



LABORATORY DATA CONSULTANTS, INC.

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

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

July 16, 2010

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

Dear Ms. Arnold,

Enclosed is the final validation report for the fraction listed below. This SDG was received on June 29, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 23510:

<u>SDG #</u>	<u>Fraction</u>
G0E270651	Dioxins/Dibenzofurans

The data validation was performed under Stage 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/Dibenzofurans Data Review, September 2005

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

Sincerely,

Erlinda T. Rauto
Operations Manager/Senior Chemist

LDC #23510 (Tronox LLC-Northgate, Henderson NV / Tronox PCS)

Stage 2B/4

LDC	SDG#	DATE REC'D	(3) DATE DUE	Dioxins (8290)																	
				W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
Matrix: Water/Soil																					
A	G0E270651	06/29/10	07/21/10	0	1																
Total	T/LR																				
																					0 1 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 1

Shaded cells indicate Stage 4 validation (all other cells are Stage 2B validation). These sample counts do not include MS/MSD, and DUPs

EDD CHECKLIST

LDC #: 23510
 SDG #: G0E270651

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_LDC23510_071510.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

**Tronox LLC Facility, PCS, Henderson, Nevada
Data Validation Reports
LDC #23510**

Dioxins/Dibenzofurans

LDC

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

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

Collection Date: May 25, 2010

LDC Report Date: July 13, 2010

Matrix: Soil

Parameters: Dioxins/Dibenzofurans

Validation Level: Stage 4

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): G0E270651

Sample Identification

SSAR3-01-1BPC

SSAR3-01-1BPCMS

SSAR3-01-1BPCMSD

Introduction

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

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

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

The following are definitions of the data qualifiers:

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

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
0154348MB	6/3/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.14 pg/g 0.25 pg/g 0.31 pg/g 0.19 pg/g 0.16 pg/g 0.11 pg/g 0.45 pg/g 0.15 pg/g 0.67 pg/g	All samples in SDG G0E270651

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
SSAR3-01-1BPC	2,3,4,6,7,8-HxCDF	0.70 pg/g	0.70U pg/g

Sample FB04062010-RZB (from SDG G0D120488) was identified as a field blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank with the following exceptions:

Field Blank ID	Sampling Date	Compound	Concentration	Associated Samples
FB04062010-RZB	4/6/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.68 pg/L 2.5 pg/L 6.2 pg/L 2.7 pg/L 1.4 pg/L 0.82 pg/L 0.94 pg/L 1.8 pg/L 1.2 pg/L 4.4 pg/L	All samples in SDG G0E270651

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

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. 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
SSAR3-01-1BPC	¹³ C-OCDD	34 (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.

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAR3-01-1BPC	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 G0E270651	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0E270651	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

XII. System Performance

The system performance was acceptable.

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

**Tronox LLC Facility, PCS, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0E270651**

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0E270651	SSAR3-01-1BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0E270651	SSAR3-01-1BPC	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0E270651	SSAR3-01-1BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0E270651	SSAR3-01-1BPC	All compounds reported as EMPC	JK (all detects)	A	Project Quantitation Limit (k)

**Tronox LLC Facility, PCS, Henderson, Nevada
Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG
G0E270651**

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0E270651	SSAR3-01-1BPC	2,3,4,6,7,8-HxCDF	0.70U pg/g	A	bl

**Tronox LLC Facility, PCS, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0E270651**

No Sample Data Qualified in this SDG

Tronox Northgate Henderson

VALIDATION COMPLETENESS WORKSHEET

LDC #: 23510A21
 SDG #: G0E270651
 Laboratory: Test America

Stage 4

Date: 7/2/10
 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	A	Sampling dates: 5/25/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ NSV	A	
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	W	
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	W	
XII.	System performance	A	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	W	FB04062010-RZB(GOD)20488)

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:

1	SSAR3-01-1BPC	S	11	0154/348MB	21	31
2	SSAR3-01-1BPCMS		12		22	32
3	SSAR3-01-1BPCMSD		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 #: _____
 SDG #: _____

VALIDATION FINDINGS CHECKLIST

Page: ___ of ___
 Reviewer: _____
 2nd Reviewer: _____

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	/			
Were the retention time windows established for all homologues?	/			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers $\leq 25\%$?	/			
Is the static resolving power at least 10,000 (10% valley definition)?	/			
Was the mass resolution adequately check with PFK?	/			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	/			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	/			
Were all percent relative standard deviations (%RSD) $\leq 20\%$ for unlabeled standards and $< 30\%$ for labeled standards?	/			
Did all calibration standards meet the Ion Abundance Ratio criteria?	/			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10 ?	/			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	/			
Were all percent differences (%D) $\leq 20\%$ for unlabeled standards and $\leq 30\%$ for labeled standards?	/			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	/			
V. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank performed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?	/			
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			

VALIDATION FINDINGS CHECKLIST

VIII. Regional Quality Assurance and Quality Control			
Were performance evaluation (PE) samples performed?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were the performance evaluation (PE) samples within the acceptance limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
IX. Internal standards			
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the minimum S/N ratio of all internal standard peaks ≥ 10 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the signal to noise ratio for each target compound and labeled standard > 2.5 ?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
For PCDF identification, was any signal ($S/N \geq 2.5$, at \pm seconds RT) detected in the corresponding PCDF channel?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Was an acceptable lock mass recorded and monitored?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XI. Compound quantitation/CRQLs			
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XII. System performance			
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XIII. Overall assessment of data			
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XIV. Field duplicates			
Field duplicate pairs were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field duplicates.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
XV. Field blanks			
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

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:

VALIDATION FINDINGS WORKSHEET

Blanks

METHOD: HRGC/HRMS Dioxins (EPA 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 whenever a sample extraction was performed?
- Y N N/A Was the method blank contaminated? If yes, please see qualification below.

Blank extraction date: 6/3/10 Blank analysis date: 6/14/10

Conc. units: pg/L Associated samples: All (b1)

Compound	Blank ID	Sample Identification	
		5X	1
F	0.14	0.7	-
G	0.25	1.25	-
K	0.31	1.55	-
L	0.19	0.95	-
M	0.16	0.8	0.70/U
N	0.11	0.55	-
O	0.45	2.25	-
P	0.15	0.75	-
Q	0.67	3.35	-

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 #: 2380A-1
 SDG #: 201000001

VALIDATION FINDINGS WORKSHEET
Internal Standards

Page: 1 of 1
 Reviewer: [Signature]
 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 Are all internal standard recoveries within the 40-135% criteria?

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications (1)
		1	J J	34 (40-135)	Y (N) A (F) (R)
		2 (MS)	J	34	No label
		3 (MSD)	J	37	✓

Internal Standards		Check Standard Used	Internal Standards	Check Standard Used
A.	¹³ C-2,3,7,8-TCDF		I.	¹³ C-OCDD
B.	¹³ C-2,3,7,8-TCDD		K.	¹³ C-1,2,3,4-TCDD
C.	¹³ C-1,2,3,7,8-PeCDF		L.	¹³ C-1,2,3,7,8,9-HxCDD
D.	¹³ C-1,2,3,7,8-PeCDD		M.	
E.	¹³ C-1,2,3,4,7,8-HxCDF		N.	
F.	¹³ C-1,2,3,6,7,8-HxCDD		O.	
G.	¹³ C-1,2,3,4,6,7,8-HpCDF		P.	
H.	¹³ C-1,2,3,4,6,7,8-HpCDD			

LDC #: 2357021
 SDG #: WPC0001

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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 Reviewer: [Signature]
 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".

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

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		<u>1</u>	<u>No 2.3.7.8-TCDF confirmation</u>		<u>None</u>
		<u>MM</u>	<u>ZMPC results</u>	<u>MM</u>	<u>JK(k)</u>

Comments: See sample calculation verification worksheet for recalculations

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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

$RRF = (A_x)(C_s)/(A_s)(C_x)$
 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_s = Area of associated internal standard
 C_s = 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 (CS3 std)	%RSD	RRF (CS3 std)	%RSD
1	1.022 (A05)	5/18/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.004		1.004		1.06	8.10	1.06	8.24
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.049		1.049		1.06	5.12	1.06	5.00
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.163		1.163		1.20	9.25	1.20	8.13
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.073		1.073		1.11	7.66	1.11	7.86
			OCDF (¹³ C-OCDD)	1.523		1.523		1.58	8.42	1.58	8.35
2			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-OCDD)								
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-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.

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

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

% Difference = $100 \times (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$ Where: ave. RRF = initial calibration average RRF
 RRF = $(A_x / C_x) / (A_s / C_s)$ RRF = continuing calibration RRF
 A_x = Area of compound, A_s = Area of associated internal standard
 C_x = Concentration of compound, C_s = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	13N104105	6/4/10	2,3,7,8-TCDF (¹³ C-2,3,7,8-TCDF)	1.004	1.01	1.01	1.01	1.1
			2,3,7,8-TCDD (¹³ C-2,3,7,8-TCDD)	1.049	1.01	1.01	3.5	3.5
			1,2,3,6,7,8-HxCDD (¹³ C-1,2,3,6,7,8-HxCDD)	1.163	1.15	1.15	1.2	1.1
			1,2,3,4,6,7,8-HpCDD (¹³ C-1,2,4,6,7,8-HpCDD)	1.073	1.08	1.08	0.7	0.7
			OCDF (¹³ C-OCDD)	1.523	1.53	1.53	0.4	0.4
2			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-OCDD)					
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-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.

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 = $100 * MSR - MSDR / (MSR + MSDR)$ MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: _____

Compound	Spike Added (PS/G)		Sample Concentration (PS/G)	Spiked Sample Concentration (PS/G)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		Reported RPD	Recalculated RPD
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.		
2,3,7,8-TCDD	21.4	21.9	0.083	20.0	21.1	93	93	96	96	5.0	5.1
1,2,3,7,8-PeCDD	107	109	ND	108	115	101	101	105	105	6.1	6.3
1,2,3,4,7,8-HxCDD	↓	↓	0.20	105	87.6	98	98	80	80	1.8	1.8
1,2,3,4,7,8,9-HpCDF	↓	↓	3.5	116	122	106	105	108	109	4.5	5.0
OCDF	214	219	20	269	268	116	116	113	113	0.39	0.37

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
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: 0154348

Compound	Spike Added (ppb)		Spiked Sample Concentration (ppb)		LCS		LCSD		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	188	NA	92	94						
1,2,3,7,8-PeCDD	100		105		105	105						
1,2,3,4,7,8-HxCDD			95.1		95	95						
1,2,3,4,7,8,9-HpCDF			94.2		92	94						
OCDF	200		188		94	94						

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 HHGC/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 ₄ ³⁵ Cl ₁₀	TCDF	4	407.7818	M+2	C ₁₂ H ₃₅ Cl ₉ ³⁷ ClO	HpCDF			
	305.8987	M+2	C ₁₂ H ₃₅ Cl ₉ ³⁷ Cl ₁₀	TCDF		M+4	409.7788	M+4	C ₁₂ H ³⁵ Cl ₅ ³⁷ Cl ₅ O	HpCDF		
	315.9419	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₁₀	TCDF (S)		M	417.8250	M	¹³ C ₁₂ H ³⁵ Cl ₉ O	HpCDF (S)		
	317.9389	M+2	¹³ C ₁₂ H ₃₅ Cl ₉ ³⁷ ClO	TCDF (S)		M+2	419.8220	M+2	¹³ C ₁₂ H ³⁵ Cl ₈ ³⁷ ClO	HpCDF		
	319.8965	M	C ₁₂ H ₄ ³⁵ Cl ₉ O ₂	TCDD		M+2	423.7767	M+2	C ₁₂ H ³⁵ Cl ₈ ³⁷ ClO ₂	HpCDD		
	321.8936	M+2	C ₁₂ H ₃₅ Cl ₈ ³⁷ ClO ₂	TCDD		M+4	425.7737	M+4	C ₁₂ H ³⁵ Cl ₇ ³⁷ ClO ₂	HpCDD		
	331.9368	M	¹³ C ₁₂ H ₄ ³⁵ Cl ₉ O ₂	TCDD (S)		M+2	435.8169	M+2	¹³ C ₁₂ H ³⁵ Cl ₈ ³⁷ ClO ₂	HpCDD (S)		
	333.9338	M+2	¹³ C ₁₂ H ₃₅ Cl ₈ ³⁷ ClO ₂	TCDD (S)		M+4	437.8140	M+4	¹³ C ₁₂ H ³⁵ Cl ₇ ³⁷ ClO ₂	HpCDD (S)		
	375.8364	M+2	C ₁₂ H ₄ ³⁵ Cl ₉ ³⁷ ClO	HxCDFPE		M+4	479.7165	M+4	C ₁₂ H ³⁵ Cl ₈ ³⁷ Cl ₂ O	NCDFPE		
	[354.9792]	LOCK	C ₉ F ₁₀	PFK		LOCK	[430.9728]	LOCK	C ₉ F ₁₇	PFK		
	2	339.8597	M+2	C ₁₂ H 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 ₃₅ Cl ₃ ³⁷ Cl ₂ O		PeCDF		M+4	443.7399	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O	OCDF
		351.9000	M+2	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O		PeCDF (S)		M+2	457.7377	M+2	C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD
		353.8970	M+4	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O		PeCDF (S)		M+4	459.7348	M+4	C ₁₂ ³⁵ Cl ₆ ³⁷ Cl ₂ O ₂	OCDD
355.8546		M+2	C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO ₂	PeCDD	M+2	469.7780		M+2	¹³ C ₁₂ ³⁵ Cl ₇ ³⁷ ClO ₂	OCDD (S)		
357.8516		M+4	C ₁₂ H ₃₅ Cl ₃ ³⁷ ClO ₂	PeCDD	M+4	471.7750		M+4	¹³ C ₁₂ ³⁵ Cl ₆ ³⁷ ClO ₂	OCDD (S)		
367.8949		M+2	¹³ C ₁₂ H ₃₅ Cl ₄ ³⁷ ClO ₂	PeCDD (S)	M+2	513.6775		M+2	¹³ C ₁₂ ³⁵ Cl ₃ ³⁷ Cl ₂ O ₂	OCDD (S)		
369.8919		M+4	¹³ C ₁₂ H ₃₅ Cl ₃ ³⁷ Cl ₂ O ₂	PeCDD (S)	M+4	[422.9278]		M+4	C ₁₂ ³⁵ Cl ₈ ³⁷ Cl ₂ O	DCDFPE		
409.7974		M+2	C ₁₂ H ₃₅ Cl ₆ ³⁷ ClO	HxCDFPE	LOCK			LOCK	C ₁₀ F ₁₇	PFK		
[354.9792]		LOCK	C ₉ F ₁₃	PFK								
3		373.8208	M+2	C ₁₂ H ₂₅ Cl ₄ ³⁷ ClO	HxCDF							
		375.8178	M+4	C ₁₂ H ₂₅ Cl ₃ ³⁷ Cl ₂ O	HxCDF							
		383.8639	M	¹³ C ₁₂ H ₂₅ Cl ₄ ³⁷ Cl ₂ O	HxCDF (S)							
		385.8610	M+2	¹³ C ₁₂ H ₂₅ Cl ₃ ³⁷ ClO	HxCDF (S)							
	389.8156	M+2	C ₁₂ H ₂₅ Cl ₃ ³⁷ ClO ₂	HxCDD								
	391.8127	M+4	C ₁₂ H ₂₅ Cl ₃ ³⁷ ClO ₂	HxCDD								
	401.8559	M+2	¹³ C ₁₂ H ₂₅ Cl ₄ ³⁷ Cl ₂ O ₂	HxCDD (S)								
	403.8529	M+4	¹³ C ₁₂ H ₂₅ Cl ₃ ³⁷ Cl ₂ O ₂	HxCDD (S)								
	445.7555	M+4	C ₁₂ H ₂₅ Cl ₃ ³⁷ Cl ₂ O	HxCDD (S)								
	[430.9728]	LOCK	C ₉ F ₁₇	OCDFPE								
				PFK								

(a) 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 #: 03570A-1
SDG #: See 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)

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

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

- 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_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. 1, F:

$$\text{Conc.} = \frac{(131124)(2000)}{(34928)(1.073)(10.43)(0.914)}$$

= 0.734 ppb/g

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification