ ³⁵ Cl ₃ ³⁷ Cl ₂ O	TCDF		409.7788	M+4	C ₁₂ H ₇ ³⁵ Cl ₅ ³⁷ Cl ₂ O	HpCDF		
	315.9419	M	¹³ C ₁₂ H ₇ ³⁵ Cl ₄ O	TCDF (S)		417.8250	M	¹³ C ₁₂ H ₇ ³⁵ Cl ₇ O	HpCDF (S)		
	317.9389	M+2	¹³ C ₁₂ H ₇ ³⁵ Cl ₃ ³⁷ ClO	TCDF (S)		419.8220	M+2	¹³ C ₁₂ H ₇ ³⁵ Cl ₆ ³⁷ ClO	HpCDF		
	319.8965	M	C ₁₂ H ₇ ³⁵ Cl ₄ O ₂	TCDD		423.7767	M+2	C ₁₂ H ₇ ³⁵ Cl ₇ O ₂	HpCDD		
	321.8936	M+2	C ₁₂ H ₇ ³⁵ Cl ₃ ³⁷ ClO ₂	TCDD		425.7737	M+4	C ₁₂ H ₇ ³⁵ Cl ₆ ³⁷ ClO ₂	HpCDD		
	331.9368	M	¹³ C ₁₂ H ₇ ³⁵ Cl ₄ O ₂	TCDD (S)		435.8169	M+2	¹³ C ₁₂ H ₇ ³⁵ Cl ₇ O ₂	HpCDD (S)		
	333.9338	M+2	¹³ C ₁₂ H ₇ ³⁵ Cl ₃ ³⁷ ClO ₂	TCDD (S)		437.8140	M+4	¹³ C ₁₂ H ₇ ³⁵ Cl ₆ ³⁷ ClO ₂	HpCDD (S)		
	375.8364	M+2	C ₁₂ H ₇ ³⁵ Cl ₅ ³⁷ ClO	HxCDFE		479.7165	M+4	C ₁₂ H ₇ ³⁵ Cl ₇ ³⁷ ClO	HpCDD (S)		
	[354.9792]	LOCK	C ₉ F ₁₃	PFK		[430.9728]	LOCK	C ₉ F ₁₇	NCDPE		
										PFK	
	2	339.8597	M+2	C ₁₂ H ₉ ³⁵ Cl ₄ ³⁷ ClO		PeCDF	5	441.7428	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ ClO	OCDF
		341.8567	M+4	C ₁₂ H ₉ ³⁵ Cl ₆ ³⁷ Cl ₂ O		PeCDF		443.7399	M+4	C ₁₂ ³⁵ Cl ₉ ³⁷ Cl ₂ O	OCDF
351.9000		M+2	¹³ C ₁₂ H ₉ ³⁵ Cl ₄ ³⁷ ClO	PeCDF (S)	457.7377	M+2		¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD		
353.8970		M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₆ ³⁷ Cl ₂ O	PeCDF (S)	459.7348	M+4		¹³ C ₁₂ ³⁵ Cl ₉ ³⁷ Cl ₂ O ₂	OCDD		
355.8546		M+2	C ₁₂ H ₉ ³⁵ Cl ₅ ³⁷ ClO ₂	PeCDD	469.7780	M+2		¹³ C ₁₂ ³⁵ Cl ₈ ³⁷ ClO ₂	OCDD (S)		
357.8516		M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₇ ³⁷ ClO ₂	PeCDD	471.7750	M+4		¹³ C ₁₂ ³⁵ Cl ₁₀ ³⁷ ClO ₂	OCDD (S)		
367.8949		M+2	¹³ C ₁₂ H ₉ ³⁵ Cl ₄ ³⁷ ClO ₂	PeCDD (S)	513.6775	M+4		¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ Cl ₂ O ₂	OCDD (S)		
369.8919		M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	PeCDD (S)	[422.9278]	LOCK		C ₁₂ ³⁵ Cl ₉ ³⁷ Cl ₂ O	DCDPE		
409.7974		M+2	C ₁₂ H ₉ ³⁵ Cl ₅ ³⁷ ClO	HpCDFE					PFK		
[354.9792]		LOCK	C ₉ F ₁₃	PFK							
3		373.8208	M+2	C ₁₂ H ₉ ³⁵ Cl ₃ ³⁷ ClO	HxCDF						
		375.8178	M+4	C ₁₂ H ₉ ³⁵ Cl ₅ ³⁷ Cl ₂ O	HxCDF						
	383.8639	M	¹³ C ₁₂ H ₉ ³⁵ Cl ₃ ³⁷ ClO	HxCDF (S)							
	385.8610	M+2	¹³ C ₁₂ H ₉ ³⁵ Cl ₆ O	HxCDF (S)							
	389.8156	M+2	C ₁₂ H ₉ ³⁵ Cl ₄ ³⁷ ClO ₂	HxCDD							
	391.8127	M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₇ ³⁷ ClO ₂	HxCDD							
	401.8559	M+2	C ₁₂ H ₉ ³⁵ Cl ₅ ³⁷ ClO ₂	HxCDD							
	403.8529	M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₈ ³⁷ ClO ₂	HxCDD							
	445.7555	M+4	¹³ C ₁₂ H ₉ ³⁵ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD (S)							
	[430.9728]	LOCK	C ₉ F ₁₇	OCDFE							

(e) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24493A21
SDG #: pe cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

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

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

Y N N/A 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?

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

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

Example:

Sample I.D. #1, OCDF:

$$\text{Conc.} = \frac{(388751000)(4000)}{(295462000)(2.12)(10.4)(0.9908)}$$

= 240 pg/g

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
		2,3,7,8-TCDF 2	20494590	(2000)	
			475704000	(1.06)(10.4)(0.9908)	
		= 7.9 pg/g			