



**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

August 19, 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 August 5, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

**LDC Project # 23730:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
G0F140437	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/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 CHECKLIST

LDC #: 23730  
 SDG #: G0F140437

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

Tronox Northgate Henderson Worksheet

EDD Area	Yes	No	NA	Findings/Comments
<b>I. Completeness</b>				
Is there an EDD for the associated Tronox validation report?	X			
<b>II. EDD Qualifier Population</b>				
Were all qualifiers from the validation report populated into the EDD?	X			
<b>III. EDD Lab Anomalies</b>				
Were EDD anomalies identified?		X		
If yes, were they corrected or documented for the client?			X	See EDD_discrepancy_form_LDC23730_081810.doc
<b>IV. EDD Delivery</b>				
Was the final EDD sent to the client?	X			

**Tronox LLC Facility, PCS, Henderson, Nevada  
Data Validation Reports  
LDC #23730**

Dioxins/Dibenzofurans

**LDC**

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

**Project/Site Name:** Tronox LLC Facility, PCS, Henderson, Nevada  
**Collection Date:** May 19, 2010  
**LDC Report Date:** August 18, 2010  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**Validation Level:** Stage 4  
**Laboratory:** TestAmerica, Inc.

**Sample Delivery Group (SDG):** G0F140437

**Sample Identification**

SSAK7-02-14BPC  
SSAK7-02-14BPCMS  
SSAK7-02-14BPCMSD

## 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) 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
G0F160000-394B	6/16/10	1,2,3,4,6,7,8-HpCDD OCDD 2,3,7,8-TCDF 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 1,2,3,4,6,7,8-HpCDF	0.15 pg/g 1.4 pg/g 0.23 pg/g 0.34 pg/g 0.63 pg/g 0.14 pg/g 1.2 pg/g 1.2 pg/g	All samples in SDG G0F140437

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

Sample FB-04072010-RZD (from SDG G0D090441) 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
FB-04072010-RZD	4/7/10	1,2,3,4,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.89 pg/L 1.5 pg/L 2.2 pg/L 8.3 pg/L 1.4 pg/L 1.6 pg/L 1.5 pg/L 1.6 pg/L 1.3 pg/L 1.4 pg/L 4.1 pg/L	All samples in SDG G0F140437

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.

### 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.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0F140437	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 G0F140437	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 G0F140437**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason (Code)</b>
G0G010578	SSAK7-02-14BPC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0G010578	SSAK7-02-14BPC	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 G0F140437**

No Sample Data Qualified in this SDG

**Tronox LLC Facility, PCS, Henderson, Nevada  
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0F140437**

No Sample Data Qualified in this SDG

**Tronox Northgate Henderson**  
**VALIDATION COMPLETENESS WORKSHEET**

LDC #: 23730A21  
SDG #: G0F140437  
Laboratory: Test America

Stage 4

Date: 8/15/10  
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: <u>5/19/10</u>
II.	HRGC/HRMS Instrument performance check	Δ	
III.	Initial calibration	Δ	
IV.	Routine calibration <del>REV</del>	Δ	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	A	
VII.	Laboratory control samples	Δ	LC
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	Δ	
X.	Target compound identifications	Δ	
XI.	Compound quantitation and CRQLs	Δ	
XII.	System performance	Δ	
XIII.	Overall assessment of data	Δ	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	FB = <u>04077210-R2D</u>

SDG # G0D090441

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:

SOIL

1	SSAK7-02-14BPC	11	<u>G0F160000-39413</u>	21		31	
2	SSAK7-02-14BPCMS	12		22		32	
3	SSAK7-02-14BPCMSD	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 #: 23730A2  
 SDG #: mu cones

**VALIDATION FINDINGS CHECKLIST**

Page: 1 of 2  
 Reviewer: FJ  
 2nd Reviewer: \_\_\_\_\_

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
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"/>	
<b>II. GC/MS Instrument performance check</b>				
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 < 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"/>	
<b>III. Initial calibration</b>				
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) ≤ 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"/>	
<b>IV. Continuing calibration</b>				
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) ≤ 20% for unlabeled standards and ≤ 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"/>	
<b>V. Blanks</b>				
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"/>	
<b>VI. Matrix spike/Matrix spike duplicates</b>				
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"/>	
<b>VII. Laboratory control samples</b>				
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 #: 23730A21  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>	
IX. Internal standards				
Were internal standard recoveries within the 40-135% criteria?	<input checked="" type="checkbox"/>			
Was the minimum S/N ratio of all internal standard peaks $\geq 10$ ?	<input checked="" 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"/>			
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"/>			
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"/>			
Did compound spectra contain all characteristic ions listed in the table attached?	<input checked="" type="checkbox"/>			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	<input checked="" type="checkbox"/>			
Was the signal to noise ratio for each target compound and labeled standard $> 2.5$ ?	<input checked="" type="checkbox"/>			
Does the maximum intensity of each specified characteristic ion coincide within $\pm 2$ seconds (includes labeled standards)?	<input checked="" type="checkbox"/>			
For PCDF identification, was any signal ( $S/N \geq 2.5$ , at $\pm$ seconds RT) detected in the corresponding PCDPE channel?	<input checked="" type="checkbox"/>			
Was an acceptable lock mass recorded and monitored?	<input checked="" 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"/>			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>			
XII. System performance				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		<input checked="" type="checkbox"/>		
Target compounds were detected in the field duplicates.			<input checked="" type="checkbox"/>	
XV. Field blanks				
Field blanks were identified in this SDG.		<input checked="" type="checkbox"/>		
Target compounds were detected in the field blanks.			<input checked="" 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**  
**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 (CS > std)	%RSD	RRF (CS > std)	%RSD
1	KCAL	6/3/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.94366	9.54294	0.94366	9.54294	1.00695	9.54294	1.00695	9.54
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.13874	5.54538	1.13874	5.54538	1.15121	5.54538	1.15121	5.54
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.13023	8.34219	1.13023	8.34219	1.19409	8.34219	1.19409	8.34
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.02417	10.06798	1.02417	10.06798	1.047	10.06798	1.047	10.07
			OCDF ( <sup>13</sup> C-OCDF)	1.48668	9.14625	1.48668	9.14625	1.55779	9.14625	1.55779	9.15
2	KCAL		2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	1.038	1.29	1.038	1.29	1.10	1.29	1.10	1.29
	PB225		<del>2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</del>								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> 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.

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

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	ceV	6/17/10 9:13	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.94346	0.98688	4.6	0.98688	4.6
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	1.13874	1.1155	2.4	1.1155	2.4
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	1.13023	1.18233	4.6	1.18233	4.6
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	1.02417	1.10698	8.1	1.10698	8.1
			OCDF ( <sup>13</sup> C-OCDF)	1.48668	1.62420	9.3	1.62420	9.3
2	ceV DB225	6/21/10	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)		0.99	9.3		9.3
			<del>2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD)</del>					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDF)					
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> 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.

LDC #: 23730A21  
 SDG #: per con

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

Page: 1 of 1  
 Reviewer: F7  
 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 =  $100 * MSR - MSDR$  MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 2 + 3

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		Reported	Recalculated
	MS	MSD		MS	MSD	Percent Recovery	RPD	Percent Recovery	RPD		
2,3,7,8-TCDD	21.3 <del>0.21</del>	20.1 <del>0.3</del>	0.31 <del>0.21</del>	19.9 <del>19.1</del>	92	92	91	91	4.3	4.3	
1,2,3,7,8-PeCDD	106 <del>0.48</del>	103 <del>0.78</del>	0.68 <del>0.35</del>	106	99	100	100	1.4	1.4		
1,2,3,4,7,8-HxCDD	106	103	0.35	104	98	93	93	7.8	7.8		
1,2,3,4,7,8,9-HpCDF	106	103	2.4	142	111	112	112	1.6	1.6		
OCDF	213	207	19.10	450	125	134	134	2.6	2.6		

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 =  $100 \cdot \frac{\text{LCS} - \text{LCSD}}{\text{LCS} + \text{LCSD}}$  LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 1057

Compound	Spike Added (ppb)		Spiked Sample Concentration		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	20.0	NA	19.7	NA	99	99	99	99						
1,2,3,7,8-PeCDD	100		109		109	109	109	109						
1,2,3,4,7,8-HxCDD	100		108		108	108	108	108						
1,2,3,4,7,8,9-HpCDF	100		97.7		98	98	98	98						
OCDF	200		211		108	108	108	108	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 <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sub>35</sub> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	HxCDF		479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	NCDPE		
										PFK	
	2	339.8597	M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>		PeCDF (S)		457.7377	M+2	<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
353.8970		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	PeCDF (S)	459.7348	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD		
355.8546		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	PeCDD (S)	513.6775	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDD (S)		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)	[422.9278]	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
409.7974		M+2	C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	HxCDF		LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>	HxCDF (S)						
	385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> C <sub>10</sub>	HxCDF (S)							
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD							
	401.8559	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	HxCDD (S)							
	403.8529	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDD (S)							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	OCDF							

(e) The following nuclidic masses were used:

- H = 1.007825
- C = 12.000000
- <sup>13</sup>C = 13.003355
- F = 18.9984
- O = 15.994915
- <sup>35</sup>Cl = 34.968853
- <sup>37</sup>Cl = 36.965903

S = internal/recovery standard