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 30, 2010

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

Dear Ms. Arnold,

Enclosed are the final validation reports for the fraction listed below. These SDGs were received on December 7, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 24524:

SDG #	Fraction
G0J130426, G0J140638, G0J150566 G0J170404, G0J200489, G0J230497 G0J270514, G0K130496	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

Stage 2B/4

LDC #24524 (Tronox LLC-Northgate, Henderson NV / Tronox PCS, Additional Sampling)

LDC	SDG#	DATE REC'D	(3) DATE DUE	Dioxins (8290)																															
Matrix:	Water/Soil			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	W	S
A	G0J130426	12/07/10	12/28/10	0	1																														
A	G0J130426	12/07/10	12/28/10	0	1																														
B	G0J140638	12/07/10	12/28/10	0	7																														
B	G0J140638	12/07/10	12/28/10	0	1																														
C	G0J150566	12/07/10	12/28/10	0	4																														
C	G0J150566	12/07/10	12/28/10	0	1																														
D	G0J170404	12/07/10	12/28/10	0	2																														
D	G0J170404	12/07/10	12/28/10	0	1																														
E	G0J200489	12/07/10	12/28/10	0	1																														
F	G0J230497	12/07/10	12/28/10	1	15																														
F	G0J230497	12/07/10	12/28/10	0	1																														
G	G0J270514	12/07/10	12/28/10	1	20																														
G	G0J270514	12/07/10	12/28/10	0	2																														
H	G0K130496	12/07/10	12/28/10	0	2																														
H	G0K130496	12/07/10	12/28/10	0	1																														
Total	T/LR			2	60	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	0	62

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 #: 24524
SDG #: G0J130426, G0J140638, G0J150566, G0J170404
G0J200489, G0J230497, G0J270514, G0K130496

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_LDC24524_122910.doc
IV. EDD Delivery				
Was the final EDD sent to the client?	X			

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	September 2, 2010
LDC Report Date:	December 23, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J130426

Sample Identification

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

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

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

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits.

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN7-04-4BPC SSAN7-04-5BPC**	2,3,7,8-TCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J130426	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

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

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	2,3,7,8-TCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J130426	SSAN7-04-4BPC SSAN7-04-5BPC**	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524A21
SDG #: G0J130426
Laboratory: Test America

Date: 12/16/10
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: 9/2/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration ~~ICV~~	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

1 †	SSAN7-04-4BPC	11	028748 1-MB	21		31	
2	SSAN7-04-5BPC**	12		22		32	
3		13		23		33	
4		14		24		34	
5		15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

LDC #: 24524A2/
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Reviewer: KP
2nd Reviewer: CV

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524A21
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer: [illegible]
2nd Reviewer: [illegible]

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524A21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: FT
2nd Reviewer: Q

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

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?

Blank extraction date: 9/27/10 **Blank analysis date:** 10/15/10 **Associated samples:** All 75X

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	028748I-MB									
F	0.089									
G	0.31									
H	0.065									
K	0.070									
L	0.043									
O	0.15									
P	0.070									
Q	0.36									

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 #: 24524A2/

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

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

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

#	Date	~~Sample ID~~ compd	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		H	x'd cal range	All	J/Pdet (e)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524A27
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: EJ
2nd Reviewer: 9

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (C33 std)	Recalculated RRF (C33 std)	Reported %RSD	Recalculated %RSD
1	ICAL	10/8/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.96170	0.96170	1.04206	1.04206	5.67386	5.674
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.10069	1.10069	1.15267	1.15267	5.92978	5.929
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.16239	1.16239	1.31722	1.31722	9.54572	9.546
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.06001	1.06001	1.12727	1.12727	4.82825	4.828
			OCDF (^{13}C-OCDD)	1.39942	1.39942	1.51113	1.51113	7.02964	7.029
2	DB225	7/26/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.056	1.056	1.02	1.02	3.32	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-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.

LDC #: 24524A21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page: 1 of 1
Reviewer: EJ
2nd Reviewer: CL

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 13:58	10/15/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.96170	1.01702	1.01702	5.8	5.8
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.10069	1.17718	1.17718	6.9	6.9
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.16239	1.17252	1.17252	0.9	0.9
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.06007	1.13967	1.13967	7.5	7.5
			OCDF (^{13}C-OCDD)	1.39942	1.49722	1.49722	7.0	7.0
2	CCV 8:41	10/26/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.056	1.15	1.15	9.0	9.0
			~~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	CCV 10:44	10/27/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.052	1.04	1.04	1.8	1.8
			~~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.

LDC #: 24524A1/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: EF
2nd Reviewer: [illegible]

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 028748/

Compound	Spike Added (pg/g)		Spiked Sample Concentration ( )		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	19.3	NA	97	97				
1,2,3,7,8-PeCDD	100		94.0		94	94				
1,2,3,4,7,8-HxCDD	100		98.3		98	98				
1,2,3,4,7,8,9-HpCDF	100		102		102	102				
OCDF	200		187		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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24524A21
SDG #: pre cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer:
2nd reviewer:

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

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

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

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

Conc. = $\frac{(33337.74)(2000)}{(2894749)(1.101)(10.43)(0.918)}$

= 2.2 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	#2	2,3,7,8-TCDF			
		$= \frac{1102412.544\ (2000)}{559\,457.648\,(1.056)\,(10.43)\,(0.918)}$			
		= 390	pg/g		

RECALC90.21

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	October 12, 2010
LDC Report Date:	December 20, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J140638

Sample Identification

SSAL2-04-1_01_BPC
SSAL2-04-2_01_BPC
SSAL2-04-3_01_BPC
SSAL2-04-4_01_BPC**
SSAL2-05-1_01_BPC
SSAL2-05-2_01_BPC
SSAL2-05-3_01_BPC
SSAL2-05-4_01_BPC
SSAL2-04-3_01_BPCMS
SSAL2-04-3_01_BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

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

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

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAL2-04-2_01_BPC	^{13}C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-04-3_01_BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	24 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAL2-04-4_01_BPC**	^{13}C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-05-1_01_BPC	^{13}C-OCDD	36 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAL2-05-2_01_BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAL2-05-3_01_BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAL2-05-4_01_BPC	^{13}C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAL2-04-1_01_BPC	2,3,7,8-TCDF 1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects)	P
SSAL2-04-4_01_BPC**	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SSAL2-05-3_01_BPC	2,3,7,8-TCDF 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J140638	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

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

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

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

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

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

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524B21
SDG #: G0J140638
Laboratory: Test America

Date: 12/16/10
Page: 1 of 1
Reviewer: EJ
2nd Reviewer: AV

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: 10/12/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	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	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

1	SSAL2-04-1_01_BPC	11	028845/	21		31	
2	SSAL2-04-2_01_BPC	12		22		32	
3	SSAL2-04-3_01_BPC	13		23		33	
4	SSAL2-04-4_01_BPC**	14		24		34	
5	SSAL2-05-1_01_BPC	15		25		35	
6	SSAL2-05-2_01_BPC	16		26		36	
7	SSAL2-05-3_01_BPC	17		27		37	
8	SSAL2-05-4_01_BPC	18		28		38	
9	SSAL2-04-3_01_BPCMS	19		29		39	
10	SSAL2-04-3_01_BPCMSD	20		30		40	

Notes:

LDC #: 24524B21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Reviewer: EO
2nd Reviewer: EO

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524B2 1
SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Reviewer:
2nd Reviewer:

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524B21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer:
2nd Reviewer:

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

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

Y N N/A Were all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

(bl)

Blank extraction date: 10/16/10 **Blank analysis date:** 10/25/10 **Associated samples:** All

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	025845	MB 5X	3	6						
E	0.060	0.30	0.21/4	0.13/4						
F	0.12	0.60	-	-						
G	0.53	2.65	1.8/4	0.85/4						
L	0.026	0.13								
O	0.19	0.95								
P	0.17	0.85								
Q	0.12	0.60								

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 #: 24524B21

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

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 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.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		9 + 1D	P	143 (79-139)	141 (79-139)	( )	3	no qual Les in
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24524B21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: [illegible]

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 were within the 40-135% criteria?
Y (N) N/A Was the S/N ratio all internal standard peaks ≥ 10?

(L)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		2	I	35 (40-135)	J | UJ | P anAL G, Q
				( )	
				( )	
		3	I	24 ( )	G, Q
			G	39 ( )	Q, P
				( )	
		4	I	33 ( )	G, Q
				( )	
		5	I	36 ( )	↓
				( )	
		6	I	30 ( )	G, Q
			G	39 ( )	Q, P
				( )	
		7	I	29 ( )	↓
			G	39 ( )	↓
				( )	
		8	I	28 (↓)	↓ G, Q
				( )	
				( )	
				( )	

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

LDC #: 24524B21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: [illegible]

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		9	H	39 (40-135)	NO qual MS
			I	23 (↓)	
			G	35 (↓)	
				( )	
		10	H	36 (40-135)	NO qual MSD
			I	19 (↑)	
			G	31 (↓)	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 24524B27

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

#	Date	~~Sample ID~~ compd	Finding	Associated Samples	Qualifications
			All compounds reported below PQL	All	J/A detects (sp)
			All compounds reported as EMPC	All	JK detects (k)
		H, O, Q	x'd cal range	1	J/Pdet (e)
		O, Q	↓	4	↓ (e)
		H, K, O, P, Q	↓	7	↓ (e)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524B4/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: F7
2nd Reviewer: AL

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (CS3 std)	Recalculated RRF (CS3 std)	Reported %RSD	Recalculated %RSD
1	ICAL	7/26/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.056	1.052	1.02	1.02	3.32	3.32
	DB225		~~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)						
2	ICAL	7/21/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.995	0.995	0.9849	0.9849	3.68	3.68
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.983	0.983	0.9681	0.9681	3.24	3.24
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.163	1.163	1.1014	1.1014	5.17	5.17
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.072	1.072	1.0735	1.0735	2.61	2.61
			OCDF (^{13}C-OCDD)	1.370	1.370	1.3500	1.3500	1.98	1.98
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.

LDC #: 24524B21
SDG #: all cons

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 5:18	10/29/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.056	1.12	1.12	6.1	6.1
	DB225		~~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)					
	CCV 19:03	10/28/10	~~2,3,7,8 TCDF~~ 1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.056	1.12	1.117	5.8	5.8
	DB225		~~OCDF (^{13}C-OCDD)~~					
2	CCV 8:24	10/26/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.995	0.94	0.94	5.6	5.6
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.983	0.94	0.94	4.1	4.1
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.163	1.22	1.22	4.8	4.8
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.072	1.08	1.08	0.8	0.8
			OCDF (^{13}C-OCDD)	1.370	1.39	1.39	1.4	1.4
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.

LDC #: 24524P21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

Page: 1 of 1
Reviewer: EJ
2nd Reviewer: [signature]

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: 9 + 10

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/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	21.1	21.2	0.093	23.9	22.9	113	113	108	108	4.6	4.6
1,2,3,7,8-PeCDD	105	106	0.15	120	125	114	114	118	118	3.8	3.8
1,2,3,4,7,8-HxCDD	↓	↓	0.15	105	110	100	100	104	104	4.7	4.7
1,2,3,4,7,8,9-HpCDF	↓	↓	7.2	158	157	143	143	141	141	0.84	0.84
OCDF	211	212	42	271	284	109	109	115	115	4.7	4.7

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

LDC #: 24327104/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 2
Reviewer: EZ
2nd Reviewer: AL

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 028851 - LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	22.0	NA	110	110				
1,2,3,7,8-PeCDD	100		118		118	118				
1,2,3,4,7,8-HxCDD	100		111		111	111				
1,2,3,4,7,8,9-HpCDF	100		123		123	123				
OCDF	200		224		112	112	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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = Internal/recovery standard

LDC #: 24524B27
SDG #: pc cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer:
2nd reviewer:

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

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

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. #4 . 2,3,7,8-TCDD

Conc. = $\frac{(222124)(2000)(\quad)(\quad)}{(75554600)(0.99)(10)(0.97)}$

= 6.12 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#4 2,3,7,8-TCDF			
		$\frac{27880300\ (2000)}{30758800\ (1.056)}$	$(10.0)(0.97)$	= 177 pg/g	

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	October 13, 2010
LDC Report Date:	December 20, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J150566

Sample Identification

SSAP3-05-1_01_BPC
SSAP3-05-2_01_BPC
SSAP3-05-3_01_BPC
SSAP3-05-4_01_BPC
SSAP3-05-5_01_BPC**
SSAP3-05-2_01_BPCMS
SSAP3-05-2_01_BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

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

V. Blanks

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

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

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

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAP3-05-1_01_BPC	^{13}C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAP3-05-2_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	30 (40-135) 21 (40-135) 25 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-05-3_01_BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	28 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-05-4_01_BPC	^{13}C-1,2,3,6,7,8-HxCDD ^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,6,7,8-HxCDF ^{13}C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 22 (40-135) 12 (40-135) 33 (40-135) 20 (40-135)	1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-05-5_01_BPC**	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAP3-05-5_01_BPC**	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J150566	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0J150566	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	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 G0J150566

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

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

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

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524C21
SDG #: G0J150566
Laboratory: Test America

Date: 12/16/10
Page: 1 of 1
Reviewer: FT
2nd Reviewer: KV

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: 10/13/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	SW	
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	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

1	SSAP3-05-1_01_BPC	11	0292315	21		31	
2	SSAP3-05-2_01_BPC	12		22		32	
3	SSAP3-05-3_01_BPC	13		23		33	
4	SSAP3-05-4_01_BPC	14		24		34	
5	SSAP3-05-5_01_BPC**	15		25		35	
6	SSAP3-05-2_01_BPCMS	16		26		36	
7	SSAP3-05-2_01_BPCMSD	17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

LDC #: 24524C21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: CF
2nd Reviewer: CF

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓	✓		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524C2 1
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer: [illegible]
2nd Reviewer: [illegible]

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524c2/

VALIDATION FINDINGS WORKSHEET
Routine Calibration

Reviewer: FT
2nd Reviewer: [illegible]

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

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

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

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

Y N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

(c)

#	Date	Standard ID	Compound	Finding %D (Limit: ≤30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	10/28/10	CCV (closing)	13C-OCDD	45.9 (≤35)		029235-MB, 6, 7	J/UJ/P (c) quant 9, 9
	11:22 22:24						

	PCDDs	Selected ions (m/z)	Ion Abundance Ratio		PCDFs	Selected ions (m/z)	Ion Abundance Ratio
	Tetra-	M/M+2	0.65-0.89		Tetra-	M/M+2	0.65-0.89
	Penta-	M+2/M+4	1.32-1.78		Penta-	M+2/M+4	1.32-1.78
	Hexa-	M+2/M+4	1.05-1.43		Hexa-	M+2/M+4	1.05-1.43
	Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59
	Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51
	Hepta-	M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	0.88-1.20
	Octa-	M+2/M+4	0.76-1.02		Octa-	M+2/M+4	0.76-1.02

LDC #: 24524c21

VALIDATION FINDINGS WORKSHEET
Blanks

Page: 1 of 1
Reviewer: FT
2nd Reviewer: CV

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was the method blank contaminated?

Blank extraction date: 10/19/10 **Blank analysis date:** 10/28/10 **Associated samples:** all

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	02923/5-MB 5X	2	3	4	5				
F	0.13	0.65	-	0.12/4	0.56/4	0.49/4				
G	0.45	2.25	1.2/4	1.8/4	-	-				

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 #: 24524C2/

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Reviewer: FT
2nd Reviewer: Q

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

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

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

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		6 + 7	E	72 (80-143)	( )	( )	2	no qual (es 1)
			H	78 (79-137)	( )	( )	↓	↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24524c2/

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
Y N N/A Are all internal standard recoveries were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

(L)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	I	26 (40-135)	J/UJ/P qual G, Q
				( )	
				( )	
		2	H	30 ( )	F
			I	21 ( )	G, Q
			G	25 ( )	O, P
				( )	
		3	I	28 ( )	G, Q
			G	39 ( )	O, P
				( )	
		4	F	35 ( )	O, P, E
			H	22 ( )	F
			I	12 ( )	G, Q
			E	33 ( )	K, L, M, N
			G	20 ( )	O, P
				( )	
		5	I	27 ( )	G, Q
			G	39 ( )	O, P
				( )	
				( )	

	Internal Standards	Check Standard Used
A	^{13}C-2,3,7,8-TCDF	
B	^{13}C-2,3,7,8-TCDD	
C	^{13}C-1,2,3,7,8-PeCDF	
D	^{13}C-1,2,3,7,8-PeCDD	
E	^{13}C-1,2,3,6,7,8-HxCDF	
F	^{13}C-1,2,3,6,7,8-HxCDD	
G	^{13}C-1,2,3,4,6,7,8-HpCDF	
H	^{13}C-1,2,3,4,6,7,8-HpCDD	
I	^{13}C-OCDD	

	Recovery Standards	Check Standard Used
K	^{13}C-1,2,3,4-TCDD	
L	^{13}C-1,2,3,7,8,9-HxCDD	
M		
N		
O		
P		
Q		
R		
T		

LDC #: 24524C2J

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: [illegible]

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		6	I	31 (40-135)	no qual MS
			G	34 (↓)	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 24524C21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

#	Date	~~Sample ID~~ compd	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 second column confirmation was performed	5	none/p (a)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524C4/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (cs3 std)	Recalculated RRF (cs3 std)	Reported %RSD	Recalculated %RSD
1	ICAL	9/14/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.984	1.05	1.05	11.8	11.8
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.1	12.1
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3
			OCDF (^{13}C-OCDD)	2.118	2.118	2.36	2.36	15.3	15.3
2	ICAL	10/27/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.01573	1.01573	1.02991	1.02991	3.51093	3.51093
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.10816	1.10816	1.16359	1.16359	4.89670	4.89670
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.15691	1.15691	1.23467	1.23467	4.42117	4.42117
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.04888	1.04888	1.16792	1.16792	8.83361	8.83361
			OCDF (^{13}C-OCDD)	1.50681	1.50681	1.65488	1.65488	8.83207	8.83207
3	ICAL	10/30/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.060	1.060	1.008	1.008	3.59	3.59
	PBMS		~~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.

LDC #: 24524c21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page: 1 of 1
Reviewer: EJ
2nd Reviewer:

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 14:22	10/28/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.91	0.91	7.2	7.2
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	0.91	0.91	11.6	11.6
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.21	1.21	5.6	5.6
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.15	1.15	1.1	1.1
			OCDF (^{13}C-OCDD)	2.118	1.92	1.92	9.4	9.4
2	CCV 22:00	11/3/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.01573	1.01097	1.01097	0.5	0.5
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.10816	1.15153	1.15153	3.9	3.9
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.15691	1.19924	1.19924	3.7	3.7
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.04888	1.09233	1.09233	4.1	4.1
			OCDF (^{13}C-OCDD)	1.50681	1.49523	1.49523	0.8	0.8
3	CCV 19:21	11/4/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.060	0.95	0.95	10.1	10.1
	DB225		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.

LDC #: 24324C41
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

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

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: 6 + 7

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/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	20.9	20.9	ND	17.6	19.3	84	84	92	92	9.1	9.1
1,2,3,7,8-PeCDD	104	104	ND	97.2	112	93	93	107	107	14	14
1,2,3,4,7,8-HxCDD	104	104	ND	84.3	88.9	81	81	85	85	5.3	5.3
1,2,3,4,7,8,9-HpCDF	104	104	73Z ND	132	133	126	126	127	127	0.82	0.82
OCDF	209	209	ND	211	202	101	101	97	97	4.4	4.4

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.

LDC #: 24307

SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: EZ
2nd Reviewer:

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0292315-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	18.0	NA	90	90				
1,2,3,7,8-PeCDD	100		99.2		99	99				
1,2,3,4,7,8-HxCDD	100		93.6		94	94				
1,2,3,4,7,8,9-HpCDF	100		107		107	107				
OCDF	200		178		89	89	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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 14524C21
SDG #: pe cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

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

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?

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. #4 , OCDF :

Conc. = $\frac{(13344.8)(4000)(\quad)}{(38958.6)(1.507)(10.19)(0.954)}$

=

13 pg/g

#	Sample ID	2,3,7,8 Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	#4 TCDF	= 1554719 (2000)			
		361446304 (1.056)(10.19)(0.954)			
			= 0.84 pg/g		

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	October 14, 2010
LDC Report Date:	December 20, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J170404

Sample Identification

SSAL4-04-2_01_BPC
SSAL4-04-3_01_BPC
SSAL4-04-4_01_BPC**

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

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

V. Blanks

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

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

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

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAL4-04-2_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 29 (40-135) 30 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAL4-04-4_01_BPC**	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,6,7,8-HxCDF ^{13}C-1,2,3,4,6,7,8-HpCDF	16 (40-135) 9.9 (40-135) 35 (40-135) 14 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAL4-04-4_01_BPC**	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J170404	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0J170404	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	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 G0J170404

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

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

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

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524D21
SDG #: G0J170404
Laboratory: Test America

Date: 12/16/10
Page: 1 of 1
Reviewer: EZ
2nd Reviewer: [illegible]

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: 10/14/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	SW	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	SSAP3-05-2_01_BPC MS/D
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

soil

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

Notes: _____

LDC #: 24524D21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: EP
2nd Reviewer: CP

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?		✓		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524D2
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer:
2nd Reviewer:

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524D21

VALIDATION FINDINGS WORKSHEET
Routine Calibration

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 Was a routine calibration was performed at the beginning and end of each 12 hour period?
Y N N/A Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled?
Y N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	10/28/10 22:24	ccv (closing)	13C- OCDD	45.9 (≤35)		All	J/UJ/P QUAL G, Q

	PCDDs	Selected ions (m/z)	Ion Abundance Ratio		PCDFs	Selected ions (m/z)	Ion Abundance Ratio
	Tetra-	M/M+2	0.65-0.89		Tetra-	M/M+2	0.65-0.89
	Penta-	M+2/M+4	1.32-1.78		Penta-	M+2/M+4	1.32-1.78
	Hexa-	M+2/M+4	1.05-1.43		Hexa-	M+2/M+4	1.05-1.43
	Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59
	Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51
	Hepta-	M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	0.88-1.20
	Octa-	M+2/M+4	0.76-1.02		Octa-	M+2/M+4	0.76-1.02

LDC #: 24524D21

VALIDATION FINDINGS WORKSHEET
Blanks

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 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?

Blank extraction date: 10/19/10 **Blank analysis date:** 10/28/10 **Associated samples:** All

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	0292315-MB		2	3						
F	0.13	0.65		-	0.56/4					
G	0.45	2.25		1.1/4	-					

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 #: 24524D21

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Reviewer: FT
2nd Reviewer: Q

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

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

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

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		SSAP3-05-2_01_BPC MS/D	E	72 (80-143)	( )	( )	none	no qual
			H	78 (79-137)	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24524D2 /

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: ✓

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	H	33 (40-135)	J/UJ/P qual F
			I	29 (↓)	↓ G, Q
			G	30 (↓)	↓ O, P
				( )	
				( )	
		3	H	16 (40-135)	J/UJ/P F
			I	9.9 (↓)	↓ G, Q
			E	35 (↓)	↓ K, L, M, N
			G	14 (↓)	↓ O, P
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

	Internal Standards	Check Standard Used
A	^{13}C-2,3,7,8-TCDF	
B	^{13}C-2,3,7,8-TCDD	
C	^{13}C-1,2,3,7,8-PeCDF	
D	^{13}C-1,2,3,7,8-PeCDD	
E	^{13}C-1,2,3,6,7,8-HxCDF	
F	^{13}C-1,2,3,6,7,8-HxCDD	
G	^{13}C-1,2,3,4,6,7,8-HpCDF	
H	^{13}C-1,2,3,4,6,7,8-HpCDD	
I	^{13}C-OCDD	

	Recovery Standards	Check Standard Used
K	^{13}C-1,2,3,4-TCDD	
L	^{13}C-1,2,3,7,8,9-HxCDD	
M		
N		
O		
P		
Q		
R		
T		

LDC #: 24524D2 /

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

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

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

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

#	Date	~~Sample ID~~ compd	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	3	none / P (a)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524D2J
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Reviewer: F7
2nd Reviewer: [illegible]

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (CS3 std)	Recalculated RRF (CS3 std)	Reported %RSD	Recalculated %RSD
1	~~CCV 14:22~~	~~10/28/10~~	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.984	1.05	1.05	11.8	11.8
	LCAL	9/14/10	2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.1	12.1
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3
			OCDF (^{13}C-OCDD)	2.118	2.118	2.36	2.36	15.3	15.3
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.

LDC #: 24524D2/
SDG #: all cons

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page: 1 of 1
Reviewer: FT
2nd Reviewer: CF

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 14:22	10/28/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.91	0.91	7.2	7.2
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	0.91	0.91	11.6	11.6
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.21	1.21	5.6	5.6
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.15	1.15	1.1	1.1
			OCDF (^{13}C-OCDD)	2.118	1.92	1.92	9.4	9.4
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.

LDC #: 02924B2/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: EZ
2nd Reviewer: AV

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 029235-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	18.0	NA	90	90				
1,2,3,7,8-PeCDD	100		99.2		99	99				
1,2,3,4,7,8-HxCDD	100		93.6		94	94				
1,2,3,4,7,8,9-HpCDF	100		107		107	107				
OCDF	200		178		89	89	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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_5{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24534D2/
SDG #: pc cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer:
2nd reviewer:

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

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

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

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

Conc. = $\frac{(364868)(2000)(\quad)}{(268591000)(0.98)(10.02)(0.923)}$

= 0.30 pg/g

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

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	September 27, 2010
LDC Report Date:	December 23, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J200489

Sample Identification

SSAN6-08-1BPC

Introduction

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

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

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

Raw data were not reviewed for this SDG. The review was based on QC data.

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

III. Initial Calibration

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0298269-MB	10/25/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.12 pg/g 0.32 pg/g 0.088 pg/g 0.13 pg/g	All samples in SDG G0J200489

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

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

X. Target Compound Identifications

Raw data were not reviewed for this SDG.

XI. Project Quantitation Limit

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

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

All compounds reported below the PQL were qualified as follows:

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

All compounds reported as EMPC were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J200489	All compounds reported as estimated maximum possible concentration (EMPC).	JK (all detects)	A

Raw data were not reviewed for this SDG.

XII. System Performance

Raw data were not reviewed for this SDG.

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

No field duplicates were identified in this SDG.

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

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B

LDC #: 24524E21
SDG #: G0J200489
Laboratory: Test America

Date: 12/17/10
Page: 1 of 1
Reviewer: R
2nd Reviewer: R

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: 9/27/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	SW	SSAP3-03-9-01-BPMS/D (no Ass. Sample)
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	SW	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples:
SOIL

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

Notes:

VALIDATION FINDINGS WORKSHEET

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

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	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 #: 24524E21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: ET
2nd Reviewer: 9

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was the method blank contaminated?

Blank extraction date: 10/25/10 **Blank analysis date:** 10/28/10 **Associated samples:** All 75%

Conc. units: pg/g

Compound	Blank ID	5X	Sample Identification							
	0298269-MB									
F	0.12	0.6								
G	0.32	1.6								
O	0.088	0.44								
Q	0.13	0.65								

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 #: 24524E21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: [illegible]

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	H	38 (40-135)	J/UJ/P QUAL F (1)
			I	22 (↓)	↓ 9, Q
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 24524E21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

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

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

#	Date	compd ~~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)
		F, G, H, I, J, K, L, M, N, O, P, Q	x'd cal Range	1	J/Pdet (c)

Comments: See sample calculation verification worksheet for recalculations

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	October 22, 2010
LDC Report Date:	December 19, 2010
Matrix:	Soil/Water
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J230497

Sample Identification

SSAP3-03-1_01_BPC
SSAP3-03-5_01_BPC
SSAP3-03-9_01_BPC
SSAP3-04-1_01_BPC
SSAP3-04-1_01_BPC_FD
SSAP3-04-5_01_BPC
SSAP3-04-9_01_BPC
SA47-1_01_BPC
SA47-2_01_BPC
SA47-3_01_BPC
SA47-4_01_BPC
SA47-4_01_BPC_FD
SA47-5_01_BPC
SA47-6_01_BPC
SA47-7_01_BPC
SA47-8_01_BPC**
EB-10222010-RZC
SSAP3-03-9_01_BPCMS
SSAP3-03-9_01_BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

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

V. Blanks

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

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

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

Sample	Compound	Reported Concentration	Modified Final Concentration
EB-10222010-RZC	OCDD 1,2,3,4,6,7,8-HpCDF	8.1 pg/L 3.3 pg/L	8.1U pg/L 3.3U pg/L
SSAP3-03-1_01_BPC	1,2,3,4,6,7,8-HpCDD	0.59 pg/g	0.59U pg/g
SSAP3-03-9_01_BPC	1,2,3,4,6,7,8-HpCDF	0.39 pg/g	0.39U pg/g
SSAP3-04-5_01_BPC	1,2,3,4,6,7,8-HpCDF OCDF	0.17 pg/g 0.29 pg/g	0.17U pg/g 0.29U pg/g
SSAP3-04-9_01_BPC	OCDD	0.32 pg/g	0.32U pg/g
SA47-2_01_BPC	OCDD	0.83 pg/g	0.83U pg/g
SA47-4_01_BPC	1,2,3,4,6,7,8-HpCDD OCDD	0.24 pg/g 0.79 pg/g	0.24U pg/g 0.79U pg/g
SA47-4_01_BPC_FD	OCDD	1.3 pg/g	1.3U pg/g
SA47-5_01_BPC	OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.25 pg/g 0.27 pg/g 0.48 pg/g	0.25U pg/g 0.27U pg/g 0.48U pg/g
SA47-6_01_BPC	1,2,3,4,6,7,8-HpCDD	0.32 pg/g	0.32U pg/g

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

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

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

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

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAP3-03-1_01_BPC	^{13}C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAP3-03-5_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	34 (40-135) 18 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-03-9_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	32 (40-135) 20 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-04-1_01_BPC	^{13}C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAP3-04-1_01_BPC_FD	^{13}C-1,2,3,7,8-PeCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	36 (40-135) 27 (40-135) 37 (40-135)	1,2,3,7,8-PeCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SSAP3-04-9_01_BPC	^{13}C-OCDD	28 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA47-1_01_BPC	^{13}C-OCDD	32 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA47-2_01_BPC	^{13}C-OCDD	26 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA47-3_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 14 (40-135) 31 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SA47-4_01_BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	27 (40-135) 36 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

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

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J230497	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0J230497	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

Samples SSAP3-04-1_01_BPC and SSAP3-04-1_01_BPC_FD and samples SA47-4_01_BPC and SA47-4_01_BPC_FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

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

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

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SA47-4_01_BPC	SA47-4_01_BPC_FD				
1,2,3,6,7,8-HxCDD	2.6U	0.15	-	2.45 (≤2.6)	-	-
1,2,3,7,8,9-HxCDD	2.6U	0.27	-	2.33 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDD	0.24	2.4	-	2.16 (≤2.6)	-	-
OCDD	0.79	1.3	-	0.51 (≤5.2)	-	-
2,3,7,8-TCDF	0.51	0.69	-	0.18 (≤0.52)	-	-
1,2,3,7,8-PeCDF	0.41	0.55	-	0.14 (≤2.6)	-	-
2,3,4,7,8-PeCDF	0.26	0.21	-	0.05 (≤2.6)	-	-
1,2,3,4,7,8-HxCDF	0.71	0.63	-	0.08 (≤2.6)	-	-
1,2,3,6,7,8-HxCDF	0.37	0.29	-	0.08 (≤2.6)	-	-
2,3,4,6,7,8-HxCDF	0.22	0.096	-	0.124 (≤2.6)	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	SA47-4_01_BPC	SA47-4_01_BPC_FD				
1,2,3,7,8,9-HxCDF	2.6U	0.15	-	2.45 (≤2.6)	-	-
1,2,3,4,6,7,8-HpCDF	1.4	1.2	-	0.2 (≤2.6)	-	-
1,2,3,4,7,8,9-HpCDF	0.62	0.45	-	0.17 (≤2.6)	-	-
OCDF	3.9	2.8	-	1.1 (≤5.2)	-	-

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

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

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J230497	SSAP3-03-1_01_BPC SSAP3-03-5_01_BPC SSAP3-03-9_01_BPC SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD SSAP3-04-5_01_BPC SSAP3-04-9_01_BPC SA47-1_01_BPC SA47-2_01_BPC SA47-3_01_BPC SA47-4_01_BPC SA47-4_01_BPC_FD SA47-5_01_BPC SA47-6_01_BPC SA47-7_01_BPC SA47-8_01_BPC** EB-10222010-RZC	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0J230497	SSAP3-04-1_01_BPC SSAP3-04-1_01_BPC_FD	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Field duplicates (Difference) (fd)

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

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

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

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524F21
SDG #: G0J230497
Laboratory: Test America

Date: 12/17/10
Page: 1 of 1
Reviewer: [illegible]
2nd Reviewer: [illegible]

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: 10/22/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	SW	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	SW	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 4, 5 11, 12
XV.	Field blanks	SW	EB = 17

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SO/L

1	SSAP3-03-1_01_BPC	11	SA47-4_01_BPC	21	1 02982G9	31	
2	SSAP3-03-5_01_BPC	12	SA47-4_01_BPC_FD	22	2 0298247	32	
3	SSAP3-03-9_01_BPC	13	SA47-5_01_BPC	23		33	
4	SSAP3-04-1_01_BPC	14	SA47-6_01_BPC	24		34	
5	SSAP3-04-1_01_BPC_FD	15	SA47-7_01_BPC	25		35	
6	SSAP3-04-5_01_BPC	16	SA47-8_01_BPC**	26		36	
7	SSAP3-04-9_01_BPC	17	EB-10222010-RZC W	27		37	
8	SA47-1_01_BPC	18	SSAP3-03-9_01_BPCMS	28		38	
9	SA47-2_01_BPC	19	SSAP3-03-9_01_BPCMSD	29		39	
10	SA47-3_01_BPC	20		30		40	

Notes: _____

LDC #: 24524 F21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

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

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?		✓		
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524F21
SDG #: all covers

VALIDATION FINDINGS CHECKLIST

Reviewer: A
2nd Reviewer: Q

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?		✓		
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524F21

VALIDATION FINDINGS WORKSHEET
Routine Calibration

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

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

Y N N/A Was a routine calibration was performed at the beginning and end of each 12 hour period?
Y N N/A Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled?
Y N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

(a) (c)

#	Date	Standard ID	Compound	Finding %D (Limit: ≤30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	10/28/10	CCV (closing)	^{13}C-OCDD	45.9 (≤35)		0298269-MB	J/UJ/P qual G, Q

	PCDDs	Selected ions (m/z)	Ion Abundance Ratio		PCDFs	Selected ions (m/z)	Ion Abundance Ratio
	Tetra-	M/M+2	0.65-0.89		Tetra-	M/M+2	0.65-0.89
	Penta-	M+2/M+4	1.32-1.78		Penta-	M+2/M+4	1.32-1.78
	Hexa-	M+2/M+4	1.05-1.43		Hexa-	M+2/M+4	1.05-1.43
	Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59
	Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51
	Hepta-	M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	0.88-1.20
	Octa-	M+2/M+4	0.76-1.02		Octa-	M+2/M+4	0.76-1.02

LDC #: 24524F2/

VALIDATION FINDINGS WORKSHEET
Blanks

Page: 1 of 2
Reviewer: FT
2nd Reviewer: 9

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was the method blank contaminated?

(bl)

Blank extraction date: 10/25/10 **Blank analysis date:** 10/29/10 **Associated samples:** All water

Conc. units: pg/L

Compound	Blank ID	Sample Identification								
	029824	7-MB	17							
G	12		8.1/U							
O	2.7		3.3/U							

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 #: 24524F21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: FT
2nd Reviewer:

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

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was the method blank contaminated?

(bl)

Blank extraction date: 10/25/10 **Blank analysis date:** 10/28/10 **Associated samples:** All soils

Conc. units: pg/g

Compound	Blank ID	SX	Sample Identification							
	0298269	MB	1	3	6	7	9	11	12	13
F	0.12	0.60	0.59/U					0.24/U		
G	0.32	1.6	-			0.32/U	0.83/U	0.79/U	1.3/U	0.25/U
O	0.088	0.44	-	0.39/U	0.17/U					0.27/U
Q	0.13	0.65	-		0.29/U					0.48/U

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 #: 24524f2/

VALIDATION FINDINGS WORKSHEET
Blanks

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 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?

(bl)

Blank extraction date: 10/25/10 **Blank analysis date:** 10/28/10 **Associated samples:** All soils

Conc. units: pg/g

Compound	Blank ID	5X	Sample Identification							
	029826	-MB	14	15	16					
F	0.12	0.60	0.32/U	-						
G	0.32	1.6	-	0.40/U						
O	0.088	0.44			0.22/U					
Q	0.13	0.65								

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 #: 24524F21

VALIDATION FINDINGS WORKSHEET
Field Blanks

Reviewer: FT
2nd Reviewer: a

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

(Y) N N/A Were field blanks identified in this SDG?
Blank units: pg/L **Associated sample units:** pg/g
Sampling date: 10/22/10
Field blank type: (circle one) Field Blank / Rinsate / Other: EB Associated Samples: All soils 75%

Compound	Blank ID	Sample Identification								
	17									
G	8.1									
O	3.3									
Q	10									
CRQL										

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

LDC #: 24524F27

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Reviewer: FT
2nd Reviewer: Q

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

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

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

(Y) N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		18 + 19	E	68 (80-143)	( )	33 (31)	3	no qual res in
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24524F21

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: FT
2nd Reviewer: [illegible]

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

Y N N/A Was a LCS required?
Y N N/A Was a LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?
Y N N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

(1)

#	Date	Lab ID/Reference	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		0298247-LCS	L	134 (76-133)	( )	( )	All water	J+/P no LCSD, MS/D
			P	131 (83-130)	( )	( )	↓	↓ ↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24524F21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
Y N N/A Are all internal standard recoveries were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	I	28 (40-135)	J/UJ/P qual G, Q
		2	H	34	F
			I	18	G, Q
			G	31	O, P
		3	H	32	
			I	20	
			G	34	
		4	I	35	G, Q
		5	D	36	B
			I	27	G, Q
			G	37	O, P
		7	I	28	G, Q
		8	I	32	G, Q

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

LDC #: 24574F2/

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: 9

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

(1)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		9	I	26 (40-135)	J/UJ/P qual G, Q
				( )	
		10	H	35 ( )	F
			I	14 ( )	G, Q
			G	37 ( )	O, P
				( )	
		11	I	27 ( )	G, Q
			G	36 ( )	O, P
				( )	
		12	H	37 ( )	F
			I	23 ( )	G, Q
			G	33 ( )	O, P
				( )	
		14	H	37 ( )	F
			I	17 ( )	G, Q
			G	39 ( )	O, P
				( )	
		15	H	37 ( )	F
			I	18 ( )	G, Q
			G	39 (✓)	O, P

	Internal Standards	Check Standard Used
A	^{13}C-2,3,7,8-TCDF	
B	^{13}C-2,3,7,8-TCDD	
C	^{13}C-1,2,3,7,8-PeCDF	
D	^{13}C-1,2,3,7,8-PeCDD	
E	^{13}C-1,2,3,6,7,8-HxCDF	
F	^{13}C-1,2,3,6,7,8-HxCDD	
G	^{13}C-1,2,3,4,6,7,8-HpCDF	
H	^{13}C-1,2,3,4,6,7,8-HpCDD	
I	^{13}C-OCDD	

	Recovery Standards	Check Standard Used
K	^{13}C-1,2,3,4-TCDD	
L	^{13}C-1,2,3,7,8,9-HxCDD	
M		
N		
O		
P		
Q		
R		
T		

LDC #: 24524F21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)
Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".
Y N N/A Are all internal standard recoveries were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		16	H	34 (40 - 135)	J | NJ | P qual: F
			I	16 (↓)	G, Q
			G	36 (↓)	↓ B, P
				( )	
				( )	
		18	I	31 (↓)	no qual MS
				( )	
		19	I	31 (↓)	no qual MSD
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

	Internal Standards	Check Standard Used
A	^{13}C-2,3,7,8-TCDF	
B	^{13}C-2,3,7,8-TCDD	
C	^{13}C-1,2,3,7,8-PeCDF	
D	^{13}C-1,2,3,7,8-PeCDD	
E	^{13}C-1,2,3,6,7,8-HxCDF	
F	^{13}C-1,2,3,6,7,8-HxCDD	
G	^{13}C-1,2,3,4,6,7,8-HpCDF	
H	^{13}C-1,2,3,4,6,7,8-HpCDD	
I	^{13}C-OCDD	

	Recovery Standards	Check Standard Used
K	^{13}C-1,2,3,4-TCDD	
L	^{13}C-1,2,3,7,8,9-HxCDD	
M		
N		
O		
P		
Q		
R		
T		

LDC #: 24524F21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

Page: 1 of 1
Reviewer: FT
2nd Reviewer: 9

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

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

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

#	Date	~~Sample ID~~ compd	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	2,3,6,7,9,13,15,16	J/P ~~de~~ mp (o)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24524F21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 2
Reviewer: FT
2nd Reviewer:

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

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

(fd)

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	4	5				
A	0.52U	0.16		0.36	≤0.52	
B	2.6U	0.36		2.24	≤2.6	
C	2.6U	0.26		2.34	≤2.6	
D	0.30	0.36		0.06	≤2.6	
E	0.37	0.70		0.33	≤2.6	
F	0.77	2.1		1.33	≤2.6	
G	3.6	5.0		1.4	≤5.2	
H	1.8	3.3		1.5	≤0.52	
I	1.5	3.5		2	≤2.6	
J	0.77	2.0		1.23	≤2.6	
K	2.3	7.3		5	≤2.6	J/A det
L	1.8	5.1		3.3	≤2.6	↓
M	0.51	1.3		0.79	≤2.6	
N	0.37	0.99		0.62	≤2.6	
O	6.7	25		18.3	≤2.6	J/A det
P	3.1	12		8.9	≤2.6	↓
Q	24	130		106	≤5.2	↓

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LDC#: 23906B4

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Reviewer: F7
2nd Reviewer: N

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

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

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	11	12				
D	2.6U	0.15		2.45	≤2.6	
E	2.6U	0.27		2.33	≤2.6	
F	0.24	2.4		2.16	≤2.6	
G	0.79	1.3		0.51	≤5.2	
H	0.51	0.69		0.18	≤0.52	
I	0.41	0.55		0.14	≤2.6	
J	0.26	0.21		0.05	≤2.6	
K	0.71	0.63		0.08	≤2.6	
L	0.37	0.29		0.08	≤2.6	
M	0.22	0.096		0.124	≤2.6	
N	2.6U	0.15		2.45	≤2.6	
O	1.4	1.2		0.2	≤2.6	
P	0.62	0.45		0.17	≤2.6	
Q	3.9	2.8		1.1	≤5.2	

LDC #: 24524-21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer: F7
2nd Reviewer: 8

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (CS3 std)	Recalculated RRF (CS3 std)	Reported %RSD	Recalculated %RSD
1	ICAL	10/28/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	~~1.275~~ 1.120	1.120	1.1574	1.1574	~~12.4~~ 4.94	4.94
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.053	1.053	1.1240	1.124	4.20	4.20
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.180	1.180	1.2326	1.2326	2.74	2.74
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.104	1.104	1.1552	1.1552	3.85	3.85
			OCDF (^{13}C-OCDD)	1.681	1.681	1.7419	1.7419	5.94	5.94
2	ICAL	9/14/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.984	1.05	1.05	11.8	11.8
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	1.032	1.06	1.06	10.8	10.8
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.141	1.25	1.25	12.7	12.7
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.134	1.26	1.26	12.3	12.3
			OCDF (^{13}C-OCDD)	2.118	2.118	2.36	2.36	15.3	15.3
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.

LDC #:_____
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page:___of___
Reviewer: EJ
2nd Reviewer: 9

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 7:11	9/10/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.120	1.06	1.06	5.0	5.0
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.053	1.17	1.17	10.8	10.8
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.180	1.27	1.27	7.3	7.3
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.104	1.14	1.14	3.5	3.5
			OCDF (^{13}C-OCDD)	1.681	1.98	1.98	17.8	17.8
2	CCV 14:22	10/28/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	0.91	0.91	7.2	7.2
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	0.91	0.91	11.6	11.6
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.21	1.21	5.6	5.6
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.15	1.15	1.1	1.1
			OCDF (^{13}C-OCDD)	2.118	1.92	1.92	9.4	9.4
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.

LDC #:_____
SDG #: per cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

Page:___of___
Reviewer: FT
2nd Reviewer: [signature]

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: 18 + 19

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/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	22.0	22.0	ND	20.6	21.1	94	94	96	96	2.6	2.6
1,2,3,7,8-PeCDD	110	110	ND	107	110	97	97	100	100	3.0	3.0
1,2,3,4,7,8-HxCDD	110	110	ND	94.3	110	86	86	100	100	16	16
1,2,3,4,7,8,9-HpCDF	110	110	ND	132	145	120	120	132	132	8.9	8.9
OCDF	220	220	ND	246	269	112	112	122	122	8.7	8.7

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

LDC #:_____
SDG #: see copy

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: EZ
2nd Reviewer: [illegible]

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 0298269-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	21.3	NA	107	107				
1,2,3,7,8-PeCDD	100		99.0		99	99				
1,2,3,4,7,8-HxCDD	100		89.3		89	89				
1,2,3,4,7,8,9-HpCDF	100		85.9		86	86				
OCDF	200		222		111	111	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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = Internal/recovery standard

LDC #:_____
SDG #: pll cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer: FF
2nd reviewer: [illegible]

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?

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. #16 . 2,3,7,8-TCDF

Conc. = $\frac{(2250223)(2000)(\quad)}{(11248040)(10.32)(1.12)(0.929)}$

= 0.37 pg/g

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

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	October 26, 2010
LDC Report Date:	December 19, 2010
Matrix:	Soil/Water
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0J270514

Sample Identification

SA183-1-01-BPC
SA183-2-01-BPC
SA183-3-01-BPC
SA183-3-01-BPC-FD
SA183-4-01-BPC
SA183-5-01-BPC
SA183-6-01-BPC
SA183-7-01-BPC
SA183-8-01-BPC**
RSAO4-1-01-BPC
RSAO4-2-01-BPC
RSAO4-3-01-BPC
RSAO4-4-01-BPC
RSAO4-5-01-BPC
RSAO4-6-01-BPC
RSAO4-7-01-BPC
RSAO4-8-01-BPC**
RSAO4-8-01-BPC-FD
SSAN6-05-1-01-BPC
SSAN6-05-2-01-BPC
SSAN6-05-3-01-BPC
SSAN6-05-4-01-BPC
EB-10262010-RZC
SA183-1-01-BPCMS
SA183-1-01-BPCMSD
SSAN6-05-3-01-BPCMS
SSAN6-05-3-01-BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

Date	Compound	%D	Associated Samples	Affected Compound	Flag	A or P
11/9/10	^{13}C-1,2,3,4,7,8-HxCDF	41	EB-10262010-RZC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC SSAN6-05-3-01-BPCMS SSAN6-05-3-01-BPCMSD 0301424-MB 0301440MB	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) UJ (all non-detects)	P

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0301420-MB	10/28/10	OCDD 1,2,3,4,6,7,8-HpCDF	0.27 pg/g 0.060 pg/g	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC-FD SA183-4-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSAO4-1-01-BPC RSAO4-2-01-BPC RSAO4-3-01-BPC RSAO4-4-01-BPC RSAO4-5-01-BPC RSAO4-6-01-BPC RSAO4-7-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD SSAN6-05-1-01-BPC SSAN6-05-2-01-BPC

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

Sample	Compound	Reported Concentration	Modified Final Concentration
SA183-2-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	0.87 pg/g 0.099 pg/g	0.87U pg/g 0.099U pg/g
SA183-3-01-BPC	OCDD	1.0 pg/g	1.0U pg/g
SA183-4-01-BPC	OCDD 1,2,3,4,6,7,8-HpCDF	0.46 pg/g 0.12 pg/g	0.46U pg/g 0.12U pg/g

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

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

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

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

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SA183-1-01-BPC	^{13}C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-2-01-BPC	^{13}C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-3-01-BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	33 (40-135) 39 (40-135)	OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SA183-3-01-BPC-FD	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	35 (40-135) 19 (40-135) 32 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SA183-4-01-BPC	^{13}C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-5-01-BPC	^{13}C-OCDD	23 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-6-01-BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 25 (40-135) 34 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P
SA183-7-01-BPC	^{13}C-OCDD	27 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SA183-8-01-BPC**	^{13}C-OCDD	30 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
RSA04-2-01-BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	38 (40-135) 25 (40-135) 35 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-05-4-01-BPC	^{13}C-OCDD	38 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN6-05-1-01-BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAN6-05-3-01-BPC	1,2,3,4,6,7,8-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects)	P
SA183-8-01-BPC** RSAO4-4-01-BPC RSAO4-6-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD EB-10262010-RZC	2,3,7,8-TCDF	2nd column confirmation was not performed for this compound.	This compound must be confirmed on the 2nd column per the method.	None	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0J270514	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0J270514	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A

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

XII. System Performance

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

XIII. Overall Assessment of Data

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

XIV. Field Duplicates

Samples SA183-3-01-BPC and SA183-3-01-BPC-FD and samples RSAO4-8-01-BPC** and RSAO4-8-01-BPC-FD were identified as field duplicates. No polychlorinated dioxins/dibenzofurans were detected in any of the samples with the following exceptions:

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

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

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

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

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

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0J270514	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC-FD SA183-4-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSAO4-1-01-BPC RSAO4-2-01-BPC RSAO4-3-01-BPC RSAO4-4-01-BPC RSAO4-5-01-BPC RSAO4-6-01-BPC RSAO4-7-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD SSAN6-05-1-01-BPC SSAN6-05-2-01-BPC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC EB-10262010-RZC	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0J270514	SA183-1-01-BPC SA183-2-01-BPC SA183-3-01-BPC SA183-3-01-BPC-FD SA183-4-01-BPC SA183-5-01-BPC SA183-6-01-BPC SA183-7-01-BPC SA183-8-01-BPC** RSAO4-1-01-BPC RSAO4-2-01-BPC RSAO4-3-01-BPC RSAO4-4-01-BPC RSAO4-5-01-BPC RSAO4-6-01-BPC RSAO4-7-01-BPC RSAO4-8-01-BPC** RSAO4-8-01-BPC-FD SSAN6-05-1-01-BPC SSAN6-05-2-01-BPC SSAN6-05-3-01-BPC SSAN6-05-4-01-BPC EB-10262010-RZC	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 G0J270514

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

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

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524G21
SDG #: G0J270514
Laboratory: Test America

Date: 12/17/10
Page: 1 of 1
Reviewer:
2nd Reviewer:

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

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 10/26/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/~~ICV~~	SW	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	SW	
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	SW	D = 3, 4 17 + 18
XV.	Field blanks	SW	EB = 23

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

Soil + water

1	SA183-1-01-BPC	11	RSAO4-2-01-BPC	21	SSAN6-05-3-01-BPC	31	030142U
2	SA183-2-01-BPC	12	RSAO4-3-01-BPC	22	SSAN6-05-4-01-BPC	32	030142U
3	SA183-3-01-BPC	13	RSAO4-4-01-BPC	23	EB-10262010-RZC W	33	030144O
4	SA183-3-01-BPC-FD	14	RSAO4-5-01-BPC	24	SA183-1-01-BPCMS	34	
5	SA183-4-01-BPC	15	RSAO4-6-01-BPC	25	SA183-1-01-BPCMSD	35	
6	SA183-5-01-BPC	16	RSAO4-7-01-BPC	26	SSAN6-05-3-01-BPCMS	36	
7	SA183-6-01-BPC	17	RSAO4-8-01-BPC**	27	SSAN6-05-3-01-BPCMSD	37	
8	SA183-7-01-BPC	18	RSAO4-8-01-BPC-FD	28		38	
9	SA183-8-01-BPC**	19	SSAN6-05-1-01-BPC	29		39	
10	RSAO4-1-01-BPC	20	SSAN6-05-2-01-BPC	30		40	

Notes:

LDC #: 24524G21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Reviewer: EV
2nd Reviewer: [illegible]

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	not	✓		
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?	✓			

LDC #: 24524G21
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer: [illegible]
2nd Reviewer: [illegible]

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524G21

VALIDATION FINDINGS WORKSHEET
Routine Calibration

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

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

Y N N/A Was a routine calibration was performed at the beginning and end of each 12 hour period?
Y N N/A Were all percent differences (%D) of RRFs ≤ 20% for unlabeled compounds and ≤ 30% for labeled?
Y N N/A Did all routine calibration standards meet the Ion Abundance Ratio criteria?

(c)

#	Date	Standard ID	Compound	Finding %D (Limit: ≤30.0%)	Finding Ion Abundance Ratio	Associated Samples	Qualifications
	11/9/10 04:56	CCV (closing)	^{13}C-1,2,3,4,7,8-HxCDF	41 (≤35)		030424-MB, 21, 22, 26, 27 + all water (030440)	J/UJ/P, all K, L, M, N

	PCDDs	Selected ions (m/z)	Ion Abundance Ratio		PCDFs	Selected ions (m/z)	Ion Abundance Ratio
	Tetra-	M/M+2	0.65-0.89		Tetra-	M/M+2	0.65-0.89
	Penta-	M+2/M+4	1.32-1.78		Penta-	M+2/M+4	1.32-1.78
	Hexa-	M+2/M+4	1.05-1.43		Hexa-	M+2/M+4	1.05-1.43
	Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59		Hexa-^{13}C-HxCDF (IS) only	M/M+2	0.43-0.59
	Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51		Hepta-^{13}C-HpCDF (IS) only	M/M+2	0.37-0.51
	Hepta-	M+2/M+4	0.88-1.20		Hepta-	M+2/M+4	0.88-1.20
	Octa-	M+2/M+4	0.76-1.02		Octa-	M+2/M+4	0.76-1.02

LDC #: 24524G2 /

VALIDATION FINDINGS WORKSHEET
Blanks

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

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

Y N N/A Were all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 10/28/10 **Blank analysis date:** 11/09/10 **Associated samples:** 1 → 20

Conc. units: ~~pg/g~~ ng/g

Compound	Blank ID	Sample Identification								
	030420	MB 5X	2	3	5	6	7	8	9	11
G	0.27	1.35	0.87/U	1.0/U	0.46/U	0.80/U	0.41/U	-	0.79/U	1.0/U
O	0.060	0.3	0.099/U	-	0.12/U		0.14/U	0.20/U		0.24/U
	↓	5X	1X	2D						
G	0.27	1.35	-	0.68/U						
O	0.060	0.3	0.25U							

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 #: 24524G2/

VALIDATION FINDINGS WORKSHEET
Field Blanks

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

(Y) N N/A Were field blanks identified in this SDG?
Blank units: pg/L **Associated sample units:** pg/g
Sampling date: 10/26/10
Field blank type: (circle one) Field Blank / Rinsate / Other: FB Associated Samples: All soils (75%)

Compound	Blank ID	Sample Identification								
	23									
G	5.6									
H	2.8									
K	3.4									
L	2.4									
O	5.9									
P	4.1									
Q	9.4									
CRQL										

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

LDC #: 24534G21

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

Page: 1 of 1
Reviewer: FT
2nd Reviewer: 9

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

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

Y N N/A Were 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.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

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

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		24, 25	some compound	were outside			1	no qual as in
			%	R limit				
		26, 27		↓			21	no qual as in

MSD90.21

LDC #: 24524G21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: 9

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	I	30 (40-135)	J|UJ|P qual G, Q
				( )	
		2	I	38 ( )	↓
				( )	
		3	I	33 ( )	G, Q
			G	39 ( )	θ, P
				( )	
		4	H	35 ( )	F
			I	19 ( )	G, Q
			G	32 ( )	θ, P
				( )	
		5	I	30 ( )	G, Q
				( )	
		6	I	23 ( )	G, Q
				( )	
		7	H	38 ( )	F
			I	25 ( )	G, Q
			G	34 (✓)	↓ θ, P
				( )	
				( )	

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

LDC #: 24524G21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT

2nd Reviewer:

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

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

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

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		8	I	27 (40-135)	J | UJ | P qual G, Q
				( )	
		9	I	30 ( )	↓
				( )	
		11	H	38 ( )	F
			I	25 ( )	G, Q
			G	35 ( )	O, P
				( )	
		12	I	26 ( )	G, Q
			G	36 ( )	O, P
				( )	
		13	H	37 ( )	F
			I	23 ( )	G, Q
			G	35 ( )	O, P
				( )	
		14	I	30 ( )	G, Q
				( )	
		15	I	24 (↓)	↓ G, Q
				( )	
				( )	

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

LDC #: 24524G21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: G

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		16	H	35 (40-135)	J | W | P QUAL F
			I	22 ( )	G, Q
			G	31 ( )	B, P
				( )	
		17	H	32 ( )	
			I	22 ( )	
			G	28 ( )	
				( )	
		18	H	39 ( )	
			I	28 ( )	
			G	33 ( )	
				( )	
		19	I	26 ( )	G, Q
			G	37 ( )	B, P
				( )	
		20	I	37 (↓)	↓ G, Q
				( )	
				( )	
				( )	
				( )	

	Internal Standards	Check Standard Used
A.	^{13}C-2,3,7,8-TCDF	
B	^{13}C-2,3,7,8-TCDD	
C	^{13}C-1,2,3,7,8-PeCDF	
D	^{13}C-1,2,3,7,8-PeCDD	
E	^{13}C-1,2,3,6,7,8-HxCDF	
F	^{13}C-1,2,3,6,7,8-HxCDD	
G	^{13}C-1,2,3,4,6,7,8-HpCDF	
H	^{13}C-1,2,3,4,6,7,8-HpCDD	
I	^{13}C-OCDD	

	Recovery Standards	Check Standard Used
K.	^{13}C-1,2,3,4-TCDD	
L	^{13}C-1,2,3,7,8,9-HxCDD	
M		
N		
O		
P		
Q		
R		
T		

LDC #: 24524921

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: [illegible]

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 were within the 40-135% criteria?
Y N N/A Was the S/N ratio all internal standard peaks ≥ 10?

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		21	H	28 (40-135)	J/UJ/P QUAL F
			I	19 ( )	G, Q
			G	30 ( )	↓ Q, P
				( )	
				( )	
		22	I	38 ( )	J/UJ/P QUAL ~~no qual~~ ~~MS~~ G, Q
				( )	
		24	I	34 ( )	no qual ~~MSD~~ MS
				( )	
		25	I	28 ( )	MSP
				( )	
		26	I	24 ( )	MS
				( )	
		27	I	37 ( )	MSD
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 24524G21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

Reviewer: FT
2nd Reviewer: [illegible]

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

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

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

#	Date	compd ~~Sample ID~~	Finding	Associated Samples	Qualifications
		H, I, K, L, O, P, Q	x'd cal Range	19	J/Pdet (e)
		O, Q	↓	21	↓ (e)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524G2 /

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

Page: 1 of 1
Reviewer: FT
2nd Reviewer: Q

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

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

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

#	Date	compd ~~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	9, 13, 15, 17, 18, 23	none/p (o)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24524G21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Reviewer: EF
2nd Reviewer: [illegible]

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

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

Compound	Concentration (pg/g)		%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
	3	4				
D	0.19	2.6U		2.41	≤2.6	
E	0.18	2.6U		2.42	≤2.6	
F	0.40	0.40		0	≤2.6	
G	1.0	1.8		0.8	≤5.3	
H	0.31	0.60		0.29	≤0.53	
I	0.47	0.71		0.24	≤2.6	
J	2.6U	0.31		2.29	≤2.6	
K	0.88	1.1		0.22	≤2.6	
L	0.65	0.95		0.3	≤2.6	
M	0.29	0.19		0.1	≤2.6	
N	0.32	0.21		0.11	≤2.6	
O	1.9	3.0		1.1	≤2.6	
P	1.1	1.6		0.5	≤2.6	
Q	4.1	6.3		2.2	≤5.3	

24524G2/

LDC#: ~~23906B4~~

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Reviewer:
2nd Reviewer:

METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)
Y N NA Were field duplicate pairs identified in this SDG?
Y N NA Were target analytes detected in the field duplicate pairs?

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

LDC #: 24524G21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Reviewer: EF
2nd Reviewer: [illegible]

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (CS3 std)	Recalculated RRF (CS3 std)	Reported %RSD	Recalculated %RSD
1	ICAL	7/21/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.995	0.995	0.9849	0.9849	3.68	3.68
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.983	0.983	0.9681	0.9681	3.24	3.24
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.163	1.163	1.1014	1.1014	5.17	5.17
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.093	1.093	1.0735	1.0735	4.49	4.49
			OCDF (^{13}C-OCDD)	1.370	1.370	1.3500	1.3500	1.98	1.98
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.

LDC #: 24524921
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Reviewer: EJ
2nd Reviewer: [illegible]

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 18:38	11/10/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.995	0.94	0.94	5.3	5.3
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.983	1.04	1.04	5.3	5.3
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.163	1.16	1.16	0	0
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.093	1.08	1.08	0.5	0.5
			OCDF (^{13}C-OCDD)	1.370	1.21	1.21	11.7	11.7
2	CCV 20:20	11/9/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)		0.96	0.96	3.7	3.7
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)		1.01	1.01	2.6	2.6
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)		1.09	1.09	6.0	6.0
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)		1.06	1.06	0.9	0.9
			OCDF (^{13}C-OCDD)		1.22	1.22	10.9	10.9
3	CCV 9:12	11/9/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)		0.93	0.93	6.3	6.3
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)		1.01	1.01	2.7	2.7
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)		1.07	1.07	7.7	7.7
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)		1.06	1.06	0.8	0.8
			OCDF (^{13}C-OCDD)		1.21	1.21	11.5	11.5

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 #: 47314G4

SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

Page: 1 of 1
Reviewer: EJ
2nd Reviewer: G

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: 24 +25

Compound	Spike Added (pg/g)		Sample Concentration (pg/g)	Spiked Sample Concentration (pg/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	21.5	20.5	ND	19.8	18.6	92	92	91	91	6.6	6.6
1,2,3,7,8-PeCDD	107	102	ND	105	98.2	98	98	96	96	6.8	6.8
1,2,3,4,7,8-HxCDD	107	102	0.054	87.4	71.5	81	81	70	70	20	20
1,2,3,4,7,8,9-HpCDF	107	102	2.2	+++	120	116	116	115	115	6.1	6.1
OCDF	215	205	10	127 191	189	84	84	87	87	0.81	0.81

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.

LDC #: 47527921
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: EZ
2nd Reviewer: [signature]

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 030142O-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20	NA	18.4	NA	92	92				
1,2,3,7,8-PeCDD	100		97.3		97	97				
1,2,3,4,7,8-HxCDD	100		72.6		73	73				
1,2,3,4,7,8,9-HpCDF	100		101		101	101				
OCDF	200	↓	151	↓	76	76	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 305.8987 315.9419 317.9389 319.8965 321.8936 331.9368 333.9338 375.8364 [354.9792]	M M+2 M M+2 M M+2 M M+2 M+2 LOCK	$C_{12}H_4{}^{35}Cl_4O$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10$ $^{13}C_{12}H_4{}^{35}Cl_4O$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$ $C_{12}H_4{}^{35}Cl_4O_2$ $C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$ $^{13}C_{12}H_4{}^{35}Cl_4O_2$ $^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$ $C_{12}H_4{}^{35}Cl_5{}^{37}ClO$ C_9F_{13}	TCDF TCDF TCDF (S) TCDF (S) TCDD TCDD TCDD (S) TCDD (S) HxCDPE PFK	4	407.7818 409.7788 417.8250 419.8220 423.7767 425.7737 435.8169 437.8140 479.7165 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O$ $^{13}C_{12}H^{35}Cl_7O$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO$ $C_{12}H^{35}Cl_6{}^{37}ClO_2$ $C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$ $^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$ $C_{12}H^{35}Cl_7{}^{37}Cl_2O$ C_9F_{17}	HpCDF HpCDF HpCDF (S) HpCDF HpCDD HpCDD HpCDD (S) HpCDD (S) NCDPE PFK
2	339.8597 341.8567 351.9000 353.8970 355.8546 357.8516 367.8949 369.8919 409.7974 [354.9792]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 LOCK	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$ $C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$ $^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$ $C_{12}H_3{}^{35}Cl_6{}^{37}ClO$ C_9F_{13}	PeCDF PeCDF PeCDF (S) PeCDF (S) PeCDD PeCDD PeCDD (S) PeCDD (S) HpCDPE PFK	5	441.7428 443.7399 457.7377 459.7348 469.7780 471.7750 513.6775 [422.9278]	M+2 M+4 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}{}^{35}Cl_7{}^{37}ClO$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O$ $C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$ $^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$ $C_{12}{}^{35}Cl_8{}^{37}Cl_2O$ $C_{10}F_{17}$	OCDF OCDF OCDD OCDD OCDD (S) OCDD (S) DCDPE PFK
3	373.8208 375.8178 383.8639 385.8610 389.8156 391.8127 401.8559 403.8529 445.7555 [430.9728]	M+2 M+4 M M+2 M+2 M+4 M+2 M+4 M+4 LOCK	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$ $^{13}C_{12}H_2{}^{35}Cl_6O$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$ $C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$ $^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$ $C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$ C_9F_{17}	HxCDF HxCDF HxCDF (S) HxCDF (S) HxCDD HxCDD HxCDD (S) HxCDD (S) OCDPE PFK					

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24524G27
SDG #: pc cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer: EV
2nd reviewer: CV

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?

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. 9 . OCDF :

Conc. = $\frac{(80737)(4000)(\quad)}{(361)(2600)(1.37)(10.05)(0.932)}$

=

0.70 pg/g

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

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	November 12, 2010
LDC Report Date:	December 20, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0K130496

Sample Identification

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

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

J+ Data are qualified as estimated, with a high bias likely to occur. False positives or false negatives are unlikely to have been reported.

J- Data are qualified as estimated, with a low bias likely to occur. False positives or false negatives are unlikely to have been reported.

J Data are qualified as estimated; it is not possible to assess the direction of the potential bias. False positives or false negatives are unlikely to have been reported.

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

R Data are qualified as rejected. There is a significant potential for the reporting of false negatives or false positives.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The analytical result may be a false positive totally attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JB The analytical result may be biased high and partially attributable to blank contamination. This qualifier is applicable to radiochemistry analysis only.

JK The analytical result is an estimated maximum possible concentration (EMPC).

X The analytical result is not used for reporting because a more accurate and precise result is reported in its place.

J-TDS The analytical result is estimated based on failure of the Total Dissolved Solids (TDS) correctness check performed in accordance with the Standard Method 1030E.

J-CAB The analytical result is estimated based on failure of the cation-anion balance correctness check performed in accordance with Standard Method 1030E.

J-TDS & CAB The analytical result is unreliable based on the failure of the cation-anion balance and TDS correctness check performed in accordance with standard Method 1030E.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

None Indicates the data was not significantly impacted by the finding, therefore qualification was not required.

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

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

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAN6-08-2.0_01_BPC	^{13}C-1,2,3,4,6,7,8-HpCDD ^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	29 (40-135) 23 (40-135) 32 (40-135)	1,2,3,4,6,7,8-HpCDD OCDD OCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects)	P

X. Target Compound Identifications

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

XI. Project Quantitation Limit

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

Sample	Compound	Finding	Criteria	Flag	A or P
SSAN6-08-2.0_01_BPC	2,3,7,8-TCDF 1,2,3,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	P
SSAN6-08-3.0_01_BPC	OCDF	Sample result exceeded calibration range.	Reported result should be within calibration range.	J (all detects)	P

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0K130496	All compounds reported below the PQL.	J (all detects)	A

All compounds reported as EMPC were qualified as follows:

Sample	Compound	Flag	A or P
All samples in SDG G0K130496	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	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 G0K130496

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

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

No Sample Data Qualified in this SDG

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

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24524H21
SDG #: G0K130496
Laboratory: Test America

Date: 12/16/10
Page: 1 of 1
Reviewer: [illegible]
2nd Reviewer: [illegible]

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: 11/12/10
II.	HRGC/HRMS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration~~/ICV~~	A	
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates	N	client specified
VII.	Laboratory control samples	A	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	SW	
X.	Target compound identifications	A	Not reviewed for Stage 2B validation.
XI.	Compound quantitation and CRQLs	SW	Not reviewed for Stage 2B validation.
XII.	System performance	A	Not reviewed for Stage 2B validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Indicates sample underwent Stage 4 validation

SOIL

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

Notes: _____

LDC #: 24524H2/
SDG #: see cover

VALIDATION FINDINGS CHECKLIST

Reviewer: RJ
2nd Reviewer: CV

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS Instrument performance check				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 25% ?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
Was the presence of 1,2,8,9-TCDD and 1,3,4,6,8-PeCDF verified?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning and end of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
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?	✓			

LDC #: 24524H2 1
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer: [illegible]
2nd Reviewer: [illegible]

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 ≥ 2.5, at ± seconds RT) detected in the corresponding PCDPE channel?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
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 #: 24524H21

VALIDATION FINDINGS WORKSHEET
Blanks

Page: 1 of 1
Reviewer: FT
2nd Reviewer:

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

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

Y N N/A Were all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was the method blank contaminated?

Blank extraction date: 11/15/10 **Blank analysis date:** 11/19/10 **Associated samples:** All (75x)

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	031926 6-MB									
G	0.36									

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 #: 24524H2 1

VALIDATION FINDINGS WORKSHEET
Internal Standards

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

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 were within the 40-135% criteria?

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

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	H	29 (40-135)	J/UJ/P qual F
			I	23 (↓)	↓ G, Q
			G	32 (↓)	O, P
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

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

LDC #: 24524 H2|

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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

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

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

#	Date	compd ~~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, I, K, L, O, P, Q	x'd cal Range	1	J/Pdet (e)
		Q	↓	2	↓ (e)

Comments: See sample calculation verification worksheet for recalculations

LDC #: 24524H21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 7
Reviewer: EF
2nd Reviewer: [illegible]

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 Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (CS3 std)	Recalculated RRF (CS3 std)	Reported %RSD	Recalculated %RSD
1	ICAL	10/20/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.120	1.120	1.1574	1.1574	4.94	4.94
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.053	1.053	1.1240	1.1240	4.20	4.20
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.180	1.180	1.2326	1.2326	2.74	2.74
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.104	1.104	1.1554	1.1554	3.85	3.85
			OCDF (^{13}C-OCDD)	1.681	1.681	1.7419	1.7419	5.94	5.94
2	ICAL	11/22/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.079	1.079	1.0473	1.0473	4.90	4.90
	DB225		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.

LDC #: 24524H21
SDG #: all cons

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Reviewer: EF
2nd Reviewer: [illegible]

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

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

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
$RRF = (A_x)(C_{is})/(A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
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 RRF (CC)	Recalculated RRF (CC)	Reported %D	Recalculated %D
1	CCV 0075	11/23/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.079	1.02	1.02	5.5	5.5
	PB225		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)					
2	CCV 21:02	11/19/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.120	1.07	1.07	4.6	4.6
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.053	1.13	1.13	7.2	7.2
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.180	1.15	1.15	2.8	2.8
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.104	1.17	1.17	5.7	5.7
			OCDF (^{13}C-OCDD)	1.687	1.64	1.64	2.2	2.2
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.

LDC #: 24524#21
SDG #: full copy

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Reviewer: EZ
2nd Reviewer: [illegible]

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

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration
SA = Spike added

RPD = I LCS - LCSD I * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 031/9266-LCS

Compound	Spike Added (pg/g)		Spiked Sample Concentration (pg/g)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	20.0	NA	19.3	NA	97	97				
1,2,3,7,8-PeCDD	100	↓	101	↓	101	101				
1,2,3,4,7,8-HxCDD	100	↓	89.1	↓	89	89				
1,2,3,4,7,8,9-HpCDF	100	↓	80.5	↓	80	80				
OCDF	200	↓	173	↓	87	87				

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	$C_{12}H_4{}^{35}Cl_4O$	TCDF	4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}C10$	TCDF		409.7788	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)		417.8250	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)		419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD		423.7767	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}C10_2$	TCDD		425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)		435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	333.9338	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)		437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE		479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
	[354.9792]	LOCK	C_9F_{13}	PFK		[430.9728]	LOCK	C_9F_{17}	PFK
2	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF	5	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF		443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}C10$	PeCDF (S)		457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF (S)		459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD		469.7780	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD		471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)		513.6775	M+4	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)		[422.9278]	LOCK	$C_{10}F_{17}$	PFK
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE					
	[354.9792]	LOCK	C_9F_{13}	PFK					
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF					
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF					
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)					
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)					
	389.8156	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD					
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD					
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)					
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)					
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE					
	[430.9728]	LOCK	C_9F_{17}	PFK					

(a) The following nuclidic masses were used:

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

S = internal/recovery standard

LDC #: 24524A2/
SDG #: pc cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer: RV
2nd reviewer: V

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?

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

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

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

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

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

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

Df = Dilution Factor.

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

Example:

Sample I.D. #3 , OCDF :

Conc. = $\frac{(180667690)(4000)(\quad)}{844(81800)(1.68)(10.13)(0.942)}$

= 533 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
	#3	2,3,7,8-TCDF			
		= 68124966 (2000)			
		451720192 (1.08)(10.13)(0.942)			
		= 29.0 pg/g			