Laboratory Data Consultants, Inc.

7750 El Camino Real, Ste. 2L Carlsbad, CA 92009

Phone 760.634.0437 **Web** www.lab-data.com **Fax** 760.634.0439

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

December 20, 2010

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

Dear Ms. Arnold,

Enclosed is the revised data validation report for the fraction listed below. Please replace the previously submitted report with the enclosed revised report.

LDC Project # 24445:

SDG #	Fraction
G0l140549	Dioxins/Dibenzofurans

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

Sincerely,

Erlinda T. Rauto
Operations Manager/Senior Chemist

Laboratory Data Consultants, Inc.

7750 El Camino Real, Ste. 2L Carlsbad, CA 92009

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

December 8, 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 October 27, 2010. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project # 24445:

SDG #	Fraction
G0H120523, G0H280490, G0I020523, G0I140549	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,

ERauto

Erlinda T. Rauto
Operations Manager/Senior Chemist

DL 10/27/10 Received final SDG 11/29/10 Attachment 1

Stage 2B/4

LDC #24445 (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	G0H120523	10/27/10	12/13/10	1	3																														
A	G0H120523	10/27/10	12/13/10	0	1																														
B	G0H280490	10/27/10	12/13/10	0	14																														
B	G0H280490	10/27/10	12/13/10	0	2																														
C	G0I020523	10/27/10	12/13/10	1	0																														
D	G0I140549	10/27/10	12/13/10	1	5																														
D	G0I140549	10/27/10	12/13/10	0	2																														
Total	T/LR			3	27	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	30

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

LDC #: 24445
SDG #: G0H120523, G0H280490, G0I020523, G0I140549

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_LDC24445_120710.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:	August 9, 2010
LDC Report Date:	December 7, 2010
Matrix:	Soil/Water
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0H120523

Sample Identification

SSAQ4-08-10BPC**
SSAQ4-08-10BPC_FD
SSAQ4-08-1BPC
SSAQ4-08-5BPC
EB-08092010

**Indicates sample underwent Stage 4 review

Introduction

This data review covers 4 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 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
0228254-MB	8/16/10	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-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	2.5 pg/L 2.0 pg/L 2.6 pg/L 7.7 pg/L 2.5 pg/L 1.9 pg/L 1.9 pg/L 1.5 pg/L 2.6 pg/L 2.7 pg/L 2.0 pg/L 3.7 pg/L	All water samples in SDG G0H120523
0228363-MB	8/16/10	1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	0.12 pg/g 0.096 pg/g 0.80 pg/g 0.064 pg/g 0.26 pg/g 0.087 pg/g 0.16 pg/g	All soil samples in SDG G0H120523

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-08092010	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	1.6 pg/L 1.2 pg/L 2.4 pg/L 4.3 pg/L 11 pg/L 2.6 pg/L 2.5 pg/L 10 pg/L	1.6U pg/L 1.2U pg/L 2.4U pg/L 4.3U pg/L 11U pg/L 2.6U pg/L 2.5U pg/L 10U pg/L

Sample EB08092010 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
EB08092010	8/9/10	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 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	1.1 pg/L 1.6 pg/L 1.2 pg/L 2.4 pg/L 4.3 pg/L 5.7 pg/L 11 pg/L 6.9 pg/L 14 pg/L 11 pg/L 2.6 pg/L 2.5 pg/L 2.7 pg/L 10 pg/L 48 pg/L	All soil samples in SDG G0H120523

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

VI. Matrix Spike/Matrix Spike Duplicates

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
SSAQ4-08-1BPC	^{13}C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAQ4-08-5BPC	^{13}C-OCDD	21 (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
SSAQ4-08-10BPC**	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)	P
SSAQ4-08-10BPC_FD SSAQ4-08-1BPC	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
SSAQ4-08-5BPC	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 G0H120523	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 G0H120523	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 SSAQ4-08-10BPC** and SSAQ4-08-10BPC_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
	SSAQ4-08-10BPC	SSAQ4-08-10BPC_FD				
2,3,7,8-TCDD	3.6	2.7	29 (≤50)	-	-	-
1,2,3,7,8-PeCDD	23	16	36 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	15	12	22 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	13	25	63 (≤50)	-	J (all detects)	A
1,2,3,7,8,9-HxCDD	17	12	34 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	120	110	9 (≤50)	-	-	-
OCDD	190	180	5 (≤50)	-	-	-
2,3,7,8-TCDF	120	99	19 (≤50)	-	-	-
1,2,3,7,8-PeCDF	450	380	17 (≤50)	-	-	-
2,3,4,7,8-PeCDF	220	190	15 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	670	650	3 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	670	590	13 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	150	150	0 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	110	98	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	2700	2700	0 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1100	1100	0 (≤50)	-	-	-
OCDF	12000	13000	8(≤50)	-	-	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H120523

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H120523	SSAQ4-08-1BPC SSAQ4-08-5BPC	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H120523	SSAQ4-08-10BPC**	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)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-10BPC_FD SSAQ4-08-1BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-5BPC	OCDF	J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H120523	SSAQ4-08-10BPC** SSAQ4-08-10BPC_FD SSAQ4-08-1BPC SSAQ4-08-5BPC EB-08092010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0H120523	SSAQ4-08-10BPC SSAQ4-08-10BPC_FD	1,2,3,6,7,8-HxCDD	J (all detects)	A	Field duplicates (RPD) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0H120523

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0H120523	EB-08092010	1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,7,8-PeCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,7,8,9-HpCDF	1.6U pg/L 1.2U pg/L 2.4U pg/L 4.3U pg/L 11U pg/L 2.6U pg/L 2.5U pg/L 10U pg/L	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H120523

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24445A21
SDG #: G0H120523
Laboratory: Test America

Date: 12/01/10
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: 8/9/10
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	A	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 = 1, 2
XV.	Field blanks	SW	EB = 5

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

3	1	SSAQ4-08-10BPC**	1 + 1	0230135	21		31	
4	1	SSAQ4-08-10BPC_FD	1 + 2	0228254	22		32	
5	1	SSAQ4-08-1BPC ✓	13		23		33	
9	1	SSAQ4-08-5BPC ✓	14		24		34	
14	2	EB-08092010	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 #: 24445 A21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: EV
2nd Reviewer: V

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 #: 24445A21
SDG #: per 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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Blanks

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 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: 8/16/10 **Blank analysis date:** 8/21/10 **Associated samples:** all water

Conc. units: pg/L

Compound	Blank ID	Sample Identification								
	022825A-MB	5								
D	2.5		1.6/U							
E	2.0		1.2/U							
F	2.6		2.4/U							
G	7.7		4.3/U							
I	2.5		11/U							
K	1.9		~~6.9/U~~							
L	1.9		–							
M	1.5		2.6/U							
N	2.6		2.5/U							
O	2.7		–							
P	2.0		10/U							
Q	3.7		–							

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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Blanks

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 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: 8/16/10 **Blank analysis date:** 8/20/10 **Associated samples:** all soils 7SX
Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	0228363-MB 5X									
E	0.12	0.6								
F	0.096	0.48								
G	0.80	4.0								
K	0.064	0.32								
O	0.26	1.3								
P	0.087	0.435								
Q	0.16	0.5								

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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Field Blanks

Reviewer: FT
2nd Reviewer: [illegible]

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: 8/9/10
Field blank type: (circle one) Field Blank / Rinsate / Other: EB Associated Samples: All soils 75X

Compound	Blank ID	Sample Identification								
	5									
C	1.1									
D	1.6									
E	1.2									
F	2.4									
G	4.3									
H	5.7									
I	11									
J	6.9									
K	14									
L	11									
M	2.6									
N	2.5									
O	27									
P	10									
Q	48									
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 #: 24445 A21

VALIDATION FINDINGS WORKSHEET
Internal Standards

Reviewer: FT
2nd Reviewer: R

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
		3	I	35 (40-135)	J/UJ/P QUAL G, Q
				( )	
		4	I	21 (↓)	↓
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

	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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Compound Quantitation and Reported CRQLs

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)
		a, p, q	x'd cal range	1	J/Part (e)
		a, q	↓	2, 3	↓
		q	↓	4	↓

Comments: See sample calculation verification worksheet for recalculations

LDC#:24445A21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

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

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)
	1	2				
A	3.6	2.7	29			
B	23	16	36			
C	15	12	22			
D	13	25	63			J/A dd
E	17	12	34			
F	120	110	9			
G	190	180	5			
H	120	99	19			
I	450	380	17			
J	220	190	15			
K	670	650	3			
L	670	590	13			
M	150	150	0			
N	110	98	12			
O	2700	2700	0			
P	1100	1100	0			
Q	12000	13000	8			

V:\FIELD DUPLICATES\24445A21.wpd

LDC #: 24485A21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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

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 (LS3 std)	Recalculated RRF (LS3std)	Reported %RSD	Recalculated %RSD
1	ICAL	7/20/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.003	1.003	1.0326	1.0326	3.13	3.13
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.142	1.142	1.0790	1.0790	11.9	11.9
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.130	1.130	1.0831	1.0831	6.08	6.08
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.079	1.079	1.0713	1.0713	1.38	1.38
			OCDF (^{13}C-OCDD)	1.467	1.467	1.4726	1.4726	3.12	3.12
2	ICAL	7/27/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.875	0.875	0.87	0.87	14.2	14.2
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.957	0.957	0.93	0.93	13.5	13.5
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.107	1.107	1.13	1.13	12.5	12.5
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.026	1.026	1.07	1.07	13.6	13.6
			OCDF (^{13}C-OCDD)	1.445	1.445	1.55	1.55	18.1	18.1
3	ICAL	8/16/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.00187	1.00187	1.00395	1.00395	4.19465	4.195
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.16617	1.16617	1.15763	1.15763	8.10393	8.104
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.14627	1.14627	1.16196	1.16196	8.30657	8.306
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.08648	1.08648	1.10879	1.10879	6.01727	6.017
			OCDF (^{13}C-OCDD)	1.54852	1.54852	1.58313	1.58313	8.41555	8.416

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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

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	7/26/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.052	1.052	1.020	1.020	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			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 #: 24445A21
SDG #: per conv

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

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	conv 17:33	8/30/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.052	0.96	0.96	8.7	8.7
	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	conv 8:40	8/24/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.875	0.88	0.88	0	0
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	0.957	0.87	0.87	9.5	9.5
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.107	1.03	1.03	7.0	7.0
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.026	1.07	1.07	4.2	4.2
			OCDF (^{13}C-OCDD)	1.445	1.58	1.58	9.2	9.2
4B 3	conv 21:35	8/20/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.00187	0.90044	0.90044	10.1	10.1
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.16617	1.0963	1.0963	6.0	6.0
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.14627	1.16495	1.16495	1.6	1.6
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.08648	1.08079	1.0879	0.5	0.5
			OCDF (^{13}C-OCDD)	1.54850	1.44162	1.44162	6.9	6.9

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 #: 24445A21

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

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

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

% Difference = 100 * (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 22:09	8/21/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.003	1.06	1.06	5.3	5.3
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.142	1.07	1.07	6.2	6.2
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.130	1.28	1.28	13.2	13.2
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.079	1.08	1.08	0.5	0.5
			OCDF (^{13}C-OCDD)	1.467	1.69	1.69	14.9	14.9
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 #: 24481 A2/
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: F2
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: 02283G3 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	17.1	NA	85	85				
1,2,3,7,8-PeCDD	100		86.6		87	87				
1,2,3,4,7,8-HxCDD	100		93.6		94	94				
1,2,3,4,7,8,9-HpCDF	100		78.9		79	79				
OCDF	200		169		84	84	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 #: 24445A2/
SDG #: pc cover

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer: FM
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. #1 . 2,3,7,8-TCDD

Conc. = $\frac{(4277440)(2000)(\quad)}{(21821770)(1.14)(10.36)(0.919)}$

= 3.6 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#1 2,3,7,8-TCDF (DB225)			
		= 2025038000 (2000			
		(334668000)(1.05)(10.36)(0.919			
		= 120	pg/g		

RECALC90.21

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	August 26, 2010
LDC Report Date:	December 8, 2010
Matrix:	Soil
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0H280490

Sample Identification

BDT-2-S-20-10BPC
BDT-2-S-20-12BPC
BDT-2-S-20-14BPC**
BDT-2-S-20-2BPC
BDT-2-S-20-4BPC
BDT-2-S-20-6BPC
BDT-2-S-20-6BPC_FD
BDT-2-S-20-8BPC
BDT-2-S-15-10BPC
BDT-2-S-15-12BPC
BDT-2-S-15-14BPC**
BDT-2-S-15-2BPC
BDT-2-S-15-4BPC
BDT-2-S-15-6BPC
BDT-2-S-15-8BPC
BDT-2-S-15-2BPC_FD
BDT-2-S-20-10BPCMS
BDT-2-S-20-10BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

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

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

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

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

The following are definitions of the data qualifiers:

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

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

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

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

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

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

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

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

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

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

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

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

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

A Indicates the finding is based upon technical validation criteria.

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

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

I. Technical Holding Times

All technical holding time requirements were met.

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

II. HRGC/HRMS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

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

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

III. Initial Calibration

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

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

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

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

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

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

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

V. Blanks

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

Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
0257312-MB	9/14/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF OCDF	0.081 pg/g 1.4 pg/g 0.089 pg/g 0.23 pg/g	All samples in SDG G0H280490

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. Although the MS/MSD percent recovery (%R) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
BDT-2-S-20-10BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	30 (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
BDT-2-S-20-14BPC**	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	37 (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
BDT-2-S-15-10BPC	^{13}C-1,2,3,4,6,7,8-HpCDF	38 (40-135)	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	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
BDT-2-S-20-2BPC BDT-2-S-20-4BPC	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
BDT-2-S-15-2BPC	2,3,7,8-TCDF 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)	P
BDT-2-S-15-4BPC BDT-2-S-15-2BPC_FD	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

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0H280490	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 G0H280490	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 BDT-2-S-20-6BPC and BDT-2-S-20-6BPC_FD and samples BDT-2-S-15-2BPC and BDT-2-S-15-2BPC_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
	BDT-2-S-20-6BPC	BDT-2-S-20-6BPC_FD				
2,3,7,8-TCDD	1.4	1.6	13 (≤50)	-	-	-
1,2,3,7,8-PeCDD	4.5	5.1	12 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	3.8	4.3	12 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	7.0	7.9	12 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	5.6	6.6	16 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	22	25	13 (≤50)	-	-	-
OCDD	49	64	27 (≤50)	-	-	-
2,3,7,8-TCDF	33	38	14 (≤50)	-	-	-
1,2,3,7,8-PeCDF	53	60	12 (≤50)	-	-	-
2,3,4,7,8-PeCDF	29	33	13 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	96	100	4 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	65	69	6 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	21	25	17 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	9.8	11	12 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	280	310	10 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	120	120	0 (≤50)	-	-	-
OCDF	590	660	11 (≤50)	-	-	-

Compound	Concentration (pg/g)		RPD (Limits)	Difference (Limits)	Flags	A or P
	BDT-2-S-15-2BPC	BDT-2-S-15-2BPC_FD				
2,3,7,8-TCDD	12	11	9 (≤50)	-	-	-
1,2,3,7,8-PeCDD	42	40	5 (≤50)	-	-	-
1,2,3,4,7,8-HxCDD	41	34	19 (≤50)	-	-	-
1,2,3,6,7,8-HxCDD	68	70	3 (≤50)	-	-	-
1,2,3,7,8,9-HxCDD	55	56	2 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDD	230	220	4 (≤50)	-	-	-
OCDD	620	540	14 (≤50)	-	-	-
2,3,7,8-TCDF	300	270	11 (≤50)	-	-	-
1,2,3,7,8-PeCDF	490	440	11 (≤50)	-	-	-
2,3,4,7,8-PeCDF	270	240	12 (≤50)	-	-	-
1,2,3,4,7,8-HxCDF	920	870	6 (≤50)	-	-	-
1,2,3,6,7,8-HxCDF	550	570	4 (≤50)	-	-	-
2,3,4,6,7,8-HxCDF	170	180	6 (≤50)	-	-	-
1,2,3,7,8,9-HxCDF	86	100	15 (≤50)	-	-	-
1,2,3,4,6,7,8-HpCDF	2800	2400	15 (≤50)	-	-	-
1,2,3,4,7,8,9-HpCDF	1100	1000	10 (≤50)	-	-	-
OCDF	6300	7000	11 (≤50)	-	-	-

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0H280490

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-14BPC**	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)
G0H280490	BDT-2-S-15-10BPC	1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
G0H280490	BDT-2-S-20-2BPC BDT-2-S-20-4BPC	1,2,3,4,6,7,8-HpCDF OCDF	J (all detects) J (all detects)	P	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-2BPC	2,3,7,8-TCDF 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)	P	Project Quantitation Limit (exceeded range) (e)
G0H280490	BDT-2-S-15-4BPC BDT-2-S-15-2BPC_FD	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)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC_FD BDT-2-S-20-8BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-2BPC BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0H280490	BDT-2-S-20-10BPC BDT-2-S-20-12BPC BDT-2-S-20-14BPC** BDT-2-S-20-2BPC BDT-2-S-20-4BPC BDT-2-S-20-6BPC BDT-2-S-20-6BPC_FD BDT-2-S-20-8BPC BDT-2-S-15-10BPC BDT-2-S-15-12BPC BDT-2-S-15-14BPC** BDT-2-S-15-2BPC BDT-2-S-15-4BPC BDT-2-S-15-6BPC BDT-2-S-15-8BPC BDT-2-S-15-2BPC_FD	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 G0H280490

No Sample Data Qualified in this SDG

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0H280490

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24445B21
SDG #: G0H280490
Laboratory: Test America

Date: 12/01/10
Page: 1 of 1
Reviewer: FD
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: 8/26/10
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	SW	D = 6, 7 15, 16
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	BDT-2-S-20-10BPC	11	BDT-2-S-15-14BPC**	21	~~025 9197~~	31	
2	BDT-2-S-20-12BPC	12	BDT-2-S-15-2BPC D	22	025 7312	32	
3	BDT-2-S-20-14BPC**	13	BDT-2-S-15-4BPC	23		33	
4	BDT-2-S-20-2BPC	14	BDT-2-S-15-6BPC	24		34	
5	BDT-2-S-20-4BPC	15	BDT-2-S-15-8BPC	25		35	
6	BDT-2-S-20-6BPC	16	BDT-2-S-15-2BPC_FD D	26		36	
7	BDT-2-S-20-6BPC_FD	17	BDT-2-S-20-10BPCMS	27		37	
8	BDT-2-S-20-8BPC	18	BDT-2-S-20-10BPCMSD	28		38	
9	BDT-2-S-15-10BPC	19		29		39	
10	BDT-2-S-15-12BPC	20		30		40	

Notes: _____

LDC #: 24445B21
SDG #: per cover

VALIDATION FINDINGS CHECKLIST

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

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 #: 24445 B21
SDG #: all cover

VALIDATION FINDINGS CHECKLIST

Reviewer: A
2nd Reviewer: J

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 #: 24445 B21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: FT

2nd Reviewer: R

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?

* EMPC

Blank extraction date: 9/14/10 **Blank analysis date:** 9/26/10 **Associated samples:** All 7SX

Conc. units: pg/g

Compound	Blank ID	Sample Identification								
	025732-MB 5X									
F	0.081 *	0.405								
G	1.4 *	7								
O	0.089 *	0.445								
Q	0.23	1.15								

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 #: 24445B21

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

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 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
		17+18	several	%R +)	% RPD were	( )	1	no qual res'n
			outside	limit)	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24445B21

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?

(i)

#	Date	Lab ID/Reference	Internal Standard	% Recovery (Limit: 40-135%)	Qualifications
		1	I	30 (40-135)	J/UJ/P G, Q
			G	37 ( )	Q, P
				( )	
		3	I	37 ( )	
			G	39 ( )	
				( )	
		9	G	38 ( )	Q, P
				( )	
				( )	
		~~8~~ 17	E	39 ( )	no qual MS
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

	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 #: 24445B21

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)
		O, Q	Ex'd cal Range	4, 5	J/P det (e)
		H, O, P, Q	↓	12	↓
		H, O, Q	↓	13, 16	↓

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24445B21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 1
Reviewer: FT
2nd Reviewer: 0

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

(fd)

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) 6	Concentration (pg/g) 7	%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
A	1.4	1.6	13	~~0.2~~		
B	4.5	5.1	12			
C	3.8	4.3	12			
D	7.0	7.9	12			
E	5.6	6.6	16			
F	22	25	13			
G	49	64	27			
H	33	38	14			
I	53	60	12			
J	29	33	13			
K	96	100	4			
L	65	69	6			
M	21	25	17			
N	9.8	11	12			
O	280	310	10			
P	120	120	0			
Q	590	660	11			

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LDC#: 23906B4

VALIDATION FINDINGS WORKSHEET
Field Duplicates

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

METHOD: Metals (EPA Method 6020/7000)

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)
	12	16				
A	12	11	9			
B	42	40	5			
C	41	34	19			
D	68	70	3			
E	55	56	2			
F	230	220	4			
G	620	540	14			
H	300	270	11			
I	490	440	11			
J	270	240	12			
K	920	870	6			
L	550	570	4			
M	170	180	6			
N	86	100	15			
O	2800	2400	15			
P	1100	1000	10			
Q	6300	7000	11			

V:\FIELD DUPLICATES\24445B21.wpd

LDC #: 24445B21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 7
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	DB225 ICAL	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)						
2	ICAL	8/30/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.169	1.169	1.2609	1.2609	5.52	5.52
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.252	1.252	1.2887	1.2887	4.00	4.00
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.202	1.202	1.252	1.252	5.27	5.27
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.180	1.180	1.2654	1.2654	5.62	5.62
			OCDF (^{13}C-OCDD)	1.892	1.892	1.9979	1.9979	6.95	6.95
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 #: 24445B21
SDG #: see cover

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

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 21:16	10/1/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.052	1.19	1.19	12.9	12.9
	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	CCV 22:42	9/28/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.169	1.10	1.10	6.3	6.3
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.252	1.14	1.14	9.3	9.3
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.202	1.18	1.18	1.0	1.0
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.180	1.21	1.21	2.9	2.9
			OCDF (^{13}C-OCDD)	1.892	1.77	1.77	6.6	6.6
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 #: 44445B21
SDG #: per cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

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

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 = I MSR - MSDR I * 2/(MSR + MSDR) MSR = Matrix spike percent recovery MSDR = Matrix spike duplicate percent recovery

MS/MSD samples: 17 + 18

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	21.3	0.36	17.3	19.5	80	80	90	90	12	12
1,2,3,7,8-PeCDD	105	106	1.4	86.8	92.4	81	81	86	86	6.3	6.3
1,2,3,4,7,8-HxCDD	105	106	1.1	79.8	89.8	75	75	83	83	12	12
1,2,3,4,7,8,9-HpCDF	105	106	37	145	308	102	102	255	255	72	72
OCDF	211	213	160	445	1230	135	135	504	504	94	94

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 #: 24443B2/
SDG #: full copy

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

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

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: 025732

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	17.6	NA	88	88				
1,2,3,7,8-PeCDD	100		92.6		93	93				
1,2,3,4,7,8-HxCDD	100		95.9		96	96				
1,2,3,4,7,8,9-HpCDF	100		84.2		84	84				
OCDF	200		153		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 #: 24445B2
SDG #: pc 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. #3 . 2,3,7,8-TCDD

Conc. = $\frac{(342868)(2000)(\quad)}{(16744850000)(1.25)(10.38)(0.934)}$

= 0.34 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		2,3,7,8 - TCDF	(DB225		
		= 17379670 (2000)			
		4669/8000 (1.00) (10.38) (0.934)			
		= 7.244 pg/g			

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	August 31, 2010
LDC Report Date:	December 8, 2010
Matrix:	Water
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0I020523

Sample Identification

EB-08312010

Introduction

This data review covers 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.

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
02651243-MB	9/8/10	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.7 pg/L 39 pg/L 4.2 pg/L	All samples in SDG G0I020523

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-08312010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.5 pg/L 34 pg/L 7.8 pg/L	3.5U pg/L 34U pg/L 7.8U pg/L

Sample EB-8312010 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-08312010	8/31/10	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 1,2,3,7,8-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	1.2 pg/L 2.3 pg/L 2.2 pg/L 3.5 pg/L 34 pg/L 2.9 pg/L 4.2 pg/L 4.2 pg/L 3.0 pg/L 2.4 pg/L 7.8 pg/L 2.8 pg/L 12 pg/L	No associated samples 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. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
0251243-LCS	2,3,4,6,7,8-HxCDF	138 (80-137)	All samples in SDG G0I020523	J+ (all detects)	P

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits.

X. Target Compound Identifications

Raw data were not reviewed for this SDG.

XI. Project Quantitation Limit

All compound quantitation and CRQLs were within validation criteria.

All compounds reported below the PQL were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG G0I020523	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 G0I020523	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 G0I020523

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I020523	EB-08312010	2,3,4,6,7,8-HxCDF	J+ (all detects)	P	Laboratory control samples (%R) (l)
G0I020523	EB-08312010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I020523	EB-08312010	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 G0I020523

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I020523	EB08312010	1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,6,7,8-HpCDF	3.5U pg/L 34U pg/L 7.8U pg/L	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Equipment Blank Data Qualification Summary - SDG G0I020523

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B

LDC #: 24445C21
SDG #: G0I020523
Laboratory: Test America

Date: 12/01/10
Page: 1 of 1
Reviewer: FM
2nd Reviewer: JC

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: 8/31/10
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	BC sample
VII.	Laboratory control samples	SW	LCS
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	
X.	Target compound identifications	N	
XI.	Compound quantitation and CRQLs	N	
XII.	System performance	N	
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	SW	EB = 1

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:
water

1	EB-08312010	11	9/25/10	21		31	
2		12	0251243	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 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 #: 24445C21

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: FT
2nd Reviewer: e

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: 9/8/10 **Blank analysis date:** 9/21/10 **Associated samples:** All (bl)

Conc. units: pg/L

Compound	Blank ID	Sample Identification								
	025 1243	- MB	1							
F	3.7		3.5/U							
G	39		34/U							
O	4.2		7.8/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 #: 24445 c21

VALIDATION FINDINGS WORKSHEET
Field Blanks

Reviewer: FT
2nd Reviewer: [illegible]

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:** none
Sampling date:
Field blank type: (circle one) Field Blank / Rinsate / Other: EB Associated Samples: none

Compound	Blank ID	Sample Identification								
	~~025-243-MB~~ #1									
C	1.2									
D	2.3									
E	2.2									
F	3.5									
G	3.4									
I	2.9									
K	4.2									
L	4.2									
M	3.0									
N	2.4									
O	7.8									
P	2.8									
Q	12									
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 #: 24445C2/1
SDG #: ME cover

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Reviewer: FT
2nd Reviewer: e

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?

(l)

#	Date	Lab ID/Reference	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		025 1243-LCS	M	138 (80-137)	( )	( )	All	J+/P no LCSD,
				( )	( )	( )		no MS/D
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

Revision 1

LDC Report# 24445D21

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name:	Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Collection Date:	September 10, 2010
LDC Report Date:	December 10, 2010
Matrix:	Soil/Water
Parameters:	Dioxins/Dibenzofurans
Validation Level:	Stage 2B & 4
Laboratory:	TestAmerica, Inc.
Sample Delivery Group (SDG):	G0I140549

Sample Identification

SSAJ8-03-1BPC
SSAJ8-03-3BPC
SSAJ8-03-3BPC_FD
SSAJ8-03-5BPC
SSAJ8-03-8BPC
SSAJ8-03-10BPC**
SSAO7-07-0BPC**
EB-09102010
SSAO7-07-0BPCMS
SSAO7-07-0BPCMSD

**Indicates sample underwent Stage 4 review

Introduction

This data review covers 9 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 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
0263138-MB	9/20/10	1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF OCDF	0.063 pg/g 0.11 pg/g 0.64 pg/g 0.062 pg/g 0.058 pg/g 0.040 pg/g 0.048 pg/g 0.14 pg/g 0.13 pg/g	All soil samples in SDG G0I140549

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
SSAJ8-03-1BPC	OCDD	3.1 pg/g	3.1U pg/g
SSAJ8-03-3BPC	OCDD	3.2 pg/g	3.2U pg/g
SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD OCDD	0.11 pg/g 0.84 pg/g	0.11U pg/g 0.84U pg/g
SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.057 pg/g 1.0 pg/g 0.16 pg/g 0.14 pg/g	0.057U pg/g 1.0U pg/g 0.16U pg/g 0.14U pg/g
SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.55 pg/g 0.93 pg/g 0.10 pg/g 0.076 pg/g	0.55U pg/g 0.93U pg/g 0.10U pg/g 0.076U pg/g

Sample EB09102010 was identified as an equipment blank. No polychlorinated dioxin/dibenzofuran contaminants were found in this blank.

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) and relative percent differences (RPD) were not within QC limits for several compounds, the LCS percent recoveries (%R) were within QC limits and no data were qualified.

VII. Laboratory Control Samples (LCS)

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

VIII. Regional Quality Assurance and Quality Control

Not applicable.

*IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
SSAJ8-03-1BPC	^{13}C-OCDD ^{13}C-1,2,3,4,6,7,8-HpCDF	27 (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
SSAJ8-03-3BPC	^{13}C-OCDD	34 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAJ8-03-5BPC	^{13}C-OCDD	35 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAJ8-03-10BPC**	^{13}C-OCDD	32 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P
SSAO7-07-0BPC**	^{13}C-OCDD	33 (40-135)	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

*Corrected %R for SSAO7-07-0BPC** in table above.

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
SSAJ8-03-10BPC**	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 G0I140549	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 G0I140549	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 SSAJ8-03-3BPC and SSAJ8-03-3BPC_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
	SSAJ8-03-3BPC	SSAJ8-03-3BPC_FD				
2,3,7,8-TCDD	0.26	0.091	-	0.169 (≤0.55)	-	-
1,2,3,7,8-PeCDD	0.68	0.21	-	0.47 (≤2.8)	-	-
1,2,3,4,7,8-HxCDD	0.50	0.11	-	0.39 (≤2.8)	-	-
1,2,3,6,7,8-HxCDD	1.0	0.29	-	0.71 (≤2.8)	-	-
1,2,3,7,8,9-HxCDD	0.81	0.17	-	0.64 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDD	3.4	0.87	-	2.53 (≤2.8)	-	-
OCDD	3.2	0.84	-	2.36 (≤5.5)	-	-
2,3,7,8-TCDF	6.0	1.2	-	4.8 (≤0.55)	J (all detects)	A
1,2,3,7,8-PeCDF	11	2.2	-	8.8 (≤2.8)	J (all detects)	A
2,3,4,7,8-PeCDF	6.0	1.1	-	4.9 (≤2.8)	J (all detects)	A
1,2,3,4,7,8-HxCDF	16	3.0	-	13 (≤2.8)	J (all detects)	A
1,2,3,6,7,8-HxCDF	13	2.7	-	10.3 (≤2.8)	J (all detects)	A
2,3,4,6,7,8-HxCDF	2.8	0.63	-	2.17 (≤2.8)	-	-
1,2,3,7,8,9-HxCDF	2.5	0.62	-	1.88 (≤2.8)	-	-
1,2,3,4,6,7,8-HpCDF	51	10	-	41 (≤2.8)	J (all detects)	A
1,2,3,4,7,8,9-HpCDF	21	4.6	-	16.4 (≤2.8)	J (all detects)	A
OCDF	110	20	-	90 (≤5.5)	J (all detects)	A

*Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Data Qualification Summary - SDG G0I140549

SDG	Sample	Compound	Flag	A or P	Reason (Code)
G0I140549	SSAJ8-03-1BPC	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)
G0I140549	SSAJ8-03-3BPC SSAJ8-03-5BPC SSAJ8-03-10BPC** SSAO7-07-0BPC**	OCDD OCDF	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Internal standards (%R) (i)
*G0I140549	SSAJ8-03-10BPC**	2,3,7,8-TCDF	None	P	Project Quantitation Limit (o)
G0I140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported below the PQL.	J (all detects)	A	Project Quantitation Limit (sp)
G0I140549	SSAJ8-03-1BPC SSAJ8-03-3BPC SSAJ8-03-3BPC_FD SSAJ8-03-5BPC SSAJ8-03-8BPC SSAJ8-03-10BPC** SSAO7-07-0BPC** EB-09102010	All compounds reported by the lab as estimated maximum possible concentration (EMPC)	JK (all detects)	A	Project Quantitation Limit (k)
G0I140549	SSAJ8-03-3BPC SSAJ8-03-3BPC_FD	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 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)	A	Field duplicates (RPD) (fd)

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG G0I140549

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I140549	SSAJ8-03-1BPC	OCDD	3.1U pg/g	A	bl

SDG	Sample	Compound	Modified Final Concentration	A or P	Code
G0I140549	SSAJ8-03-3BPC	OCDD	3.2U pg/g	A	bl
G0I140549	SSAJ8-03-3BPC_FD	1,2,3,4,7,8-HxCDD OCDD	0.11U pg/g 0.84U pg/g	A	bl
G0I140549	SSAJ8-03-8BPC	1,2,3,4,7,8-HxCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.057U pg/g 1.0U pg/g 0.16U pg/g 0.14U pg/g	A	bl
G0I140549	SSAJ8-03-10BPC**	1,2,3,4,6,7,8-HpCDD OCDD 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	0.55U pg/g 0.93U pg/g 0.10U pg/g 0.076U pg/g	A	bl

Tronox LLC Facility, PCS Additional Sampling, Henderson, Nevada
Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG G0I140549

No Sample Data Qualified in this SDG

Tronox Northgate Henderson
VALIDATION COMPLETENESS WORKSHEET
Stage 2B/4

LDC #: 24445D21
SDG #: G0I140549
Laboratory: Test America

Date: 12/01/10
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: 9/10/10
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	SW	D = 2, 3
XV.	Field blanks	ND	EB = 8

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	1	SSAJ8-03-1BPC	11	02 63138.	21		31	
2	1	SSAJ8-03-3BPC	12	02 8 1099	22		32	
3	1	SSAJ8-03-3BPC_FD	13		23		33	
4	1	SSAJ8-03-5BPC	14		24		34	
5	1	SSAJ8-03-8BPC	15		25		35	
6	1	SSAJ8-03-10BPC**	16		26		36	
7	1	SSAO7-07-0BPC**	17		27		37	
8	2	EB-09102010	18		28		38	
9		SSAO7-07-0BPCMS	19		29		39	
10		SSAO7-07-0BPCMSD	20		30		40	

Notes:

LDC #: 24445 D21
SDG #: see cover

VALIDATION FINDINGS CHECKLIST

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

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

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS instrument performance check.				
Was PFK exact mass 380.9760 verified?	✓			
Were the retention time windows established for all homologues?	✓			
Was the chromatographic resolution between 2,3,7,8-TCDD and peaks representing any other unlabeled TCDD isomers ≤ 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 #: 24445D2|
SDG #: all cores

VALIDATION FINDINGS CHECKLIST

Reviewer: FT
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 #: 24445 D21

VALIDATION FINDINGS WORKSHEET
Blanks

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 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: 9/20/10 **Blank analysis date:** 10/6/10 **Associated samples:** All soils

Conc. units: ng/g

Compound	Blank ID	Sample Identification								
	0263138-MB 5%	1	2	3	5	6				
C	0.063	0.315			0.11/U	0.057/U				
F	0.11	0.55					0.55/U			
G	0.64	3.2	3.1/U	3.2/U	0.84/U	1.0/U	0.93/U			
K	0.062	0.31								
L	0.058	0.29								
M	0.040	0.20				0.16/U	0.10/U			
N	0.048	0.24				0.14/U	0.076/U			
O	0.14	0.70								
Q	0.13	0.55				(4.8)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 #: 21144SD21

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

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 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+10	several	%R +)	% RPD)	( )	7	no qual resin
			out	side (limit	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		

LDC #: 24445D21

VALIDATION FINDINGS WORKSHEET
Internal Standards

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 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
		1	I	27 (40-135)	J/UJ/P QMAL G,Q
			G	37 ( )	G,P
				( )	
		2	I	34 ( )	G,Q
				( )	
		4	I	35 ( )	G,Q
				( )	
		6	I	32 ( )	G,Q
				( )	
		7	I	33 ( )	G,Q
				( )	
		9	H	29 ( )	no qual MS
			I	20 ( )	
			G	29 ( )	
				( )	
		10	H	24 ( )	MSD
			I	17 ( )	
			G	24 ( )	
				( )	
				( )	

	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 #: 24445D27

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	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	6	~~J~~R. mne/p (e)

Comments: See sample calculation verification worksheet for recalculations

LDC#: 24445D21

VALIDATION FINDINGS WORKSHEET
Field Duplicates

Page: 1 of 1
Reviewer: EJ
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?

(1d)

Compound	Concentration (pg/g) 2	Concentration (pg/g) 3	%RPD ≤50	(pg/g) Difference	(pg/g) Limits	Qualifications (Parent Only)
A	0.26	0.091		0.169	≤0.55	
B	0.68	0.21		0.47	≤2.8	
C	0.50	0.11		0.39	≤2.8	
D	1.0	0.29		0.71	≤2.8	
E	0.81	0.17		0.64	≤2.8	
F	3.4	0.87		2.53	≤2.8	
G	3.2	0.84		2.36	≤5.5	
H	6.0	1.2		4.8	≤0.55	J/A det
I	11	2.2		8.8	≤2.8	↓
J	6.0	1.1		4.9	≤2.8	↓
K	16	3.0		13	≤2.8	↓
L	13	2.7		10.3	≤2.8	↓
M	2.8	0.63		2.17	≤2.8	-
N	2.5	0.62		~~1.92~~ 1.88	≤2.8	-
O	51	10		41	≤2.8	J/A det
P	21	4.6		16.4	≤2.8	↓
Q	110	20		90	≤5.5	↓

LDC #: 24445 D21
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	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
2	DB225	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
			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 #: 24445 D2/
SDG #: per cover

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 19:46	10/5/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	0.984	1.03	1.03	4.5	4.5
			2,3,7,8-TCDD (^{13}C-2,3,7,8-TCDD)	1.032	1.03	1.03	0.1	0.1
			1,2,3,6,7,8-HxCDD (^{13}C-1,2,3,6,7,8-HxCDD)	1.141	1.27	1.27	11.6	11.6
			1,2,3,4,6,7,8-HpCDD (^{13}C-1,2,4,6,7,8,-HpCDD)	1.134	1.25	1.25	10.2	10.2
			OCDF (^{13}C-OCDD)	2.118	2.0	2.0	5.4	5.4
2	CCV DB225 21:11	10/8/10	2,3,7,8-TCDF (^{13}C-2,3,7,8-TCDF)	1.052	1.04	1.04	1.2	1.2
			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 #: 44443-24
SDG #: per cover

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

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

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

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

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

RPD = I MSR - MSDR I * 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	19.8	20.1	3.1	28.5	26.6	129	129	117	117	6.9	6.9
1,2,3,7,8-PeCDD	98.7	100	8.6	116	113	109	109	104	104	2.6	2.6
1,2,3,4,7,8-HxCDD	↓	↓	5.9	124	126	119	119	119	119	1.9	1.9
1,2,3,4,7,8,9-HpCDF	↓	↓	250	893	460	654	654	212	212	64	64
OCDF	198	201	1200	4800	2010	1800	1800	381	381	82	82

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 #: 44453W4/
SDG #: [illegible]

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: 02-63138-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	20.7	NA	104	104				
1,2,3,7,8-PeCDD	100	↓	111	↓	111	111				
1,2,3,4,7,8-HxCDD	100		102		102	102				
1,2,3,4,7,8,9-HpCDF	100		109		109	109				
OCDF	200	↓	176	↓	88	88				

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_6F_{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_6F_{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_6F_{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_6F_{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	O = 15.994915
C = 12.000000	^{35}Cl = 34.968853
^{13}C = 13.003355	^{37}Cl = 36.965903
F = 18.9984	

S = Internal/recovery standard

LDC #: 24445D21
SDG #: pu 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. #7, 2,3,7,8-TCDD

Conc. = $\frac{(10753240)(2000)(\quad)}{(6626010003)(1.03)(10.40)(0.9918)}$

= 3.1 pg/g

#	Sample ID	Compound	Reported Concentration ( )	Calculated Concentration ( )	Qualification
		#7 2,3,7,8-TCDF			
		= 215320000 (2000)			
		(4926450000)(1.056)(10.40)(0.9918)			
		= 80 pg/g			

RECALC90.21