

## LABORATORY DATA CONSULTANTS, INC.

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August 6, 2008

ERM 2525 Natomas Park Drive, Suite 350 Sacramento, CA 95833 ATTN: Ms. Maria Barajas-Albalawi

SUBJECT: BRC Tronox Parcel F, Data Validation

Dear Ms. Barajas-Albalawi

Enclosed are the final validation reports for the fractions listed below. This SDG was received on July 11, 2008. Attachment 1 is a summary of the samples that were reviewed for each analysis.

## LDC Project # 19091:

- SDG # Fraction
- F8F110177 Volatiles, Semivolatiles, Chlorinated Pesticides, Polychlorinated Biphenyls, Metals, Wet Chemistry, Gasoline Range Organics, Diesel Range Organics, Polynuclear Aromatic Hydrocarbons, Dioxins/Dibenzofurans

The data validation was performed under EPA Level III and Level IV guidelines. The analyses were validated using the following documents, as applicable to each method:

- USEPA, Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999
- USEPA, Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004
- EPA SW 846, Third Edition, Test Methods for Evaluating Solid Waste, update 1, July 1992; update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998; IIIB, November 2004; Update IV, February 2007

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

Sincerely,

Erlinda T. Rauto Operations Manager/Senior Chemist

LDC #13091 (ERM-Sacramento / BRC Tronox Parcel F)           stora         byte         001         environ formation         000         stora         stora <t< th=""><th>BOX3         LOC #1001         ERX TOON Parcel         PART         Diversion of the part o</th><th></th><th>5,203 Pages-CD R2</th><th>CD R3</th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th>¥#</th><th>Attachment 1</th><th>ent</th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th></t<>	BOX3         LOC #1001         ERX TOON Parcel         PART         Diversion of the part o		5,203 Pages-CD R2	CD R3												¥#	Attachment 1	ent																			
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## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel F
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Collection Date: June 10, 2008

LDC Report Date: August 6, 2008

Matrix: Soil/Water

Parameters: Volatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

## Sample Delivery Group (SDG): F8F110177

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30' TB-2 6/10/08 TSB-FJ-02-02-30'MS TSB-FJ-02-02-30'MSD

## \*\*Indicates sample underwent EPA Level IV review

## Introduction

This data review covers 7 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 8260B for Volatiles.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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.
- 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. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination ( $r^2$ ) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all volatile target compounds and system performance check compounds (SPCCs) were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/12/08	Ethanol	0.00148 (≥0.05)	All soil samples in SDG F8F110177	J (all detects) UJ (all non-detects)	A

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0% for calibration check compounds (CCCs).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
6/19/08 (LCAL0317)	lodomethane	67.71684	TB-2 6/10/08 F8F200000-125	J+ (all detects)	A

The percent differences (%D) of the second source calibration standard were less than or equal to 25.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
5/28/08 (LICV9881)	lodomethane	31.67513	All water samples in SDG F8F110177	J+ (all detects)	A
5/28/08 (LICV9881)	2-Hexanone	25.04476	All water samples in SDG F8F110177	J- (all detects) UJ (all non-detects)	A
6/9/08 (XICV2280)	Methylene chloride	29.90220	All soil samples in SDG F8F110177	J- (all detects) UJ (all non-detects)	A

All of the continuing calibration RRF values were within method and validation criteria.

## V. Blanks

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

Method Blank ID	Analysis Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
F8F120000-446	6/12/08	Tetrachloroethene	1.5 ug/Kg	All soil samples in SDG F8F110177

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Sample	Compound	Reported	Modified Final
	TIC (RT in minutes)	Concentration	Concentration
TSB-FR-02-02-20'	Tetrachloroethene	1.4 ug/Kg	5.6U ug/Kg

Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
TSB-FR-02-02-30'**	Tetrachloroethene	1.3 ug/Kg	7.2U ug/Kg
TSB-FJ-02-02-10'**	Tetrachloroethene	1.6 ug/Kg	6.6U ug/Kg
TSB-FJ-02-02-20'**	Tetrachloroethene	1.3 ug/Kg	6.1U ug/Kg
TSB-FJ-02-02-30'	Tetrachloroethene	1.2 ug/Kg	6.5U ug/Kg

Sample TB-2 6/10/08 was identified as a trip blank. No volatile contaminants were found in this blank with the following exceptions:

Trip Blank ID	Sampling Date	Compound	Concentration	Associated Samples
TB-2 6/10/08	6/10/08	Acetone Chloroform	2.9 ug/L 0.14 ug/L	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'

Sample concentrations were compared to concentrations detected in the field blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated field blanks.

## VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits with the following exceptions:

Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
F8F200000-125	Bromofluorobenzene	117 (79-115)	All TCL compounds	J+ (all detects)	Р

## VII. 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 some compounds, the MS/MSD percent recoveries (%R) were within QC limits and no data were qualified.

## VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Although the relative percent differences (RPD) for one compound and the percent recoveries for some compounds in the LCS/LCSD were not within QC limits, the LCS and MS/MSD percent recoveries (%R) were within QC limits and no data were qualified.

## IX. Regional Quality Assurance and Quality Control

Not applicable.

## X. Internal Standards

All internal standard areas and retention times were within QC limits with the following exceptions:

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-FR-02-02-30'**	1,4-Dichlorobenzene-d4	172980 (187131-748522)	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichlorobenzene 1,2,4-Trimethylbenzne 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3-Dichlorobenzene 1,3-Dichlorobenzene 2-Chlorotoluene Bromobenzene Isopropylbenzene n-Butylbenzene n-Propylbenzene p-Cymene sec-Butylbenzene tert-Butylbenzene 1,3,5-Trichlorobenzene Nonanal Bromoform	J (all detects) UJ (all non-detects)	Ρ

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-FJ-02-02-10'**	1,4-Dichlorobenzene-d4	180609 (187131-748522)	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzne 1,2-Dichlorobenzene 1,3-5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene Bromobenzene Isopropylbenzene n-Butylbenzene n-Propylbenzene p-Cymene sec-Butylbenzene tert-Butylbenzene 1,3,5-Trichlorobenzene Nonanal Bromoform	J (all detects) UJ (all non-detects)	Ρ
TSB-FJ-02-02-20'**	1,4-Dichlorobenzene-d4	171259 (187131-748522)	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,2-A-Trimethylbenzene 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,3-Dichlorobenzene 2-Chlorotoluene Bromobenzene Isopropylbenzene n-Butylbenzene n-Propylbenzene p-Cymene sec-Butylbenzene tett-Butylbenzene 1,3,5-Trichlorobenzene Nonanal Bromoform	J (all detects) UJ (all non-detects)	Ρ

Sample	Internal Standards	Area (Limits)	Compound	Flag	A or P
TSB-FJ-02-02-30'	1,4-Dichlorobenzene-d4	168365 (187131-748522)	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,3-5-Trimethylbenzene 1,3-5-Trimethylbenzene 1,4-Dichlorobenzene 2-Chlorotoluene Bromobenzene Isopropylbenzene n-Butylbenzene n-Propylbenzene p-Cymene sec-Butylbenzene tert-Butylbenzene 1,3,5-Trichlorobenzene Nonanal Bromoform	J (all detects) UJ (all non-detects)	Ρ

## XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

## XII. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

## XIII. Tentatively Identified Compounds (TICs)

All tentatively identified compounds were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

## XIV. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

## XV. Overall Assessment of Data

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

## XVI. Field Duplicates

No field duplicates were identified in this SDG.

## BRC Tronox Parcel F Volatiles - Data Qualification Summary - SDG F8F110177

SDG	Sample	Compound	Flag	A or P	Reason
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Ethanol	J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F110177	TB-2 6/10/08	Iodomethane	J+ (all detects)	A	Continuing calibration (%D)
F8F110177	TB-2 6/10/08	lodomethane	J+ (all detects)	A	Continuing calibration (ICV %D)
F8F110177	TB-2 6/10/08	2-Hexanone	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Methylene chloride	J- (all detects) UJ (all non-detects)	A	Continuing calibration (ICV %D)
F8F110177	TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3-5-Trimethylbenzene 1,3-Dichlorobenzene 2-Chlorotoluene Bromobenzene Isopropylbenzene n-Butylbenzene n-Butylbenzene p-Cymene sec-Butylbenzene tert-Butylbenzene 1,3,5-Trichlorobenzene Nonanal Bromoform	J (all detects) UJ (all non-detects)	Ρ	Internal standards (area)

## BRC Tronox Parcel F Volatiles - Laboratory Blank Data Qualification Summary - SDG F8F110177

SDG	Sample	Compound TIC (RT in minutes)	Modified Final Concentration	A or P
F8F110177	TSB-FR-02-02-20'	Tetrachloroethene	5.6U ug/Kg	A
F8F110177	TSB-FR-02-02-30'**	Tetrachloroethene	7.2U ug/Kg	A
F8F110177	TSB-FJ-02-02-10'**	Tetrachloroethene	6.6U ug/Kg	A
F8F110177	TSB-FJ-02-02-20'**	Tetrachloroethene	6.1U ug/Kg	A
F8F110177	TSB-FJ-02-02-30'	Tetrachloroethene	6.5U ug/Kg	A

## BRC Tronox Parcel F Volatiles - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: <u>19091A1</u>	VALIDATION COMPLETENESS WORKSHEET	Date: 7/19/08
SDG #:	_ Level III/IV	Page:/of/_
Laboratory: Test America		Reviewer: <u>Fi</u>
METHOD: GC/MS Volatiles (E	EPA SW 846 Method 8260B)	2nd Reviewer:

## METHOD: GC/MS Volatiles (EPA SW 846 Method 8260B)

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
Ι.	Technical holding times	4	Sampling dates: 6/10/08
11.	GC/MS Instrument performance check	4	
III.	Initial calibration	A	% PSD, 12 20.990
IV.	Continuing calibration/ICV	SW	101 4 25
V.	Blanks	sw	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	SW	Rinsate - 2
VIII.	Laboratory control samples	SW	
IX.	Regional Quality Assurance and Quality Control	N	
Х.	Internal standards	SW	
XI.	Target compound identification	4	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	Δ	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	Δ	Not reviewed for Level III validation.
XIV.	System performance	Δ	Not reviewed for Level III validation.
XV.	Overall assessment of data	4	
XVI.	Field duplicates	N	
XVII.	Field blanks	∽s√	$TB = L_0$

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 Level IV validation

			<u> </u>				
71	TSB-FR-02-02-20'	11	F8F120000-446	21	8164446	31	
<b>1</b> 2	TSB-FR-02-02-30'**	122	F8F200000175	22	817-212	32	
3 /	TSB-FJ-02-02-10'**	133	F8 F2 00000 - 36	23	8172361 NON9	33	
4 /	TSB-FJ-02-02-20'**	14	<b>`</b>	24		34	
5 /	TSB-FJ-02-02-30'	15		25		35	
6 V	3: Nonana' W	16		26		36	
7	TSB-FJ-02-02-30'MS	17		27		37	
8	TSB-FJ-02-02-30'MSD	18		28		38	
9		19		29		39	
10		20		30		40	

LDC #: 1909(A) SDG #: pu coner

Page: / of 2 Reviewer: 77 2nd Reviewer: /

## Method: Volatiles (EPA SW 846 Method 8260B)

Validation Area	Yes	No	NA	Findings/Comments
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All technical holding times were met.	1/		ļ	
Cooler temperature criteria was met.	-			
U. SEANS INSTAILED AND AND AND AND AND AND AND AND AND AN				
Were the BFB performance results reviewed and found to be within the specified criteria?	-			
Were all samples analyzed within the 12 hour clock criteria?		Na men in Jame Con		
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	1			
Was a curve fit used for evaluation?	<u> </u>			
Did the initial calibration meet the curve fit acceptance criteria of $\geq 0.990?$	1			
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?				
		<u>Andres</u>		
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<			
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	~			
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?		7		
			-	
Was a method blank associated with every sample in this SDG?	-			
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?	-			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/			
Were all surrogate %R within QC limits?		_		
If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R outside of criteria?		1		· · · · · · · · · · · · · · · · · · ·
MIL MARK AT RECORDED AND A DESCRIPTION OF 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.	~			
Was a MS/MSD analyzed every 20 samples of each matrix?				
Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?		1		· · ·
Vas an LCS analyzed for this SDG?	1			

## VALIDATION FINDINGS CHECKLIST

LDC #:	19091A)
SDG #:	pil cover

Page:	2	of	2

Reviewer: \_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?		Ľ		
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
INCREMENTATION ASSOCIATION COMPLETENCE AND A SECOND AND A S				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?				
Were internal standard area counts within -50% or +100% of the associated calibration standard?		/		
Were retention times within + 30 seconds of the associated calibration standard?		r		
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?		r		
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?				
And formative contractions (198) in the second s				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?				
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?	/			
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?				
System performance was found to be acceptable.	_			
Overall assessment of data was found to be acceptable.				
ND THURSDAY AND A CONTRACT OF A				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			7	
XVII. Perly Micks 2014 Company and a second s				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.	7			

## TARGET COMPOUND WORKSHEET

# METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	00. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohoi
C. Vinyl choride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachiorobutadiene	FFFF. Acrolein
E. Methylene chloride	Y. 4-Methyl-2-pentanone	SS. 1,3-Dichloropropane	MMM. Naphthalene	GGGG. Acrylonitrile
F. Acetone	Z. 2-Hexanone	TT. 1,2-Dibromoethane	NNN. 1,2,3-Trichiorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	000. 1,3,5-Trichlorobenzene	IIII. Isobutyi alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
1. 1,1-Dichloroethane*	CC. Toluene**	WW. Bromobenzene	QQQ. cis-1,2-Dichloroethene	KKKK. Propionitrile
J. 1,2-Dichloroethene, total	DD. Chlorobenzene*	XX. 1,2,3-Trichloropropane	RRR. m,p-Xylenes	LLLL. Ethyl ether
K. Chloroform**	EE. Ethylbenzene**	YY. n-Propylbenzene	SSS. o-Xylene	MMMM. Benzyl chloride
L. 1,2-Dichloroethane	FF. Styrene	ZZ. 2-Chlorotoluene	TTT. 1,1,2-Trichloro-1,2,2-trifluoroethane	NNNN.
M. 2-Butanone	GG. Xylenes, total	AAA. 1,3,5-Trìmethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	0000.
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chiorotoluene	VVV. 4-Ethyltoluene	PPPP.
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	ୟସୟସ.
P. Bromodichloromethane	JJ. Dichlorodifiuoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRR.
Q. 1,2-Dichloropropane**	KK. Trichlorofluoromethane	EEE. sec-Butylbenzene	YYY. tert-Butanol	SSSS.
R. cis-1,3-Dichloropropene	LL. Methyl-tert-butyl ether	FFF. 1,3-Dichlorobenzene	ZZZ. tert-Butyl alcohol	TTT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAAA. Ethyl tert-butyl ether	υυυυ.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBBB. tert-Amyl methyl ether	vvvv.

\* = System performance check compounds (SPCC) for RRF ; \*\* = Calibration check compounds (CCC) for %RSD.

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LU cones LDC #: 1909114 1 SDG #: 1909114 1

# VALIDATION FINDINGS WORKSHEET

Page: / of / 8 Reviewer:\_\_\_\_\_\_\_2nd Reviewer:\_\_\_\_\_\_\_

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

question: analysis respon tance cr %RSD	Qualifications														
	Associated Samples			F&F120000-446											
	Finding RRF (Limit: <u>&gt;</u> 0.05)			0.00148											
	Finding %RSD (Limit: ≤30.0%)														
	Compound			ر س س س											
cations below for all quid the laboratory perforence of the percent relative stras a curve fit used for the initial calibration tere all %RSDs and Ru	Standard ID	X 1CAL-1		1CALX-BRC											
Please see qualific N N/A N/A Di X N N/A W X N/N/A W V N/A Di V N/A Di	# Date	6/2/9	-	80 21 7	-										

LDC # 1909 A)

## VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

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

METHOD: GC/MS VOA (EPA SW 846 Method 8260B) Please see qualifications אמומני ב- - -"

N N/A N N/A 5 22 000 L 9 08	Were all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF ?       Associated Samples         Nere all %D and RRFs within the validation criteria of ≤25 %D and ≥0.05 RRF       Finding RRF       Associated Samples         Notated B       compound       (Limit: <25.0%)       Finding RRF       Associated Samples         Notated B       Locdom ethance       31.6712       RF       Associated Samples         Notated B       Z       >5.04474       All wated       All wated         X1L0V2180       E       34.90210       FSF12.0080-4446       All Soil	s within the validation cri compound Z Z Z E E	teria of ≤25 %U and > Finding %D (Limit: ≤25.0%) 31. 675/3 25. ¢4474 25. ¢4474	Finding RRF (Limit: <u>&gt;</u> 0.05)	Associated Samples F&F2.00 vob-156, All water F&F12 0000-446 All Soil	Qualifications 1+/A det 1-/41/A 1-/ 41/A	
6/19/08	LCALOZIT	Todomethane	48916.12		F&F20000-12,	J1/Adrt	

N.

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19091	, LE
	SDG #:

## VALIDATION FINDINGS WORKSHEET **Blanks**

Page: 2nd Reviewer; Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". Was a method blank associated with every sample in this SDG? A/N N

A/N N

Was a method blank analyzed at least once every 12 hours for each matrix and concentration? Was there contamination in the method blanks? If yes, please see the qualifications below. // Ø 6/12/08 B/ank analysis date:\_\_\_\_ Y/N N/Y

Conc. units: Up / Key	3	As	Associated Samples	iples:	A //	201/105	~
Compound	Blank ID			S	Sample Identificat	lon	
	F8F12000	4	r	4	12		

		T	T						Ī
					-				
							_		
			-						
			i   			_		 -	
		5							
	h	1.2 16.54	2.2/						
	4	1.3 6.14							
	n	1.6 16.64		_					
	4	1.2 V							
-	1	1.4/564 1.3	_						
<b>ボ</b> エ ロ / ひ の っ つ 9	-441.	1.5							
		AA							
		<u>Methylene chloride</u> AA	Acetone						CROL

Blank analysis date:

Associated Samples: Cone. units:

Compound	Blank ID			ŭ	Sample Identification	tion		
			-					
Methyiene chloride		-						
Acetone								
						<b>-</b>		
CRAL							-	

All results were qualified using the criteria stated below except those circled.

Note: Common contaminants such as Methylene chloride, Acetone, 2-Butanone, Carbon disulfide and TICs that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC # 19091& 1 SDG # LOULY			VALIDATION FINDINGS WORKSHEET Field Blanks	S WORKSHI Inks	ET		Page:
METHOD: GC/MS VOA (EPA SW 846 Method 8260B) Y N/N/A Were field blanks identified in this SDG Y N N/A Were target compounds detected in th	A SW 846 Me anks identifie compounds c	AS VOA (EPA SW 846 Method 8260B) Were field blanks identified in this SDG? Were target compounds detected in the	? fiejd blanks?				2nd Reviewer:
Blank units: Walk Associated sample units: Walk Field blank type. (circle one) Field Blank / Rinsate / Trip Blan	ciated samp ) Field Blank	le units: us / Rinsate / Tri	/Trid Blank / Other: TD	Associated Samples:	Samples:	5 4-1	(ND)
Compound	Blank ID 6	Blank ID		ø	Sample Identification	Ę	
and a state of the	6  m/oX						
Methylene chloride							
Acetone	5.5						
Chloroform	hl·o						
CRQL							
Blank units:Associated sample units: Field blank type: (circle one) Field Blank / Rinsate / Trip Blank / Other:	Associated sample units: e one) Field Blank / Rinsate	le units: / Rinsate / Tri	p Blank / Other:	Associated Samples:	Samples:		
Compound	Blank ID	Blank ID		0	Sample Identification	u	
Sampling Data							
Methyiene chloride							
Acetone	-						
Chloroform							
CRQL.							
CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: Common contaminants such as Methylene chloride, Acetone, 2-Butanone and Carbon disulfide that were detected in samples within ten times the associated field blank concentration were qualified as not detected, "U". Other contaminants within five times the field blank concentration were qualified as not detected, "U".	DUALIFIED. ALI thylene chloride, within five times	. RESULTS NOT Acetone, 2-Buta the field blank co	CIRCLED WERE QUALIFIED BY one and Carbon disuifide that were ncentration were also qualified as n	THE FOLLOWING S detected in samples of detected, "U".	TATEMENT: within ten times the	associated field blank	concentration were qualified as not

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LDC #: 1	SDG #:

## VALIDATION FINDINGS WORKSHEET Surrogate Spikes

Ъ Page: Reviewer: 2nd Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y N N/A</u> Were all surrogate %R within QC limits?

If the percent recovery (%R) for one or more surrogates was out of QC limits, was a reanalysis performed to confirm samples with %R out of outside of criteria? ≤

		юна;			-	
#	Date	Sample ID	Surrogate	%Recovery (Limits)	nits)	Qualifications
		F8 P2 10000 - 12	BFB	117	( 29-1155	J+/Palet
					(	
					)	
					( )	
					(	
					· · · · · · · · · · · · · · · · · · ·	
					(	
					)	
	-				(	
					( )	
					(	
					(. )	
					( )	
					( )	
					( )	
					. (	
SMC1 ( SMC2 ( SMC3 ( SMC4 (	TOL) = Toluene BFB) = Bromof DCE) = 1,2-Dicl DFM) = Dibrom	SMC1 (TOL) = Toluene-d8 <u>QC Limits (Soil)</u> SMC2 (BFB) = Bromofluorobenzene 74-121 SMC3 (DCE) = 1,2-Dichloroethane-d4 80-120 SMC4 (DFM) = Dibromofluoromethane 80-120		<u>OC Limits (Water)</u> 88-110 86-115 80-120 86-118		

SUR.1SB

ngs on LDC #: 19091 A SDG #:

Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

đ Page: Reviewer: 2nd Reviewer:

METHOD : GC/MS VOA (EPA SW 846 Method 8260B)

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

N/A AN

ź

Was a MS/MSD analyzed every 20 samples of each matrix? Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

}								
#	Date	di dsw/sw	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		946		(221-6L) hal	( )	( )	v	NO OUAL MAPIN
			Tudomethan		( )	( 07 ) be	~	nigen/en (
					())	( )		
				( )	( )	( )		-
				(	· ·	( )		
				( )	(	()		
		Rinsak 2 MSID	Indo methanc	Anc ( )	( )	12 ( 20 )	More	NO QU ₽
				()	(	( )		
				(	( )	( )		
				( )	)	( )		
	-			( )		()		
ł					(	( )		
				( )	(	( )		
				( )		( )		
				( )				
	-			(	_	( )		
				( ) ·		( )		
				()	)	( ) (		
		Compound	und		Limits (Soil)	RPD (Soil)	QC Limits (Water)	RPD (Water)
	Н	1,1-Dichloroethene		29-1	59-172%	≤ 22%	61-145%	<u> </u>
	ы.	Trichloroethene		62-1	62-137%	≤ 24%	71-120%	≤ 14%
		Benzene		66-1	66-142%	<u>&lt;</u> 21%	76-127%	<u>&lt;</u> 11%
	Ю.	Toluene		59-1	59-139%	≤ 21%	76-125%	<u>&lt;</u> 13%
	DD.	Chlorobenzene		-09	60-133%	<u>&lt;</u> 21%	75-130%	<u>&lt;</u> 13%

MSD.1SB

LDC #: [4071 A) SDG #: 140 CO

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

/ of /

Page: \_\_\_\_\_ Reviewer: \_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

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

N/A A/A

-I≯

Was a LCS required? Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

	Ś																							
Qualifications	Hogual Leadi	JU WS/Pin																						
Associated Samples	All worker +	F&F2 00000-125														·								
RPD (Limits)	112 ( 20 )	- ( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	(	()	( )	( )	()	( )	( )	( )	( )
LCSD %R (Limits)	(	181 (45-140)	(	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( · )	( )	( )	( )	( )	(	( )	( )	()	( )	)
LCS %R (Limits)	293( 42-140	) ماما	)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	(	· · · · · · · · · · · · · · · · · · ·	· · ·	(	( )
Compound	Y	lodomethane																						
rcs/rcsd ID	8172125-455																							
Date																								
#																								

LCSLCSD.1SB

LDC #: 190 SDG #: 190	19091 A 1	<b>N</b>	VALIDATION FINDINGS WORKSHEET Internal Standards	SHEET	Page: of Beviewer:
METHOD: GC/N	METHOD: GC/MS VOA (EPA SW 846 Method 8260B)	46 Method 8260B)			
Please see quali	ifications below for a Were all internal sta Were the retention t	all questions answered "N andard area counts within times of the internal stand	Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". X N/A Were all internal standard area counts within -50 to +100% of the associated calibration standard? Y N/A Were the retention times of the internal standards within +/- 30 seconds of the retention times of the	ifications below for all questions answered "N". Not applicable questions are identified as "N/A". Were all internal standard area counts within -50 to +100% of the associated calibration standard? Were the retention times of the internal standards within +/- 30 seconds of the retention times of the associated calibration standard?	n standard?
# Date	Sample ID	Internal Standard	Area (Limits)	RT (Limits)	Qualifications
	2	HACK	225871-1E1181) 0862L1	22) []	d/rn/
	n		180609 ( 1 )		
	Ŧ	->	( ) 656121		
	ک		168365 ( J )		
					Y, WY with see
-					
(BEM) = Bromochloromethane (DFB) = 1,4-Difluorobenzene (CBZ) = Chlorobenzene-d5	ē	(PFB) = Pentafluorobenzene (4DCB) = 1,4-Dichlorobenzene-d4 (2DCB) = 1,2-Dichlorobenzene-d4	(FBZ) = Fluorobenzene 4		

INTST.1SB

## Volatile Internal Standards

Fluorobenzene	Chlorobenzene-d5 1,1,1,2-Tetrachloroethane 1,2-Dibromoethane 1,3-Dichloropropane 1-Chlorobenzene Dibromochloromethane Ethylbenzene m,p-Xylene o-Xylene Styrene Tetrachloroethene 1,1,2-Trichloroethane To Wene	1,4-Dichlorobenzene-d4 1,1,2,2-Tetrachloroethane - 1,2,3-Trichlorobenzene- 1,2,3-Trichloropropane - 1,2,4-Trichlorobenzene- 1,2-Dichlorobenzene - 1,2-Dibromo-3-chloropropane - 1,3-5-Trimethylbenzene - 1,3-5-Trimethylbenzene - 1,4-Dichlorobenzene - 2-Chlorotoluene - Bromobenzene - Hexachlerobutacliene -
Chloroethane Chloroethane cis-1,2-Dichloroethene cis-1,3-Dichloropropene Dibromomethane Dichlorodifluoromethane Methylene chloride Methyl-tert-butyl ether 2-Butanone Frichloroethene Foluene rans-1,2-Dichloroethene rans-1,3-Dichloropropene richlorofluoromethane finyl chloride Carborn Dis Wiffide	trans-1.3-Dichloropropene 2-Nitropropane 4-Methyl-2-pentanone 2-Hexanone Dimethyl disultide Xylones (total)	Isopropyibenzene r Mathyl isobutyl ketene n-Butylbenzene - n-Propyibenzene - Napithalene p-Isopropyitoluene, p- Cymene sec-Butylbenzene - tert-Butylbenzene - 1, 3, 5 - Trichloro ben zene Nona nal Bromform r

Iodomethane

Acetonitrile Yinyi Acetate 1,1,2-Trichloror1,1,2-Trifluoroethane Ethanol 3,3-Dimethylpentane 2,3-2,2-2,4-3-Trimethylbutane 3-Ethylpentane 2-Methylhtxane 3-

Test America ERM/BKC

Heptane

1,2-Dichloro ethene (total)

LDC #: 19091 A SDG #: eu coner

## VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

∕ of Page: Reviewer: 2nd Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

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

 $\label{eq:RFF} RFF = (A_x)(C_x)(A_x)(C_x) \\ average RFF = sum of the RRFs/number of standards \\ %RSD = 100 \ ^{(S/X)} \\ \end{cases}$ 

A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound, S = Standard deviation of the RRFs X = Mean of the RRFs

A<sub>a</sub> = Area of associated internal standard C<sub>b</sub> = Concentration of internal standard

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (	RRF ( بری std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
-	1001	6 90 6 9	Vinyl chloride		0.34552	0.33147	7747	5:136	5.136
			Ethyl Benzerternal standard)	1604-2	2.30991	2. 19908	2.19908	b.13989	6-139
			1.2 - Di on lo moleunzene	1.20993	66462.1	1.28078	3202-1	5.32452	<del>رد.</del> ک
7	ICAL-XBRC	6 (2 68	2, 2 - Pine fler (perference) (1st interval standard)	0. 52673	52672	650250	62025.0		4.99617
			(2nd internal standard)						
			(3rd internal standard)						
m			(1st internal standard)						
			(2nd internal standard)						
			(3rd internal standard)						
4			(1st internal standard)						
			(2nd internal standard)						
			(3rd internal standard)						

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 <u>results.</u>

LDC # 19091A1 SDG #: ALL COURS

## **Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

ç Page: Reviewer: 2nd Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

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 =  $(A_x)(C_n)/(A_x)(C_x)$ 

 $A_{h} = Area$  of associated internal standard  $C_{h} = Concentration of internal standard$ 

2 4 4						Reported	Recalculated	Reported	Recalculated
XcAL231Lb $l_1 l_2 l_3 c_3$ $v_1 v_3 l_3 c_3 c_3 c_3 c_3 c_3 c_3 c_3 c_3 c_3 c$	*	Standard ID	Calibration Date	Compound (Reference internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	%D	۵%
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	-	XCAL2316	6/12/058	Viny Chlonde	0.33747	0.30844	14802-0	8.60390	8.6039
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$				Ethur (2nd internal standard)	2.1990X	うしっしたって	2-3701L	SLAOB-L	7.50675
(1st internal standard)       (1st internal standard)         (2nd internal standard)       (2nd internal standard)         (2nd internal standard)       (1st internal standard)         (1st internal standard)       (1st internal standard)         (2nd internal standard)       (1st internal standard)         (2nd internal standard)       (1st internal standard)         (2nd internal standard)       (1st internal standard)				11.4 - 0.05	1.28078	1-38717	1-38777	21855.2	8-3537
	2			(1st internal standard)		-			
				(2nd internal standard)					
				(3rd internal standard)					
	ы			(1st internal standard)					
				(2nd internal standard)					
				(3rd internal standard)					
(3rd internal standard) (3rd internal standard)	4			(1st internal standard)					
(3rd internal standard)				(2nd internal standard)					
				(3rd internal standard)					

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

LDC #: 1909 (A) SDG #: <u>su cour</u>

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page:_	<u>of</u>
Reviewer:	<u>*7</u>
2nd reviewer:	-V
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## METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:  $\# \mathcal{V}$ 

Percent Percent Surrogate Recovery Recovery Recalculated Surrogate Percent Spiked Found Reported Difference 53.0191 106 υ 50.0 106 Toluene-d8 112 55. 9784 Bromofluorobenzene 112 119 59.4200 1,2-Dichloroethane-d4 119 110 55.0604 Dibromofluoromethane 110

## Sample ID:\_\_\_\_\_

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene	·····				
1,2-Dichloroethane-d4	······································				
Dibromofluoromethane					

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8					
Bromofluorobenzene					
1,2-Dichloroethane-d4					
Dibromofluoromethane					

LDC #: 19091 A

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

5 Page: Reviewer: 2nd Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

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:

MSDC = Matrix spike duplicate percent recovery

MSC = Matrix spike percent recovery

SC = Sample concentration

% Recovery = 100 \* (SSC - SC)/SA Where: SSC = Spiked sample concentration SA = Spike added

RPD = I MSC - MSDC I \* 2/(MSC + MSDC)

MS/MSD sample: 74 8

	. w	Spike	Sample	Spiked Sample	ample	Matrix Spike	Spike	Matrix Spike Duplicate	Duplicate	W	USM/SM
	≺ .	dded	Concentration	Concentration	ration						
Compound	7) 	W BY	(mx 12)	( m	AL I	Percent Recovery	ecovery	Percent Recovery	ecovery		RPD
	WS	1 Msp	0	D SM	U MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
1,1-Dichloroethene	039	6.49 039	dn	Ŀ?	1.4	1012	901	01	911	м О	3,0
Trichloroethene				13,4	C).4L	113	113		N I	C Á	7.0
Benzene				66.2	LS. 2	201	102	C101	100	e (	1.6
Toluene				68.7	67.7	901	901	101	201	1-10	- <sup>-</sup>
Chlorobenzene	~		~	0.99	4.21	101	La	101	101	68.0	

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

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## VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

б Х Reviewer:\_\_ Page: 2nd Reviewer:

METHOD: GC/MS VOA (EPA SW 846 Method 8260B)

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

Where: SSC = Spiked sample concentration SA = Spike added LCS = Laboraotry control sample percent recovery

RPD = | LCS - LCSD | \* 2/(LCS + LCSD)

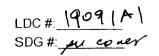
LCS ID: 816 4446-LES

LCSD = Laboratory control sample duplicate percent recovery

	Spike	, e	Spiked S	ample		Cs Cs		q		CS/LCSD
Compound	Addec ( US	ed/kg	Concentration (バテルタイ	Iration 14X	Percent Recovery	tecovery	Percent Recovery	ecoverv	Cax	c
		1 CSD	1 CS	1 CSD	Renorted	Bacalc	Donot	Baada	11	
1,1-Dichloroethene	50. U	WА	0.84	NA	96	96				Recalculated
Trichloroethene			53.0		901	901				
Benzene			21.5		201	102				
Toluene			52.2		hol	101				
Chlorobenzene	Å	Ý	که،ک	-	101	101	54			
-										
		4								
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	Y Control San	nple findings	worksheet for	list of qualifi	cations and a	ssociated sa	mples when re	ported result	s do not agree wi	thin 10.0% of the

LCSCLC.1SB

recalculated results.



only.

## VALIDATION FINDINGS WORKSHEET **Sample Calculation Verification**

Page:_	<u>/_</u> of_	_/
Reviewer:_ 2nd reviewer:_	- P.	$\overline{\mathcal{V}}$

## METHOD: GC/MS VOA (EPA SW 846 Method 8260B)



Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds agree within 10.0% of the reported results?  $M = 3/\sqrt{6}$ I

Example:

=

Conce	ntratio	$n = \frac{(A_{\downarrow})(I_{\downarrow})(DF)}{(A_{\omega})(RRF)(V_{\omega})(\%S)}$
A,	=	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
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
RRF	=	Relative response factor of the calibration standard.
V <sub>o</sub>	=	Volume or weight of sample pruged in milliliters (ml) or grams (g).
Df	=	Dilution factor.
%S	=	Percent solids, applicable to soils and solid matrices

Sample I.D. #2 . \_\_\_\_\_ Chloro jorm

 $\begin{array}{c} \text{Conc.} = (55801) (50) (\\ 5612188) (0.58039 0.3) (\\ 0.31) (0.58039 0.3) (\\ 0.32) (\\ 0$ 

12 ug / kg

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

## Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: BRC Tror	ox Parcel F
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Collection Date: June 10, 2008

LDC Report Date: July 23, 2008

Matrix: Soil

Parameters: Semivolatiles

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

## Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

## Introduction

NUMBER AND A DESCRIPTION OF A DESCRIPTIO

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8270C for Semivolatiles.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XVI.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals.

All ion abundance requirements were met.

## III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 15.0% for each individual compound and less than or equal to 30.0% for calibration check compounds (CCCs).

In the case where %RSD was greater than 15.0%, the laboratory used a calibration curve to evaluate the compound. All coefficients of determination ( $r^2$ ) were greater than or equal to 0.990.

For the purposes of technical evaluation, all compounds were evaluated against the 30.0% (%RSD) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

Average relative response factors (RRF) for all semivolatile target compounds and system performance check compounds (SPCCs) were greater than or equal to 0.05 as required with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid n-(Hydroxymethyl)phthalimide	0.01422 (≥0.05) 0.04408 (≥0.05)	All samples in SDG F8F110177	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A

## IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences (%D) between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0% for calibration check compounds (CCCs).

For the purposes of technical evaluation, all compounds were evaluated against the 25.0% (%D) National Functional Guideline criteria. Unless noted above, all compounds were within the validation criteria.

The percent difference (%D) of the second source calibration standard were less than or equal to 25.0% for all compounds.

All of the continuing calibration RRF values were within method and validation criteria with the following exceptions:

Date	Compound	RRF (Limits)	Associated Samples	Flag	A or P
6/18/08	Phthalic acid	0.01330 (≥0.05)	All samples in SDG F8F110177	J (all detects) UJ (all non-detects)	A
	n-(Hydroxymethyl)phthalimide	0.04331 (≥0.05)		J (all detects) UJ (all non-detects)	

### V. Blanks

Method blanks were reviewed for each matrix as applicable. No semivolatile contaminants were found in the method blanks.

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Internal Standards

All internal standard areas and retention times were within QC limits.

### XI. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XII. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XIII. Tentatively Identified Compounds (TICs)

All tentatively identified compounds were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XIV. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XV. Overall Assessment of Data

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

### **XVI. Field Duplicates**

No field duplicates were identified in this SDG.

### BRC Tronox Parcel F Semivolatiles - Data Qualification Summary - SDG F8F110177

SDG	Sample	Compound	Flag	A or P	Reason
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Phthalic acid n-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Initial calibration (RRF)
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Phthalic acid n-(Hydroxymethyl)phthalimide	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Continuing calibration (RRF)

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### BRC Tronox Parcel F Semivolatiles - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Semivolatiles - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #:	VALIDATION COMPLETENESS WORKSHEET	Date: 7/19/08
SDG #:F8F110177	Level III/IV	Page: / of /
Laboratory: Test America	-	Reviewer: 🗶

2nd Reviewer:

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270C)

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
١.	Technical holding times	4	Sampling dates: 6 10 08
11.	GC/MS Instrument performance check	4	
111.	Initial calibration	SW	% PND, (2 20.99D
IV.	Continuing calibration/ICV	SW	161 = 25
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	TSB - GJ -08-10
VIII.	Laboratory control samples	SA	KCS
IX.	Regional Quality Assurance and Quality Control	N	
<b>X</b> .	Internal standards	4	
XI.	Target compound identification	4	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	Ą	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	4	Not reviewed for Level III validation.
XIV.	System performance	Ą	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	$\sim$	

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 Level IV validation

	sol				 	
1	TSB-FR-02-02-20'	11	8168439	21	 31	
2	TSB-FR-02-02-30'**	12		22	 32	
3	TSB-FJ-02-02-10'**	13		23	 33	
4	TSB-FJ-02-02-20'**	14		24	34	
5	TSB-FJ-02-02-30'	15		25	35	
6	F8 F160000 - 439	16	8168439	26	36	
7		17		27	 37	
8		18		28	 38	
9		19		29	39	
10		20		30	40	

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### VALIDATION FINDINGS CHECKLIST

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

### Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
A CONTRACT CONTRACTS				
All technical holding times were met.		ł		
Cooler temperature criteria was met.	- 19-12-45+ 2-1			
IN SAVAS AND INTERACTION RECEIPTED				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?				
Did the laboratory perform a 5 point calibration prior to sample analysis?		ţ	ļ	
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Was a curve fit used for evaluation?				· · · · · · · · · · · · · · · · · · ·
Did the initial calibration meet the curve fit acceptance criteria of $\geq$ 0.990?				
Were all percent relative standard deviations (%RSD) $\leq$ 30% and relative response factors (RRF) $\geq$ 0.05?	the		1	
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?				
Were all percent differences (%D) $\leq$ 25% and relative response factors (RRF) $\geq$ 0.05?	W	$\checkmark$		
Was a method blank associated with every sample in this SDG?	$\angle$			
Was a method blank analyzed for each matrix and concentration?	$\square$			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			/	
Were all surrogate %R within QC limits?				
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?				- · ·
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?			2	
A. ABOL MINABOL SCIENCE				
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.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	$\leq$			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
ALL CRETCHLY ZI NEW SCHOOLS				
Was an LCS analyzed for this SDG?				

### VALIDATION FINDINGS CHECKLIST

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	1	T	1	1
Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	$\downarrow \checkmark$	<b>F</b>	ŀ	· · · · · · · · · · · · · · · · · · ·
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/	F	
121 Real the Cambin discussion of Grand Cambra		and a state of the second		
Were performance evaluation (PE) samples performed?			/	Ť.
Were the performance evaluation (PE) samples within the acceptance limits?				-
CONCURD STEMPTICS				
Were internal standard area counts within -50% or +100% of the associated calibration standard?		-		
Were retention times within + 30 seconds from the associated calibration standard?	/			
XXX TEMEL AND DECIMAL PERMINENCE				
Were relative retention times (RRT's) within ± 0.06 RRT units of the standard?				
Did compound spectra meet specified EPA "Functional Guidelines" criteria?				
Were chromatogram peaks verified and accounted for?				
XII SCEDESTICATERINE (NOVATING				
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?		/		
STD TEAM ELEMENDER EDDEMART Pro-				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	/	-		
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?		-		
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?		_		
System performance was found to be acceptable.				
Overall assessment of data was found to be acceptable.	T	· ·		
Field duplicate pairs were identified in this SDG.		7		
Target compounds were detected in the field duplicates.				
Field blanks were identified in this SDG.			ar an In	an a
		4		
Target compounds were detected in the field blanks.			1	

VALIDATION FINDINGS WORKSHEET

METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

l

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachloropheno!**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroanitine	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol⁺	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	li. 4-Nitrophenoi⁺	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene*⁺	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenoi**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	000. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methyiphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenoi**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	00. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	.'H
M. isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	WV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

Notes:\* = System performance check compound (SPCC) for RRF, \*\* = Calibration check compound (CCC) for %RSD.

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### VALIDATION FINDINGS WORKSHEET **Initial Calibration**

ō Reviewer: 2nd Reviewer: Page:

METHOD: GC/MS BNA (EPA SW 846 Method 8270)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A"  $\frac{1}{N}$  N/A. Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?

Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?

Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? Did the initial calibration meet the acceptance criteria?

N N/A

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	Qualifications			Jun A																			
	Associated Samples			AIL+BIK	1 ·																		
nd ≥0.05 RRF ?	Finding RRF (Limit: <u>&gt;</u> 0.05)		o. OHIV	0-01354	191400	0.04400														•			
criteria of ≤30 %RSD a	Finding %RSD (Limit: <u>≤</u> 30.0%)				m)	Ο															•		
s within the validation o	Compound			Phthalic Acid	N-(hydroxymeth	phythalimide NO																	
Were all %HSUs and HHFs within the validation criteria of ≤30 %RSD and ≥0.05 RRF ?	Standard ID			JICAL SPEC	-																		
Y (N W/A W	Date			60819	-																		
Ĩ	#																						

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### VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

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METHOD: GC/MS BNA (EPA SW 846 Method 8270) Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". N NA Was a continuing calibration standard analyzed at least once every 12 hours of sample analysis for each instrument? N N/A Were percent differences (%D) and relative response factors (RRF) within method criteria for all CCC's and SPCC's ?

- ? Finding RRF (Limit: >0.05) 0. 04 33/ 0. 04 33/	Finding %D (Limit: ≤25.0%)	a of <25 %D and >0.05 RRF Finding %D (Limit: <25.0%)		0.0/330 A1/2 B1/2 J/4)					
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### VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

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METHOD: GC/MS BNA (EPA SW 846 Method 8270)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>X N N/A</u> Was a LCS required? <u>Y N N/A</u> Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within t

Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

	1010						T		T					Ī											
Qualifications	NO ONAL IN																								
Associated Samples	A 11+B1K		-																						
RPD (Limits)	( )	()	(	)	<b>`</b>	)	( )	( )	( )	( )	( )	(	-		(	· · ·	)	•	( )	~	( )	(	( )	-	(
LCSD %R (Limits)	( · · )	( )	( )	( )	( )	( )	( )	( )	( )	( )	(	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	)	<b>^</b>	( )	( )
 LCS %R (Limits)	19 (54-90)	(	( )	(	( )	( )	( )		( )	( )	( )	(	(	~	<b>^</b>	( )	(	( )	( )	•	( )	( )	( )	( )	)
Compound	HH																								
LCS/LCSD ID	8168439-105																								
Date																									
*																									

LCSLCSD.2S

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### Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

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

 $\label{eq:RFF} RFF = (A_x)(C_x)/(A_y)(C_x)$  average RRF = sum of the RRFs/number of standards %RSD = 100 \* (S/X)

A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound, S = Standard deviation of the RRFs,

A<sub>is</sub> = Area of associated internal standard

|--|

l standard	
C <sub>is</sub> = Concentration of internal	X = Mean of the PPCs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	RRF (50 std)	RRF ( CO std)	Average RRF (initial)	Average RRF (initial)	%RSD	%RSD
-	1CA1-J	60200	Phenol (1st internal standard)	1.637853	1.87853		1528.1	CITO-1	1.070
		•	Naphthalene (2nd internal standard)	1.09450	1.09438		1.1090	1.329	1-223
			Fluorene (3rd internal standard)	1-41-13	1.41778	1.41229	6211-	0.573	0.573
			Pentachlorophenol (4th internal standard)	6.202h0	arora	0.19634	0.19634	10. 255	10.25
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.90763	0.90163	0.86543	0. 26343	9.524	d rav
			Benzo(a)pyrena (6th internal standard)	1-13808	1.13808	1. 11.02	1.11.182	194.0	1961
~	rear BRIX	7 1/18	Act Top Nerve ( Bhanol (15) internal standard)	01212.0	92615.0	0.51274	0-51274	0.71511	14120
		/ 1 /	Naphthalene (2nd internal standard)						
	ICAL SPEC	9119 C	Rueronadord interdal standard) phythali mide	0.04162	0. pyllo2	0.04408	Sopto.O	8.41339	N 41320
			Pentachlorophenol (4th internal standard)						1 2 1 2
			Bis(2-ethylhexyl)phthalate (5th internal standard)						
			Benzo(a)pyrene (6th internal standard)						
m			Phenol (1st internal standard)						
			Naphthalene (2nd internal standard)						
			Fluorene (3rd internal standard)						
			Pentachlorophenol (4th internal standard)						
			Bis(2-ethylhexyl)phthalate (5th internal standard)						
			Benzo(a)pyrene (6th internal standard)						

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

1406142 Les cores SDG #:~/ LDC #:

### **Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

j Page: 2nd Reviewer: Reviewer:

METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

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\_x)/(A\_x)(C\_x)

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF Where:

A<sub>x</sub> = Area of compound, C<sub>x</sub> = Concentration of compound,

 $A_{is}$  = Area of associated internal standard  $C_{is}$  = Concentration of internal standard

Standard)         Average RRF (initial)         RRF (initial)         RRF (cc)         RRF (cc)           1.55537         1.87174         1.87174         1.87174           1.10361         1.10135         1.10135         1.10135           1.11229         1.378537         0.20370         0.20370           1.1122         0.141227         0.20370         0.20370           1.1122         0.1507         1.11507         1.11507           MhalfmML         0.51214         0.52185         0.53185           1.11122         0.14351         0.04351         0.04351           1.11122         0.04351         0.04351         1.11507           1.1112         0.04351         0.04351         1.11507           1.1112         0.04351         0.04351         0.04351           1.01         0.04351         0.04351         1.11507           1.01         0.04351         0.04351         1.11507           1.01         0.04351         0.04351         1.11507           1.01         0.04351         0.04351         1.11507           1.01         0.04351         0.04351         1.11507           1.01         0.01351         0.04351         1.11507						Reported	Recalculated	Reported	Recalculated
JCALSIGS       C.IL.8 00       Prenol (1st internal standard)       L:K5537       L:R114       L:K114         Naphthalene (2nd internal standard)       1:V0301       1:V035       1:10135       1:10135         Rusch       Pentachtorophenol (4th internal standard)       1:V0301       1:10135       1:10135         Pentachtorophenol (4th internal standard)       0:V6343       0:20370       0:20370         Bis(2-ettryfnexy0phthalate (5th internal standard)       0:V6343       0:X708K       0:20370         JCAL5191       6/18/00       Amarciapases of th internal standard)       0:V6343       0:S2185       0:S2185         JCAL5191       6/18/00       Amarciapases of th internal standard)       0:V63443       0:S2185       0:S2185       0:S2185         JCAL5191       6/18/00       Amarciapases of th internal standard)       0:V63443       0:S2185       0:S2185       0:S2185         JCAL5191       6/18/00       0:S1214       0:S2185       0:S2185       0:S2185       0:S2185         JCAL5191       6/18/00       0:S1214       0:S2185       0:S2185       0:S2185       0:S2185         JCAL5191       6/18/00       0:S2185       0:S2185       0:S2185       0:S2185       0:O4333         JCAL5191       6/18/00       0:S214	*		Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF (CC)	RRF (CC)	Q%	Q°/
Nephthalene (2nd internal standard)1. UOGO11. UOI351. UO135Fluorene (3rd internal standard)1. H12201. 33 & 111. 33 & 11Pentachlorophenol (4th internal standard)0. 19L230. 203300. 20330Bis(2-ethythesy()phthalate (5th internal standard)0. 19L320. 203300. 20330JCAL S194b0. 18 & 0. 87 & 0. 87 & 0. 87 & 0. 88 & 108 & 0. 87 & 0. 87 & 0. 88 & 108 & 0. 87 & 0. 88 & 108 & 0. 88 & 0.	-	JCALSPOS		Phenol (1st internal standard)	1-85537	HL1 L81	+L123.1	0.8270	0.87310
Fluorene (3rd internal standard) $1.41229$ $1.33801$ $1.33801$ Pentachlorophenol (4th internal standard) $0.194534$ $0.202310$ $0.23390$ Bis(2-ethythexy)phthalate (3th internal standard) $0.194534$ $0.202310$ $0.23390$ JCAL519b $  y 03$ Rexard pursens (3th internal standard) $0.11122$ $1.11122$ $1.11507$ JCAL519b $  y 03$ Rexard pursens (3th internal standard) $0.51214$ $0.223557$ $0.531857$ JCAL5197 $  y 03$ Rexard pursens (3th internal standard) $0.51214$ $0.523557$ $0.531857$ JCAL5197 $  y 03$ Rexard pursens (3th internal standard) $0.51214$ $0.523557$ $0.531857$ JCAL5197 $  y 03$ Rexard pursens (3th internal standard) $0.51214$ $0.523557$ $0.531857$ JCAL5197 $  y 03$ Rescaled of internal standard) $0.51214$ $0.523557$ $0.531857$ JCAL5197 $  y 03$ Rescaled of internal standard) $0.51214$ $0.523557$ $0.531857$ Partachlorophenol (4th internal standard) $0.51214$ $0.521557$ $0.504333$ Rescaled by the ethologomenol (3th internal standard) $0.51214$ $0.521557$ $0.504333$ Rescaled by the ethologomenol (3th internal standard) $0.51214$ $0.521557$ $0.504335$ Rescaled by the ethologomenol (3th internal standard) $0.51214$ $0.521557$ $0.504335$ Rescaled by the ethologomenol (3th internal standard) $0.51214$ $0.521557$ $0.504335$ Rescaled by the etholoformal standard)Rescaled resc				Naphthalene (2nd internal standard)	10001.1	1.10135	1.10135	0.69070	0,000,0
Pentachiorophenol (4th internel standard) $c.13L^{3}$ H $c.20370$ $a.20370$ Bis(2-ethythesyl)prthalate (5th internal standard) $c.13L^{3}$ H $c.20310$ $a.83704$ JCAL519b $                                    $				Fluorene (3rd internal standard)	1.41229	1.39 80	1. 3980)	aso10.1	BSOID-101-1
Bis(2-ethylhexyl)phthalate (5th internal standard)     0. Kb3 43     0. Kb3 43     0. Kb3 7     Ks310K       JCAL 519L     b ly 0B     Rever(1) thermal standard)     0. S1214     0. S2185     0. S2185       JCAL 519L     b ls 0C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S2185       JCAL 5197     b ls 0C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S1185       JCAL 5197     b ls 0C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S2185       JCAL 5197     b ls 0C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S1857       JCAL 5197     b ls 0C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S1857       JCAL 5197     b ls 20C     Rever(1) thermal standard)     0. S1214     0. S2185     0. S1857       Pentachiorophenol (4th internal standard)     Pentol (1st internal standard)     0. S1214     0. S2185     0. S1857       Revo(a) Naphthalate (5th internal standard)     Phenol (1st internal standard)     0. S1214     0. S2185     0. S1857       Revo(a) Naphthalete (2nd internal standard)     Phenol (1st internal standard)     0. S1214     0. S1333     0. S1857       Revo(a) Naphthalete (3th internal standard)     Phenol (1st internal standard)     0. S1214     0. S1333     0. S1333   <				Pentachiorophenol (4th internal standard)	0.19634	01502.0	0.20330	3.74980	3-74980
JCAL5191b     L [1]     L [1]     L [1]     L [1]       JCAL5191b     L [1]     DO     Amonth (1st theme istandard)     D. S [12]     L [1]     D. S [12]       JCAL5191b     L [1]     DO     Amonth (1st theme istandard)     D. S [12]     D. S [12]     D. S [12]       JCAL5191b     L [1]     DO     Amonth (1st theme istandard)     D. Muth alt multicity     D. S [12]     D. S [12]       JCAL5191b     L [1]     L [1]     DO     Amonth altered (3nd internal standard)     D. Muth alt multicity     D. D. DU       Pentachlorophenol (4th internal standard)     Renz (c)phrane (5th internal standard)     D. D. DU     D. D. DU     D. D. DU       Renz (c)phrane (6th internal standard)     Phenol (1st internal standard)     D. D. DU     D. D. DU     D. D. DU       Maphthalene (2nd internal standard)     Maphthalene (2nd internal standard)     D. D. DU     D. D. DU     D. D. DU       Phenol (1st internal standard)     Fluorene (3th internal standard)     D. D. DU     D. D. DU     D. D. DU       Renz (c)phrenol (4th internal standard)     Berz (c)thytheolyl)phthalate (5th internal standard)     D. D. DU     D. D. DU       Renz (c)phrenol (4th internal standard)     Berz (c)thytheolyl)phthalate (5th internal standard)     D. D. DU     D. D. DU       Berz (c) phrenol (4th internal standard)     D. D. DU     D. DU </th <th></th> <td></td> <td></td> <td>Bis(2-ethylhexyl)phthalate (5th internal standard)</td> <td>0.86343</td> <td>23062.0</td> <td>9801-8·0</td> <td>0.86222</td> <td>0.6222</td>				Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	23062.0	9801-8·0	0.86222	0.6222
JCALS19L       L<				Benzo(a)pyrene (6th internal standard)	1. 11122	1-WS07	Las11.1	0221250	CN2P2-0
JCAL E 197       6 118 00       Maphthalene (2nd internal standard)       0.0433)       0.0433)         Pentachlorophenol (4th internal standard)       Pentachlorophenol (4th internal standard)       0.0433)       0.0433)         Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)       0.0433)       0.0433)         Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)       0.0433)       0.0433)         Phenol (1st internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)       0.0433       0.0433         Phenol (1st internal standard)       Phenol (1st internal standard)       0.0433       0.0433         Phenol (1st internal standard)       Phenol (1st internal standard)       0.0433       0.0433         Phenol (1st internal standard)       Phenol (1st internal standard)       0.04133       0.0433         Phenol (1st internal standard)       Phenol (1st internal standard)       0.04133       0.0433         Pentachlorophenol (4th internal standard)       Pentachlorophenol (4th internal standard)       0.04133       0.04133         Pentachlorophenol (4th internal standard)       Pentachlorophenol (4th internal standard)       0.01410       0.014133         Benzo(a)bortene (6th internal standard)       Pentachlorophenol (4th internal standard)       0.01410       0.	2		6 18 08	Remoti (1st internal standard)	0.51274	0.52185	0.52165	いココレンク	1.77032
コーベビートリリ、6/18/00       和iscenet (3d internal standard) へかけっくは (4th internal standard)       の・ロリマンS       0・ロリマンS       0・ロリマンS       0       0・ロリマンS       0				Naphthalene (2nd internal standard)					
Pentachlorophenol (4th internal standard)       Pentachlorophenol (4th internal standard)         Bis(2-ethythexyl)phthalate (5th internal standard)       Bis(2-ethythexyl)phthalate (5th internal standard)         Phenol (1st internal standard)       Phenol (1st internal standard)         Naphthalene (2nd internal standard)       Phenol (1st internal standard)         Phenol (1st internal standard)       Phenol (1st internal standard)         Pentachlorophenol (4th internal standard)       Phenol (1st internal standard)         Barzo(a)byrene (6th internal standard)       Phenol (1st internal standard)		1419720	6 18 02	Autorene (3)d internal standard) O hith a U my	LL D. OYYOX	0.0432)	( ~~ 0.0	P125 L-1	1.12.819
				Pentachlorophenol (4th internal standard)					1.201
				Bis(2-ethylhexyl)phthalate (5th internal standard)					
		· · ·		<u>Renzo(a)pyrene (6th internal standard)</u>					
Naphthalene (2nd internal standard)       Fluorene (3rd internal standard)       Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)       Benzo(a) bytene (6th internal standard)	٣			Phenol (1st internal standard)					
Fluorene (3rd internal standard)     Fluorene (3rd internal standard)       Pentachlorophenol (4th internal standard)     Bis(2-ethylhexyl)phthalate (5th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)     Bis(2-ethylhexyl)phthalate (5th internal standard)				Naphthalene (2nd internal standard)					
Pentachlorophenol (4th internal standard)       Bis(2-ethylhexyl)phthalate (5th internal standard)       Benzo(a)pyrene (6th internal standard)				Fluorene (3rd internal standard)					
Bis(2-ethylhexyl)phthalate (5th internal standard) Benzo(a)pwrene (5th internal standard)				Pentachlorophenol (4th internal standard)					
Benzo(a) bytene (6th internal standard)				Bis(2-ethylhexyl)phthalate (5th internal standard)					
				Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results. LDC #: 1909142

SDG #: <u>ere cone</u>

### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: / of / Reviewer: // 2nd reviewer: //

### METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270C)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID:  $\gamma$ 

Percent Percent Surrogate Surrogate Found Recovery Recovery Percent Spiked Reported Recalculated Difference Nitrobenzene-d5 50 35.2132 O 70 70 2-Fluorobiphenyl 37.0385 74 74 Terphenyl-d14 36.164 72 12 V 52.8544 Phenol-d5 75 70 70 69 2-Fluorophenol 52.0442 69 2,4,6-Tribromophenol 74 55.2829 74 2-Chlorophenol-d4 1.2-Dichlorobenzene-d4

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14				· · · · · · · · · · · · · · · · · · ·	
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

### Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					1
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol		· · ·			
2-Chlorophenoi-d4					
1.2-Dichlorobenzene-d4					

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

Page: of devicements of Control o

METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

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 \* (SSC - SC)/SA

RPD = I MS - MSD I \* 2((MS + MSD)

MS = Matrix spike percent recovery

TSB-GJ-08-16

MS/MSD samples: \_\_\_\_

SSC = Spiked sample concentration SA = Spike added

Where:

MSD = Matrix spike duplicate percent recovery

SC = Sample concentation

Recalculated 2.7 2.2 30 Q かん MS/MSD RPD 3 Ú 2.7 <del>ار</del> 0 2.2 Renorted ۶ کر Г Matrix Spike Duplicate Recalc 67 62 3 Percent Recovery 32 60 Reported 65 6 3 5 5 5 Recalc Percent Recovery 0 20 Matrix Spike 3 10 11 7 Reported 5 201 74 のち F F 2390 26-70 2690 2230 2450 2620 MSD Spiked Sample Concentration <u>}</u> 2730 2760 2460 2300 2490 2640 MS Sample Concentration All Bull 202 3570 Msp Added Spike 0155 ЯN N-Nitroso-di-n-propylamine 4-Chloro-3-methylphenol Compound Pentachlorophenol Acenaphthene Phenol Pyrene

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

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# VALIDATION FINDINGS WORKSHEET

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

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

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

% Recovery = 100 \* (SC/SA

Where: SSC = Spike concentration SA = Spike added

RPD = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS = Laboratory control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 21/0 8439

	S	pike <sup>.</sup>	Sp	Spike		cs		CSD		
Compound	~ ~ ~	Added ( ug/ke)	Concent	Concentration	Percent Recovery	lecoverv	Percent Recovery		ā	
				R I				/ In const	2	
	SOL	1 CSD	L C.S	Icsn	Reported	Recalc	Renorted	Recalc	Renorted	Recalculated
Phenol	3330	<b>0</b> 00/€-1	こうんた	A M	71	F				
N-Nitroso-di-n-propylamine			OLX			12				
4-Chloro-3-methylphenol		_	3500			11				
Acenaphthene			2210		15	75				
Pentachtorophenol			0422		67	67				
Pyrene			2350		cι	al	NR			
				>	-					

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 1909142 SDG #: per cover

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

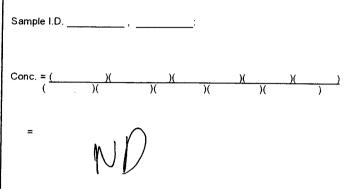
Page: / of / Reviewer: // 2nd reviewer:

### METHOD: GC/MS BNA (EPA SW 846 Method 8270C)



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

Concentration =  $(A_x)(I_x)(V_1)(DF)(2.0)$ Example:  $(A_{is})(RRF)(V_{o})(V_{i})(%S)$ Area of the characteristic ion (EICP) for the compound to Α, = be measured Area of the characteristic ion (EICP) for the specific = A<sub>is</sub> internal standard Amount of internal standard added in nanograms (ng) = ١, ٧, Ξ Volume or weight of sample extract in milliliters (ml) or grams (g). V, = Volume of extract injected in microliters (ul) = Volume of the concentrated extract in microliters (ul) V, = Df = Dilution Factor. %S Percent solids, applicable to soil and solid matrices only. =



### 2.0 = Factor of 2 to account for GPC cleanup

#	Sample ID	Compound	Reported Concentration	Calculated Concentration	Qualification
					duantouton
	· · ·				
			Υ.		

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

Project/Site Name:	BRC Tronox Parcel F
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Soil

Collection Date: June 10, 2008

LDC Report Date: July 22, 2008

Matrix:

Parameters: Chlorinated Pesticides

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

### Sample Identification

TSB-F-02-02-20' TSB-F-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8081A for Chlorinated Pesticides.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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. GC/ECD Instrument Performance Check**

Instrument performance was acceptable unless noted otherwise under initial calibration and continuing calibration sections.

### III. Initial Calibration

Initial calibration of single and multicomponent compounds was performed for the primary (quantitation) column and confirmation column as required by this method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all compounds.

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Standard	Channel	Compound	%D	Associated Samples	Flag	A or P
6/18/08	KCAL081	A	2,4'-DDT	16.2	TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	J- (all detects) UJ (all non-detects)	A

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

The individual 4,4'-DDT and Endrin breakdowns (%BD) were less than or equal to 15.0%.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### V. Blanks

Method blanks were reviewed for each matrix as applicable. No chlorinated pesticide contaminants were found in the method blanks.

No field blanks were identified in this SDG.

### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### VII. Matrix Spike/Matrix Spike Duplicates

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

### VIII. Laboratory Control Samples (LCS)

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

### IX. Regional Quality Assurance and Quality Control

Not applicable.

### X. Pesticide Cleanup Checks

### a. Florisil Cartridge Check

Florisil cleanup was not required and therefore not performed in this SDG.

### **b. GPC Calibration**

GPC cleanup was not required and therefore not performed in this SDG.

### **XI. Target Compound Identification**

All target compound identifications were within validation criteria for samples on which an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XII. Compound Quantitation and Reported CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which an EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel F Chlorinated Pesticides - Data Qualification Summary - SDG F8F110177

SDG	Sample	Compound	Flag	A or P	Reason
F8F110177	TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	2,4'-DDT	J- (all detects) UJ (all non-detects)	A	Continuing calibration (%D)

### BRC Tronox Parcel F

Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: <u>19091A3a</u>	VALIDATION COMPLETENESS WORKSHEET	Date: 7/19/08
SDG #: <u>F8F110177</u>	Level III/IV	Page: / of /
Laboratory: Test America		Reviewer:
		2nd Reviewer:
METHOD: GC Chlorinated P	esticides (EPA SW 846 Method 8081A)	

### METHOD: GC Chlorinated Pesticides (EPA SW 846 Method 8081A)

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
Ι.	Technical holding times	Δ	Sampling dates: 6/10/08
١١.	GC/ECD Instrument Performance Check	Δ	, ,
111.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	SW	1cV = 15
V.	Blanks	Δ	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	75B-GJ-08-10
VIII.	Laboratory control samples	A	409
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification	4	Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs	Δ	Not reviewed for Level III validation.
XIII.	Overall assessment of data	A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

Note:

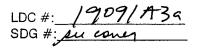
A = Acceptable N = Not provided/applicable SW = See worksheet

ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Level IV validation

1	TSB-FR-02-02-20'	11	F8F160000-164	21	8168164	31	
2	TSB-FR-02-02-30'**	12		22		32	
3	TSB-FJ-02-02-10'**	13		23		33	
4	TSB-FJ-02-02-20'**	14		24		34	
5	TSB-FJ-02-02-30'	15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	



### Method: Pesticides/PCBs (EPA SW 846 Method 8081/8082)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.		ł		
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?	/ /	t		
III. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?				
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		/		
Did the initial calibration meet the curve fit acceptance criteria?			$\lfloor \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$	
Were the RT windows properly established?	/			
Were the required standard concentrations analyzed in the initial calibration?				
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R	/	1		
Were Evaluation mix standards analyzed prior to the initial calibration and sample analysis?	/	-		
Were endrin and 4,4'-DDT breakdowns $\leq$ 15%.0 for individual breakdown in the Evaluation mix standards?				
Was a continuing calibration analyzed daily?		-		
Were all percent differences (%D) $\leq$ 15%.0 or percent recovieries 85-115%?	Ø	A,		
Were all the retention times within the acceptance windows?				
V. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Were extract cleanup blanks analyzed with every batch requiring clean-up?				
Was there contamination in the method blanks or clean-up blanks? If yes, please see the Blanks validation completeness worksheet.			_	
VI. Surrogate spikes				
Were all surrogate %R within the QC limits?	7	_		
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			/	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				

Validation Area	Yes	No	NA	Findings/Comments
VII: 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.				
Was a MS/MSD analyzed every 20 samples of each matrix?	/	-		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII. 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?				
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			<	
Were the performance evaluation (PE) samples within the acceptance limits?				/
X. Target compound identification				
Were the retention times of reported detects within the RT windows?			/	
XI. Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?				
XII. System performance				
System performance was found to be acceptable.	$\square$			
XIII. Overall essessment of data				
Overall assessment of data was found to be acceptable.	$\nearrow$			
KIV, Field duplicates				
Field duplicate pairs were identified in this SDG.		$\square$		
Target compounds were detected in the field duplicates.			7	
∜. Field blanks				
Field blanks were identified in this SDG.		1		
Farget compounds were detected in the field blanks.			7	

LDC #: 1909/733 SDG #: su cone VALIDATION FINDINGS WORKSHEET

METHOD: Pesticide/PCBs (EPASW 846 Method 8081/8082)

A. alpha-BHC	1. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG.
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II.
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	.tt
E. Heptachior	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	IL.
G. Heptachlor epoxide	0. 4,4"DDT	W. Aroclor-1221	Ü	MM.
H. Endosulfan I	P. Methoxychior	X. Aroclor-1232	Ľ	NN.

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Notes:

C:\docs\Work\Pesticides\COMPLST-3S.wpd

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LDC #: 19	SDG #:	~

HPLC

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METHOD:

### VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

5 Page: / Reviewer:

2nd Reviewer:

Please see gualifications below for all questions answered "N". Not applicable questions are identified as "N/A". What type of continuing calibration calculation was performed? \_\_%D or \_\_RPD <u>Y\_N\_NA</u> Were continuing calibration standards analyzed at the required frequencies?

Did the continuing calibration standards meet the %D / RPD validation criteria of <15.0%? N N/A

ever/v only

Were the retention times for all calibrated compounds within their respective acceptance windows? N NA

-	_				_				_		_				1	- 1		Т	Ť	Ť		
Qualifications	J-/WJ/A																					
Associated Samples	4, S																					
RT (limit)	) (	) (	( )	(	( )	( )	( )	( )	) (	] (	( )	( )	)	)	) (	( )	) (	) (	( )	) (	)	( )
%D / RPD (Limit ≤ 15.0)	16.2																					
Compound	2,4-PDT																					
Detector/ Column																						
	KCA1081																					
Date	6/12/08	, ,																				
#																						

LDC #: 19091432 SDG #: 11 conn

### VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

萬 Page: 1 of 2nd Reviewer: Reviewer:

APLC O METHOD: GC

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

CF = A/C average CF = sum

average CF = sum of the CF/number of standards %RSD = 100 \* (S/X)

A = Area of compound, C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	0. 025 std)	CF 01025 std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
	1691	80/91/9	endowhan I chA	530216040	530216040 530216040		04/26015 04/566015	3.148	3-148
			me those chief I	161496680	16149640	029262251 08996,191 p8996, 191	02942251		5/1527
			0						
2			l chB	285001720	orlians polio on Sta	17552576	ZIRESELE ZILEESELE	85 36 28	12596.2 1
			~	44217640	UNALIZAT DAAL 1844	0 75 2622 h	UNE 22 740	6.01883	2 8:0/89
							-		
4									
<u> </u>									

ted samples when reported results do not agree within 10.0% of the recalculated results. õ

LDC # 19091430 SDG #: Rev court

### **Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

∕ of Page: Reviewer: 2nd Reviewer:\_

HPLC METHOD: GC

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

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where: ave. CF = initial calibration average CF CF = continuing calibration CF A = Area of compound C = Concentration of compound

Recalculated	3.7 0-8	2.7		
Reported %D	3.7 0-8	1.0		
Recalculated CF/Conc.	ссv 0. లస్ 0. 0 X J	0.0757		
Reported CF/Conc.	ccv 0.0259 0.0252	0.0XZ		
Average CF(Ical)/	D. D.25			
	compound endesu/pan/ chA mc/hoxychlor/			
Calibration	Late	6/18/08		
	standard ID k сAL06У	2 <u>kcALOXO</u> 6/18/00		
	# - #		6	4

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

LDC	#:	19	091	<u>A</u> 3a
SDG	#:	su	cone	N
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### VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

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Reviewer:	
2nd reviewer:_	
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### METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

### #2 Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	ChA	0.02	0.01839	92	92	0
Tetrachloro-m-xylene DCB	V	4	0.01682	84	84	D
Decachlorobiphenyl						
Decachlorobiphenyl						

### Sample ID:\_\_\_\_\_

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachioro-m-xylene						
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

### Sample ID:\_\_\_\_\_

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachioro-m-xylene						· · · · · · · · · · · · · · · · · · ·
Tetrachioro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

### Sample ID:\_\_\_\_\_

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene						
Tetrachioro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl		······································				·····

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LDC #	SDG #:

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



METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

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

% Recovery = 100\* (SSC-SC)/SA

RPD = I MS - MSD I \* 2/(MS + MSD)

MS/MSD samples:\_\_\_

MSD) MS = Matrix spike percent recovery  $T \leq B - Q - I O Q - I O$ 

MSD = Matrix spike duplicate percent recovery

SC = Concentration

SSC = Spiked sample concentration SA = Spike added

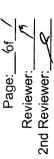
Where:

Compound         ( )           MS         MS         MSD           gamma-BHC         17.7         17.5           4,4'-DDT         人         人		Concentration		Matrix	Matrix Spike	Matrix Spik	Matrix Spike Duplicate	W	MS/MSD
MS MS	•	_	itration )	Percent	Percent Recovery	Percent	Percent Recoverv		RPD
HC 17.7		WS	dsm	Reported	Recalc	Denortod		1	
<i>A</i>		15.10	4 21	3	64/		C/J	Keported	Recalculated
				00	90	10	Ø	2,5	) i
		12-6	10:0	88	22	73	93	4.4	2, 2, 2
	· ·					-			

Comments: Refer of Matrix Spike/Matrix Spike Duplicates findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0%

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LDC #: 19091 A34	SDG #: put come

## aboratory Control Sample/Laboratory Control Sample Duplicate Results Verification VALIDATION FINDINGS WORKSHEET



METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)

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

% Recovery = 100\* (SSC-SC)/SA

RPD = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS = Laboratory control sample percent recovery

SSC = Spiked sample concentration SA = Spike added

Where:

LCSD = Laboratory control sample duplicate percent recovery

SC = Concentration

LCS/LCSD samples: *% | しょ)し* オピン

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	ds	bike	Spike	d Sample		LCS		LCSD		LCS/LCSD
Compound	ž )	ug /48	Conc	( US / PS	Percent	Percent Recovery	Percent	Percent Recoverv		Caa
	n SD1	LCSD	rcs	rcsD	Reported	Recalc.	Reported	Recal	Deported	
gamma-BHC	16.7	NA	15.0	× 4	91)	9/)			Vebolled	Vecalu.
4,4'-DDT	1	1	16.8	1	/ 0/	/0/	WA			
			-							

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

LDC #: 19091 A30 SDG #: pu coner

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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### METHOD: GC Pesticides/PCBs (EPA SW 846 Method 8081/8082)



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

Example:		
Sample I.D.	<u> </u>	
Conc. = <u>(</u> (		)
=		
	ND	

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

Note:

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

Project/Site Name:	BRC Tronox Parcel F
Collection Date:	June 10, 2008
LDC Report Date:	July 22, 2008
Matrix:	Soil
Parameters:	Polychlorinated Biphenyls
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

### Sample Delivery Group (SDG): F8F110177

### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8082 for Polychlorinated Biphenyls.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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. GC/ECD Instrument Performance Check**

Instrument performance was acceptable unless noted otherwise under initial calibration and continuing calibration sections.

#### **III. Initial Calibration**

Initial calibration of multicomponent compounds was performed for the primary (quantitation) column as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all compounds.

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

#### IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

Retention times (RT) of all compounds in the calibration standards were within QC limits for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

#### V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated biphenyl contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

#### VII. Matrix Spike/Matrix Spike Duplicates

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

#### VIII. Laboratory Control Samples (LCS)

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

#### IX. Regional Quality Assurance and Quality Control

Not applicable.

#### X. Pesticide Cleanup Checks

#### a. Florisil Cartridge Check

Florisil cleanup was not required and therefore not performed in this SDG.

#### b. GPC Calibration

GPC cleanup was not required and therefore not performed in this SDG.

#### XI. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### XII. Compound Quantitation and Reported CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### XIII. Overall Assessment of Data

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

#### XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel F Polychlorinated Biphenyls - Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Polychlorinated Biphenyls - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: 19091A3b VALIDATION COMPLETENESS WORKSHEET	Date: 7/18/08
SDG #: <u>F8F110177</u> Level III/IV Laboratory: Test America	Page: <u>/</u> of <u>/</u> Reviewer: <u>/</u>
METHOD: GC Polychlorinated Biphenyls (EPA SW 846 Method 8082)	2nd Reviewer:

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
١.	Technical holding times	Δ	Sampling dates: 61008
١١.	GC/ECD Instrument Performance Check	NΔ	
111.	Initial calibration	Δ	۲
IV.	Continuing calibration/ICV	A	$101 \leq 15$
<b>V</b> .	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	AH	client specifical TSB-G1-08-10
VIII.	Laboratory control samples	A	Les
IX.	Regional quality assurance and quality control	N	
Xa.	Florisil cartridge check	N	
Xb.	GPC Calibration	N	
XI.	Target compound identification		Not reviewed for Level III validation.
XII.	Compound quantitation and reported CRQLs		Not reviewed for Level III validation.
XIII.	Overall assessment of data	7	
XIV.	Field duplicates	N	
xv.	Field blanks	N	

Note:

A = Acceptable N = Not provided/applicable SW = See worksheet ND = No compounds detected R = Rinsate FB = Field blank

D = Duplicate TB = Trip blank EB = Equipment blank

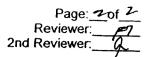
Validated Samples:

mples: \*\* Indicates sample underwent Level IV validation

1	TSB-FR-02-02-20'	11	F&F160000-162	21	8168162	31	
21	TSB-FR-02-02-30'**	12		22		32	
31	TSB-FJ-02-02-10'**	13		23		33	
4	TSB-FJ-02-02-20'**	14		24		34	
<u>5</u> \	TSB-FJ-02-02-30'	15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Page: /of 1/ Reviewer: \_\_\_\_7 2nd Reviewer: \_\_\_\_

Method:GCHPLC				
Validation Area	Yes	No	NA	Findings/Comments
L Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.	V	1		·
		創資		
Did the laboratory perform a 5 point calibration prior to sample analysis?	V			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?				
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		~		
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?	1/	<u>t</u>		
IV/Confidence and the second			1.	
What type of continuing calibration calculation was performed?%D or%R	[			
Was a continuing calibration analyzed daily?	1			
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?	1			
Were all the retention times within the acceptance windows?				
		國家		
Was a method blank associated with every sample in this SDG?	/			
Was a method blank analyzed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
Witspiningane spikesu				
Were all surrogate %R within the QC limits?				
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?			_	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				
VIDMalox solkerMatox spike duplicates (at the set in the	<b>新</b> 的			
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.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?			$\top$	
Nere the MS/MSD percent recoveries (%R) and the relative percent differences RPD) within the QC limits?	7			· ·
All teborelogy control samples as a service state of the service o				
Vas an LCS analyzed for this SDG?	7	T		
Vas an LCS analyzed per extraction batch?	7			
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) vithin the QC limits?	/			



	T	i	<u> </u>	
Validation Area	Yes	No	NA	Findings/Comments
OX. Regional Duality Assurance and Quality Control	<b>1</b> 965			
Were performance evaluation (PE) samples performed?			12	
Were the performance evaluation (PE) samples within the acceptance limits?			1	
x manual and the model and the second sec				
Were the retention times of reported detects within the RT windows?		CHARACTER IN		
	-	-		
M. Dempound quantitation (cr(g)). A start of the second start of t				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?		-		
any angle level of applicable to level to validation?	$\square$			
System performance was found to be acceptable.	17		T	
Overall assessment of data was found to be acceptable.	/	-	1	
Field duplicate pairs were identified in this SDG.		1	-	
larget compounds were detected in the field duplicates.			7	
	nik si			
ield blanks were identified in this SDG.		1		
arget compounds were detected in the field blanks.		F	T	
			I	

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LDC # 19091 A3 b cert 3 SDG #:

## Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: 2nd Reviewer. Reviewer.

HPLC METHOD: GC\_

The calibration Factor (CF), average CF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following

CF = A/C average CF = sum of the CF/number of standards %RSD = 100 \* (S/X)

- A = Area of compound, C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs

				· · · · · · · · · · · · · · · · · · ·					
		:		Reported	Recalculated	Reported	Recalculated	Reported	
*	Standard (D	Calibration Date	Compound	CF ()coOstd)	CF	Average CF	Average CF		
+	ICA L	52108			1100001	(initial)	(initial)	%RSD	KRSD
		) -	Arector 1260 CHA	L181-6	27377	LTPLE	27977	50	0.41
	-	_	L ChB	38 550	78580	29164	29165	a Can	9.00.0
								2002	Anel
2									
Ι									
1			-						
,									
7									
4									
T									
Comme	lents: <u>Refer to I</u>	nitial Calibratio	Comments: Refer to Initial Calibration findings worksheet for list of gualifications and associated samples when reported results do not accountation of the second se	ons and associ	<u>ated samples w</u>	then reported res	uits do not same		
		,					אווים חה ווהו מחוםם	WITHIN 10.0%	of the recalculs

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4 5 1 40 41 43 P en care SDG #:

**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

Reviewer. 2nd Reviewer; Page:

HPLC METHOD: GC

The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the compounds identified below

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where: ave. CF = initial calibration average CF CF = continuing calibration CF A = Area of compound C = Concentration of compound

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10,0% of the Recalculated ۲-۲ ý å و Reported h τ, Χ '،' ف ۵v 2061.2510 Recalculated PEE. 75 10 CF/Conc. CCV 937.3342 952. 1902 CF/Conc. CCV Reported Average CF(Ical)/ CCV Conc. 0001 0001 1260 CAA GND 7260 Compound بولەمىم کمرا مر 60/2119 6 118 08 Calibration Date PCAL 100 PCAL089 Standard ID 16:03 13:04 \* 3 ო

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## VALIDATION FINDINGS WUKKSHEEI Surrogate Results Verification

rage: 01 / Reviewer: 2nd reviewer:

METHOD: \_\_\_\_\_GC \_\_\_\_ HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
DCB	ChA	06	C1552 .91	8	R	٥

### Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

### Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

LUU #. 1-10-11 4% D 50205 SDG #: 20

# <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

Page: / of Z Reviewer: 2nd Reviewer:\_\_\_

METHOD: GC HPLC The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using

Where

the following calculation: %Recovery = 100 \* (SSC - SC)/SA

RPD =(({SSCMS - SSCMSD} \* 2) / (SSCMS + SSCMSD))\*100

SSC = Spiked sample concentration SA = Spike added MS = Matrix spike

MSD = Matrix spike duplicate SC = Sample concentration

> 01-80-65 T 5 B -MS/MSD samples:

L

	Spike Added	Sample Conc	Spike Sample	ole	Matrix	Matrix spike	Matrix Spik	Matrix Spike Duplicate	USW/SW	
Compound	L Val KX	IL was her	NA RM )		Percent	Percent Recovery	Percent Recovery	Perover.		
「「「「「「「」」」、「「」」、「」」、「」」、「」」、「」、「」、「」、「」、	MS MSD	5 1	WS	0 MSD	Denord	,			אאח	
Gasoline (8015)						Recalc.	Kepored	Recalc.	Reported	Recalc.
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)				Ī						
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)				T						l
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
Amecler 1260	SLI LLI	dn	1 1 1 1 1	a L					-	-
			-	- <del> </del> -  -	40	4	50	501	7.4	7.4
Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of gualifications and associated samples when reported results do not acrossified to not acrossified	e/Matrix Spike Dup	licates findings	s worksheet for lis	t of qualific	l ations and as	sociated sam	l ples when rec	orted results	do not acrea	10 00%

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Page: \_\_\_\_\_of \_\_\_\_ Reviewer: \_\_\_\_\_\_ 2nd Reviewer: \_\_\_\_\_

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

%Recovery = 100 \* (SSC - SC)/SA

Where SSC = Spiked concentration SA = Spike added

SC = Sample concentration

LCS = Laboratory Control Sample percent recovery

LCSD = Laboratory Control Sample duplicate percent recovery

KPD =(((ssclcs - ssclcsD) - 2) / (ssclcs + ssclcsD))-100

LCS/LCSD samples: LCS

	Spi	e te	Sample	Spike Sample	ample	rcs	Ş		0	LCS/LCSD	csD
Compound	1 walke	Key	I walkey	Concent VS	ration KA	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	0
	LCS U	LCSD	0 • 1	LCS U	Gesp	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Aroda 1260	791	42		171	24 2	501	103	4 2			

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of gualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: SDG #:	LDC #: 1909 1 A3 b SDG #: 224 conor	VALIDAT <u>Samp</u> l	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	SHEET ion	Page: <u>of</u>
MET	METHOD:GCHPLC				2nd Reviewer:
zz ≻≻	NIA NIX	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	or all level IV samples? npounds within 10% of the re	sported results?	
•	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100) A= Area or height of the compound to be measured Fv= Final Volume of extract	0) Basured Sample ID.		Compound Name	dn
Df= RF=/ Ss=/ Ss=/	Df= Dilution Factor RF= Average response factor of the compound In the initial calibration Vs= Initial volume of the sample Ws= Initial weight of the sample %S= Percent Solid	Concentration =	81		
*	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
		-			
Comments:	ients:				

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

Project/Site Name:	BRC Tronox Parcel F
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Collection Date: June 10, 2008

LDC Report Date: July 23, 2008

Matrix: Soil

Parameters: Metals

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

#### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

#### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Methods 6010B, 6020, and 7000 for Metals. The metals analyzed were Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Mercury, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, Zinc, and Zirconium.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the methods stated above.

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.

Blanks are summarized in Section III.

Field duplicates are summarized in Section XIII.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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. Calibration

An initial calibration was performed.

The frequency and analysis criteria of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
PB (prep blank)	lron	12.1 mg/Kg	All samples in SDG F8F110177
ICB/CCB	Antimony Thallium Tungsten Vanadium	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L	All samples in SDG F8F110177

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.

#### IV. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

The criteria for analysis were met.

#### V. Matrix Spike Analysis

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

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-FJ-06-02-101MS/MSD (All samples in SDG F8F110177)	Antimony Barium Copper Magnesium Niobium Phosphorus Tungsten Zinc	50.0 (75-125) 61.1 (75-125) 73.2 (75-125) 43.4 (75-125) 38.8 (75-125) 43.6 (75-125) 71.5 (75-125) -	50.0 (75-125) 61.0 (75-125) - 34.8 (75-125) 39.3 (75-125) 63.8 (75-125) 71.0 (75-125) 74.8 (75-125)	-	J- (all detects) UJ (all non-detects)	A

#### VI. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable.

#### VII. Laboratory Control Samples (LCS)

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

#### VIII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits for samples on which a EPA Level IV review was performed with the following exceptions:

Sample	Internal Standard	%R (Limits)	Analyte	Flag	A or P
TSB-FJ-02-02-10'**	Sc <sup>45</sup>	132.5 (30-120)	Strontium	J (all detects) UJ (all non-detects)	А

Raw data were not evaluated for the samples reviewed by Level III criteria.

#### IX. Furnace Atomic Absorption QC

Graphite furnace atomic absorption was not utilized in this SDG.

#### X. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria were met with the following exceptions:

Diluted Sample	Analyte	%D (Limits)	Associated Samples	Flag	A or P
TSB-FJ-06-02-10'L	Calcium Phosphorus Titanium	13.8 (≤10) 15.6 (≤10) 19.2 (≤10)	All samples in SDG F8F110177	J (all detects) J (all detects) J (all detects)	A

#### XI. Sample Result Verification

All sample result verifications were acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### XII. Overall Assessment of Data

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

#### XIII. Field Duplicates

No field duplicates were identified in this SDG.

#### BRC Tronox Parcel F Metals - Data Qualification Summary - SDG F8F110177

SDG	Sample	Analyte	Flag	A or P	Reason
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Antimony Barium Copper Magnesium Niobium Phosphorus Tungsten Zinc	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
F8F110177	TSB-FJ-02-02-10'**	Strontium	J (all detects) UJ (all non-detects)	A	Internal standards (%R)
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Calcium Phosphorus Titanium	J (all detects) J (all detects) J (all detects)	A	ICP serial dilution (%D)

#### BRC Tronox Parcel F Metals - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Metals - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: 19091A4	VALIDATION COMPLETENESS WORKSHEET	Date: 7/>>/-8
SDG #: F8F110177	Level III/IV	Page: (of )
Laboratory: Test America		Reviewer:
-		2nd Reviewer:
METHOD: Metals (EPA SW 8	346 Method 6020/6010B/7000)	9

#### METHOD: Metals (EPA SW 846 Method 6020/6010B/7000)

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
١.	Technical holding times	A	Sampling dates: 6/10/08
11.	Calibration	A	
1.	Blanks	ŚW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	Ś	2 MS/MSD TSB-FJ-06-2-101
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	À	Lug
VIII.	Internal Standard (ICP-MS)	9W	Not benievered for 1-ene 3
IX.	Furnace Atomic Absorption QC	N	Not treniend for level 3 hit utilized
Х.	ICP Serial Dilution	5W	a
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	Ň	
XIV.	Field Blanks	N	



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

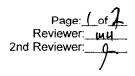
Validated	Samples:	
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\*\* Indicates sample underwent Level IV validation

1	TSB-FR-02-02-20'	11	21	31	
2	TSB-FR-02-02-30'**	12	22	32	
3	TSB-FJ-02-02-10'**	13	23	33	
4	TSB-FJ-02-02-20'**	14	24	34	
5	TSB-FJ-02-02-30'	15	25	35	
6	PB	16	26	36	
7	\	17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:

#### VALIDATION FINDINGS CHECKLIST



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#### Method:Metals (EPA SW 846 Method 6010/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical fiolding times	A		國語	
All technical holding times were met.	1		ļ	
Cooler temperature criteria was met.				
II.:Calibration		1994 1		
Were all instruments calibrated daily, each set-up time?	1		<u> </u>	
Were the proper number of standards used?	1			
Were all initial and continuing calibration verification %Rs within the 90-110% (80- 120% for mercury and 85-115% for cyanide) QC limits?	1			
Were all initial calibration correlation coefficients > 0.995? (Level IV only)				
III Blanks			i i i	
Was a method blank associated with every sample in this SDG?	1			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.				
M ICR Interference Check Sample:		CSE.		
Were ICP interference check samples performed daily?	1			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			808885787	
IV-Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, Indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.		/		
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of +/- RL(+/-2X RL for soil) was used for samples that were $\leq$ 5X the RL, including when only one of the duplicate sample values were $\leq$ 5X the RL.	\			
V-Laboratory control samples		<u> jiz</u> ji		
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?	$\angle$			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			
Vi, Furnace Atomic Absorption QC		5		
If MSA was performed, was the correlation coefficients > 0.995?			<u> </u>	
Do all applicable analysies have duplicate injections? (Level IV only)			$\angle$	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			1	
Were analytical spike recoveries within the 85-115% QC limits?	]			

#### LDC # 1909 144 SDG # See cover

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#### VALIDATION FINDINGS CHECKLIST

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

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Validation Area	Yes	No	NA	Findings/Comments
MI. ICR Serial Dilution			99988 9468	
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	$\mathbb{N}$			>100× may for replan
Were all percent differences (%Ds) < 10%?				· · · · · · · · · · · · · · · · · · ·
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.		/	ł	
VIII. Internal Standards (EPA SW 846 Method 6020)				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	ĸ	$\checkmark$		
If the %Rs were outside the criteria, was a reanalysis performed?	X			
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
X Sample Result Ventication Sec. 2 7 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
XI: Overall assessment of dela				
Overall assessment of data was found to be acceptable.	/			
XII: Field duplicates				
Field duplicate pairs were identified in this SDG.		~		
Target analytes were detected in the field duplicates.				
XIII Field blanks				
Field blanks were identified in this SDG.	Ī	7	/	
Target analytes were detected in the field blanks.			7	

LDC #: 1909 A4 SDG #: \_\_\_\_\_\_ e w

#### VALIDATION FINDINGS WORKSHEET <u>Sample Specific Element Reference</u>

Page:_	of/
Reviewer:	MU
2nd reviewer:	4
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All circled elements are applicable to each sample.

		Target Analyte List (TAL)
Sample ID	1	
15	501	Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,
	,	
1-+	Soi)	Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,
		Analysis Method
ICP		Lis-7
ICP-MS		Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, S,
ICP-MS		(Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,)
GEAA		Al Sb As Ba Be Cd Ca Cr Co Cu Fe Pb Mg Mn Hg Ni K Se Ag Na Ti V Zn Mo B Si CN

Comments: Mercury by CVAA if performed // Nb: Niobium, Pd: Palladium, P: Phosphorus, Pt: Platinum, S: Sulfur, W: Tungsten, U: Uranium, Zr: Zirconium

		V 846 N
		Metals (EPA SW 846 N
**	/er	e Metals
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LDC #: 19091A	SDG #: See	METHOD: ]
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# VALIDATION FINDINGS WORKSHEET <u>PB/ICB/CCB QUALIFIED SAMPLES</u> Soil preneration fortor applied.

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Page:	Reviewer:	2nd Reviewer:

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	Autom         Maximum	Sample	Concentrati	Sample Concentration units, unless otherwise noted: mg/Kg	less otherw	ise noted:	mg/Kg	Associ	Associated Samples: All (ND	: All (ND or >	RL)			ZNG Kevlewer.	/er:
alve         Maximum         Maximum         Bank         Maximum         Maxi	Wathware       Maximum       Maximum <th></th> <th>ntification</th> <th></th> <th></th> <th></th>											ntification			
	Sb       1:3       1	Analyte													
	ico         32,1         1         12,	Sb			1.3										
	TH         1         0.22         1         0.23         1<	е Е	12.1			121									
	W         14 </td <td>Ē</td> <td></td> <td></td> <td>1.1</td> <td>0.22</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Ē			1.1	0.22									
		3			1.4										
		>			2.7										
	Sample with the dimension state as associated (10, CCB with the dimension state as associated (10, CCB with the dimension state as associated (10, CCB with the dimension state associated (10, CCB with the dimensited (10, CCB with the dimension state associated (10, CC														
	Samples with in the times with the times withe associated with the times with the times with the														
	Samples with analyte concentrations within free the associated CIB. CCB or PE concentration are liked above with the identification. Concentrations with the identification. <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>														
	Samples with analyte concentrations within the times the associated (C.B. C.B. or PB. Or PB. C.B. or PB. Or PB. C.B. or PB. C.B. or PB. C.B. or PB. C.B. or PB. Or PB. Or PB. C.B. or PB. Or PB. C.B. or PB. Or P														
	Samples with an at Net concentration swith the function of the functine functine function of the function of the function of the functi														
	Samples with first concentrations with first concentration are listed above with the functioner from the funct														
	Samples with analyte concentrations within five times the associated (CB or PB concentration are listed above with the identification Conventence (Variation Con														
	Samples with analyte concentrations with the identifications from the National Actional Actionactive Actionactive Actional Actional Actional Actional Actional Ac														
	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identification Convolutions Arouse Without Arouse														
	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identifications from the Validation Convolutions Workshoed, T-Look														
	Samples with analyte concentrations with five times the associated ICB. CCB or PB concentration are listed above with the identification Complexed Made above Without the identification Complexed Without the identitication Complexed Without the identification Complexed														
	Samples with analyte concentrations with five times the associated ICB. CCB or PB concentration are listed above with the identification Complementer Of the ide														
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	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identifications from the Validation Completence Marketone Theorem														
	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identifications from the Validation Completences Madadact Theorem														
	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identifications from the Validation Completences Madanast Theorem														
	Samples with analyte concentrations within five times the associated ICB. CCB or PB concentration are listed above with the identifications from the Validation Completence Montonto Thomas														

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Matrix Spike/Matrix Spike Duplicates VALIDATION FINDINGS WORKSHEET

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METHOD: Trace metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y N NA</u> Was a matrix spike analyzed for each matrix in this SDG? <u>Y N NA</u> Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.

Were all duplicate sample relative percent differences (RPD)  $\leq$  20% for water samples and  $\leq$ 35% for soil samples? N N/A

EVEL IV ONLY:

Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations. A N N/A

្រក		-	<u> </u>		<b>r</b>		T	1	1		1	-	T	-	1		 -	1		1	T	3	1
	QualKications	J-/ 41/A	-						<u> </u>														
	Associated Samples	<u>_</u>																					
	RPD (Limits)																						
	MSD %Recovery	مرمع	61.0		34,8	39,3	63.8	- e · l u	74.8													4 X	
	MS %Recovery	o vs	6 (、)	13.2	43.4	38,8	43.6	<u> 11, F</u>														イン い レキメ	
	Analyte	sb	Ba	C.	Me	Nb <sup>0</sup>	4	N I	tr													51.50	· · · · · · · · · · · · · · · · · · ·
	Matrbx	Soì.							-													te My	<b>`</b>
-	MS/MSD ID	75B-FJ-06-	101-20	-												-	÷					Comments: K, T	
	*	1			-						_		_	_					_		_	Comr	

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## VALIDATION FINDINGS WORKSHEET Internal Standards (ICP-MS)

Reviewer: Page: 2nd Reviewer;

METHOD: Metals (EPA SW 846 Method 6020)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>Y M N/A</u> Were all internal standard percent recoveries within 30-120% of the intensity of the internal standard in the initial calibration standard ?

Y M N/A

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*	/ Internal Standard		al Standard Associated Matrices	ze tay in the second second	
Ŀ	the second secon	Associated Metals	XR (Limits)	Associated Samples	Qualifications
_	%	۶۲	132.5	~	T/2/2
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L					

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## VALIDATION FINDINGS WORKSHEET **ICP Serial Dilution**

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METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

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

If analyte concentrations were > 50X the MDL (ICP) ,or >100X the MDL (ICP/MS), was a serial dilution analyzed? Were ICP serial dilution percent differences (%D) ≤10%? Is there evidence of negative interference? If yes, professional judgement will be used to qualify the data. EVEL IV ONLY: Y N N/A Y N/A N/A

$\Sigma$	Y) N N/A	Were recalculated resul	Its acceptable?	See Level IV	Recalculation Wor	Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.		
#	Date	Diluted Sample ID	Matrix	Analvte	•/AD (1 imits)	Associated Samples	Qualifications	
		TSB-FJ-06-02-10	101 Sor	رمر	13.8	Å)	77+/4	
				4	12.6	-		
				エバ	19.2		~	
T								
T								_
j j	Comments:	TAMX == > / IN	19M					1

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VALIDATION FINDINGS WORKSHEET Initial and Continuing Calibration Calculation Verification

Page: <u>/ of /</u> Reviewer: <u>////</u> 2nd Reviewer: <u>/</u>

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

Where, Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution True = concentration (in ug/L) of each analyte in the ICV or CCV source %R = Found × 100 True

					Recalculated	Reported	
Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	%R	%R	Acceptable (Y/N)
MI	ICP (Initial calibration)	L,	4037	4000	( روم ا	(00,9	٢
	GFAA (Initial calibration)			-		~	
M	CVAA (Initial calibration)	(tg	152	2.5	( no. Y	120.4	٢
ANI	ICP (Continuing calibration)	s	08925	Pro00	105.4	اوۍ ځ	1
	GFAA (Continuing calibration)				/		
ra	CVAA (Continuing calibration)	Ha	4.9	Suo	98r0	98.6	7
TCV	ICP/MS (Initial calibration)	6p Gl	1019,4	الدى	( 0   1 )	(*/~)	
נגא	ICP/MS (Continuing callbatton)	4	3.881.6	4000	9 N. 0	97,0	4

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

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## VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet

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METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result). True = Concentration of each analyte in the source. %R = <u>Found</u> x 100 True

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

S = Original sample concentration D = Duplicate sample concentration
Where,
× 100
RPD = <u>IS-DI</u> (S+D)/2
RPC

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

= Initial Sam	SDR = Serial Dilution Re
Where, I	SDR = Seri
%D = [-SDR] × 100	
×	
Ľ	

ere, 1 = Initial Sample Result (mg/L) R = Serial Dilution Result (rng/L) (Instrument Reading x 5)

Sample ID     Type of Analysis     Element       TUCAN     ICP interference check     2n       TUCY     Laboratory control sample     Q       TSB     FJ-ob     Matrix spike     b	lent	Equad ( e / )				
nce check antrol sample		(units)	True / D / SDR (units)	%R / RPD / %D	%R / RPD / %D	Acceptable (Y/N)
ontrol sample	2	ていかの	ه ه )	[ » 4	( ه <i>√</i>	λ
		049	147	104.9	1048	_
		$\langle \iota l \iota, \iota$ (AS-ASS)	2.655-1	89.3	89.4	
1 Duplicate A		62119	0116	6~8	6 . 8	
1 ICP serial dilution	er s	(3-37-5 09-52)	2-61.42	ent of	2	X

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

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#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".(N N/A)Have results been reported and calculated correctly?(N N/A)Are results within the calibrated range of the instruments and within the linear range of the ICP?(N N/A)Are all detection limits below the CRDL?

Detected analyte results for \_\_\_\_\_\_ were recalculated and verified using the following equation:

Recalculation:

 Concentration =
 (RD)(FV)(Dil) (In. Vol.)(%S)

 RD =
 Raw data concentration

 FV =
 Final volume (ml)

 In. Vol. =
 Initial volume (ml) or weight (G)

 Dil =
 Dilution factor

S= 9.314 mg/~x 0.1 Rx 2 / 1000 f/ry = 2688 mg/ry 0.59 x 0.6931 = 2688 mg/ry

%S = Decimel percent solids

Sample ID	Analyte	Reported Concentration ( WK( KS))	Calculated Concentration ( L <sup>M</sup> J M )	Acceptable (Y/N)
2	Lì	133	133	Y
	5	2690	2693	ľ
	AC	18200	(8200)	
	A3	35.5	305	
·····	Ba	56.2	56.~	
	12e	0.97	0.97	
	Β	27.8	27.8	
	Co	23400	11400	
	(x	26,4	26,4	
	Co	8.8	8.8	
	<u> </u>	28.8	2818	
	Fe	19900	19900	
	pb	10.6	(0,6	
	Ing	4.400	45100	
	lug	310	310	
	M D	219	2,8	
		20.17	20,7	
	Pd	0.48	0.48	
	P	82	812	
	K	3780	3780	
	Si	1200	1200	
	Ag	0.19	0.19	J

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LDC #: 909 AU SDG #: See we

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: **Reviewer:** 2nd reviewer:

METHOD: Trace Metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". $(1 \ N \ N/A)$ Have results been reported and calculated correctly? $(1 \ N \ N/A)$ Are results within the calibrated range of the instruments and within the linear range of the ICP? $(2 \ N \ N/A)$ Are all detection limits below the CRDL?

Detected analyte results for \_\_\_\_\_\_ were recalculated and verified using the following equation:

Concentration = (RD)(FV)(Dil) (In. Vol.)(%S) **Recalculation:** 

RD Raw data concentration F۷ Final volume (ml) = initial volume (mi) or weight (G) In. Vol. = Dil **Dilution factor** = Decimal percent solids %S =

Na = 668.431 w/2xalex 5 = 964.4 mg/2y

Sample ID	Analyte	Reported Concentration ( Wg/M-f )	Calculated Concentration ( \\\\)	Acceptable (Y/N)
7	NR Sr Tì	964	964	У
	Sr.	219	219	/
	Tì	866	866	
	Ч	6.7	6.7	
	V	60,9	60.9	
	<u> </u>	650	65%	
	<del>2</del> Ý	44.4	44.4	Y
			· · · · · · · · · · · · · · · · · · ·	
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#### Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: BRC Tr	ronox Parcel F
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Collection Date: June 10, 2008

LDC Report Date: July 23, 2008

Matrix: Soil

Parameters: Wet Chemistry

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

#### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

#### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA Method 300.0 for Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate as Nitrogen, Nitrite as Nitrogen, Orthophosphate as Phosphorus, and Sulfate and EPA SW 846 Method 9071B for Oil & Grease.

The review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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.

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#### 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. Calibration

#### a. Initial Calibration

All criteria for the initial calibration of each method were met.

#### b. Calibration Verification

Calibration verification frequency and analysis criteria were met for each method when applicable.

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Concentration	Associated Samples
МВ	Orthophosphate as P	1.1 mg/L	All samples in SDG F8F110177
ICB/CCB	Orthophosphate as P	0.237 mg/L	All samples in SDG F8F110177

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.

#### IV. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

#### V. Duplicates

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

#### VI. Laboratory Control Samples

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

#### **VII. Sample Result Verification**

All sample result verifications were acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### **VIII. Overall Assessment of Data**

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

#### **IX. Field Duplicates**

No field duplicates were identified in this SDG.

BRC Tronox Parcel F Wet Chemistry - Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Wet Chemistry - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: 19091A6	VALIDATION COMPLETENESS WORKSHEET	Date: 7/22/08
SDG #: F8F110177	_ Level III/IV	Page: <u>[</u> of_/
Laboratory: Test America		Reviewer: <u>My</u>
	s/ //	2nd Reviewer:
	1-N 1-N	<u> </u>

METHOD: (Analyte) Bromide, Bromine, Chlorate, Chloride, Chorine, Fluoride, Nitrate, Nitrite, Orthophosphate-P, Sulfate (EPA Method 300.0), O & G (EPA SW846 Method 9071B)

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
١.	Technical holding times	A	Sampling dates: 6/10/08
lla.	Initial calibration	A	
llb.	Calibration verification	A	
11).	Blanks	5~	
IV	Matrix Spike/Matrix Spike Duplicates	A	2 M5/Vup TSB-FJ-06-02-101
v	Duplicates	A	
VI.	Laboratory control samples	A	Loz
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Lx	Field blanks	Ń	

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:
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Note:

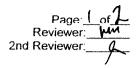
\*\* Indicates sample underwent Level IV validation

	<u></u>				
1	TSB-FR-02-02-20'	11	21	31	
2	TSB-FR-02-02-30'**	12	22	32	
3	TSB-FJ-02-02-10'**	13	23	33	
4	TSB-FJ-02-02-20'**	14	24	34	
5	TSB-FJ-02-02-30'	15	25	35	
6	MB	16	26	36	
7	· • •	17	27	37	
8		18	28	38	
9		19	29	39	
10		20	30	40	

Notes:

LDC #: (90) Ab SDG #: <u>Cel cove</u>

#### VALIDATION FINDINGS CHECKLIST



Method: Inorganics (EPA Method Fee could				
Validation Area	Yes	No	NA	Findings/Comments
I Technical holding times				ALLEL OF LEAST AND AVAILUES
All technical holding times were met.	V	L		
Coolcr tcmpcraturc critcrie was met.	1			
Ill'scalabration	洋柏			
Were all instruments calibrated daily, each set-up time?	1			
Were the proper number of standards used?	1			
Were all initial calibration correlation coefficients > 0.995?	1			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?	/			
Were titrant checks performed as required? (Level IV only)			/	
Were balance checks performed as required? (Level IV only)	V			
Was a method blank associated with every sample in this SDG?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	/			
WiMado Spike Matricipies and Duplicates and Duplicates and the second states and the sec				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	-			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/			
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq$ 20% for waters and $\leq$ 35% for soil samples? A control limit of $\leq$ CRDL( $\leq$ 2X CRDL for soil) was used for samples that were $\leq$ 5X the CRDL, including when only one of the duplicate sample values were $\leq$ 5X the CRDL.				
V Eaboraton/Poontrolsamples		<b>.</b> .		
Was an LCS anaylzed for this SDG?				
Was an LCS analyzed per extraction batch?	$\land$			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?				
VI. Regional Ideality Asstirance and Quality Control				
Were performance evaluation (PE) samples performed?			$\Delta$	
Were the performance evaluation (PE) samples within the acceptance limits?		- 1	ł	-

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#### VALIDATION FINDINGS CHECKLIST

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Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification		14. 14. 7 4. 7	e a la calegaria de la calegar	
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	1			
Were detection limits < RL?				
				<b></b>
Overall assessment of data was found to be acceptable.	1			
Field duplicate pairs were identified in this SDG.		$\checkmark$		
Target analytes were detected in the field duplicates.			7	
如果在Participation。这些中国的"中国"的中国中国的中国				
Field blanks were identified in this SDG.		~		
Target analytes were detected in the field blanks.			7	

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appliant total reaction

#### VALIDATION FINDINGS WORKSHEET Sample Specific Analysis Reference

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All circled methods are applicable to each sample.

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Sample ID	<u>Matrix</u>	Parameter
1-5	Soil -	Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> (O+O/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO₄ O-PO₄ Chlorate ClO₄ O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
·		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate CIO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine CI Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH
		Br Bromine Cl Chlorine F NO <sub>3</sub> NO <sub>2</sub> SO <sub>4</sub> O-PO <sub>4</sub> Chlorate ClO <sub>4</sub> O+G/TPH

Comments:

LDC #: (9°9) AS

VALIDATION FINDINGS WORKSHEET Blanks

Page: \_\_\_\_of 2nd Reviewer: Reviewer:

METHOD: Inorganics, Method

Ser cour

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". <u>A N/A</u> Were all samples associated with a given method blank? <u>A N/A</u> Were any inorganic contaminants detected above the reporting limit in the method blanks? If yes, please see qualifications below.

11 W g/ we

_				 		 	 	 		-	_	_		 _	
															•
7	Sample Identification				•										
~~ ~	Sampl												:		
SS: H'L															
Associated Samples.				 					-						
ASSOUR															
												 			1
	Blank	Action Limit			-										
4	Maximum	<u> </u>	الردره				-								
	Blank ID	a M	1-1							-					
Conc. units:	Analyte		9-404-0												

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT: All contaminants within five times the methoc blank concentration were qualified as not detected, "U".

BLANKS.6

(g=91A6	Ser Court
LDC #:	SDG #:

# Initial and Continuing Calibration Calculation Verification Validatin Findings Worksheet

2nd Reviewer: Reviewer: Page:

Lee Com Method: Inorganics, Method \_

8-181/3 The correlation coefficient (r) for the calibration of <u>c</u> was recalculated.Calibration date: \_ An initial or continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

%R = Found X 100

True

Where,

Found = concentration of each analyte measured in the analysis of the ICV or CCV solution True = concentration of each analyte in the ICV or CCV source

Type of analysis Analyte Star Initial calibration CI s	Standard s1					
5	s1	Conc. (ug/L)	Area	r or r <sup>2</sup>	r or r²	(N/N)
		200	0.04			
	s2	500	0.091	0.99984	0.99991	$\succ$
	s3	1000	0.191			-
	s4	2500	0.474			
	s5	5000	0.989			
ccd Calibration allowth	400	4%		Tc)	M	8
calibration F ( e	000	4,2561		5 'Eo)	کلادا	
$\begin{array}{c c} \mathcal{L} \in \mathcal{V} \\ \mathcal{L} = \mathcal{L} \\ \text{Calibration verification} \\ \end{array} \qquad \qquad$	٥٠٩	2016.6		fron )	(= 0, E	7

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

$\begin{split} \label{eq:constraint} \end{tabular} \end$	LDC #: (9.91) AL SDG #: (.2.2 con	- Ac	VAL	VALIDATION FINDINGS WORKSHEET Level IV Recalculation Worksheet	vgs workshe ation Workshee	ET t	2nd I	Page: of d Reviewer: wun 2nd Reviewer:
&A) for a laboratory control sample         Where,       Found =         True       True =         ate relative percent difference (RPD         0       Where,       S =         0       Where,       S =         10       Where,       S =         10       Where,       S =         11       D =       D =         12       D =       D =         13       D =       D =         14       D =       D =         15       D =       D =         16       D =       D + G         17       D =       D + G         16       D + G       D + G         17       D + G       D + G         16       D + G       D + G         16       D + G       D + G         17       D + G       S + G         16       D + G       S + G         16       D + G       S + G	METHOD: Inorga	nics, Method	r and					
Where,       Found =         True       =         ate relative percent difference (RPD         0       Where,       S =         0       Where,       S =         0       Where,       S =         1       D =       D =         1       D =       D =         1       D =       D =         Aboratory control sample       0.4 G         Vatrix spike sample       0.4 G         Duplicate sample       0.4 G         Duplicate sample       0.4 G         So d       So d         So propriate worksheet for list of qu	Percent recoverie	s (%R) for a laboratory co	ntrol sample and	a matrix spike samp	le were recalculated	I using the following	formula:	
A semple and duplicate relative percent difference (RPD) was recalculated using the following formula: RPD = <u>15-D1</u> × 100 Where, S = Original sample concentration (5+D)/2 × 100 Where, S = Original sample concentration <b>Example ID</b> Type of Analysis = Cound. (analysis = True, ID <b>Example ID</b> Type of Analysis = Cound.) = (analysis = True, ID <b>Example ID</b> Type of Analysis = Cound.) = (analysis = True, ID <b>Example ID</b> Type of Analysis = Cound.) = (analysis = True, ID <b>Example ID</b> (analysis = Cound.) = (analysis = True, ID <b>Example ID</b> (analysis = Cound.) = (analysis = True, ID <b>Example ID</b> (and the second teal to a constant associated samples when reported teal to the recalculated samples when reported teal to regree within 10.0% of the recalculated saturated to the transformated t	%R = <u>Found_</u> x1 True	Where		centration of each an and = SSR (spiked sa centration of each an	latyte <u>measured</u> in mple result) - SR (s alyte in the source.	the analysis of the s ample result).	ample. For the matr	ix spike calculation,
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	A sample and du	plicate relative percent diff	erence (RPD) wa	s recalculated using	the following formu	a:		
Sample IDType of AnalyticElementFound / STrue / DReactioninedReactioninedReactioninedReactioninedSample IDType of AnalyticElementFound / STrue / Dwith ywith yNat / RPDAcceptable $U_{CY}$ Luboratory control sample $0 + \frac{1}{7}$ $(1, \frac{1}{7})$ $(33 \circ B)$ $B$ $B$ $T$ $T(S_{D}-T_{T})$ Matrix splke sample $0 + \frac{1}{7}$ $(1, \frac{1}{7})$ $(33 \circ B)$ $B$ $B$ $T$ $T(S_{D}-T_{T})$ Matrix splke sample $0 + \frac{1}{7}$ $(1, \frac{1}{7})$ $(33 \circ B)$ $B$ $B$ $T$ $T(S_{D}-T_{T})$ Matrix splke sample $0 + \frac{1}{7}$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $T(S_{D}-T_{T})$ Matrix splke sample $U_{L}$ $U_{T}$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $(1, \frac{1}{7})$ $D_{uplicate sampleD_{uplicate sampleD_{uplicate sampleD_{uplicate sample(1, \frac{1}{7})(1, \frac{1}{7})(1, \frac{1}{7})(1, \frac{1}{7})D_{uplicate sampleS \circ UVTVTVT(1, \frac{1}{7})(1, \frac{1}{7})(1, \frac{1}{7})D_{uplicate sampleS \circ UVTVTVT(1, \frac{1}{7})(1, \frac{1}{7})D_{uplicate sampleS \circ UVTVT(1, \frac{1}{7})(1, \frac{1}{7})(1, \frac{1}{7})D_{uplicate sampleS \circ UVTVTVT(1, \frac{1}{7})(1, \frac{1}{7})D_{uplicate sample$	RPD = <u>!S-D!</u> × (S+D)/2		-	inal sample concentr licate sample concen	ation htration			,
Sample IDType of AnalysisElementFound /sTuel / (units)Tuel / (units)K/ RPDK/ RPDAcceptable $LCY$ Luboratory cartrol sample $0 + fy$ $ I, \gamma\rangle$ $ 3\rangle$ $ 3\rangle$ $ 3\rangle$ $ 3\rangle$ $ 3\rangle$ $ 2\rangle$ $ 1\rangle$ $TSB-EJ$ Matrix spike sample $0 + fy$ $ I, \gamma\rangle$ $ 1\rangle$ $ 3\rangle$ $ 3\rangle$ $ 3\rangle$ $ 2\rangle$ $ 2\rangle$ $TSB-EJ$ Matrix spike sample $0 + fy$ $ 1\rangle$ $ 1\rangle$ $ 1\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $TSB-EJ$ Matrix spike sample $0 + fy$ $ 1\rangle$ $ 1\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $D_{Dollade sample}$ $D_{Dollade sample}$ $ 1\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $D_{Dollade sample}$ $D_{Dollade sample}$ $ 1\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $D_{Dollade sample}$ $D_{Dollade sample}$ $ 1\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $D_{Dollade sample}$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $D_{Dollade sample}$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample}$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $ 2\rangle$ $D_{Dollade sample 2\rangle 2\rangle 2\rangle 2\rangle 2\rangle$						Recelculated	Reported	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	SR / RPD	048 / 8%	Acceptable (Y/N)
LCy $0.4 \text{ fg}$ $1.1 \text{ Jz}$ $1.32 \text{ ggg}$ $gggggggggggggggggggggggggggggggggggg$		Laboratory control sample						
TSB-EJMatrix spike sample(SSR-SR)(SSR-SR)(SSR-SR) $D(D)$ <td>10</td> <td></td> <td></td> <td>5611</td> <td>(}30</td> <td>88</td> <td>28</td> <td><b>7</b></td>	10			5611	(}30	88	28	<b>7</b>
$\mathcal{D}$ b $\mathcal{D}$ 21-1       Duplicate semple $\mathcal{D}$ Duplicate semple $\mathcal{D}$ Duplicate semple $\mathcal{D}$ <td>[]-{{S}</td> <td></td> <td>drat.</td> <td>(rs-rsc)</td> <td>(4) (4)</td> <td><i>ر د</i> ا</td> <td>۲۵/</td> <td></td>	[]-{{S}		drat.	(rs-rsc)	(4) (4)	<i>ر د</i> ا	۲۵/	
$\int_{\text{Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.}$	-1-20-90-			$\sim \sim \sim$	(int.	<pre>/ `</pre>	1	
Comments: Refer to appropriate worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.	$\rightarrow$	Duplicate sample	tas	くな	いたい	ý	1.2	<u> </u>
results.	Comments: Refe	l r to appropriate workshee	L t for list of qualific	ations and associate	ed samples when re	ported results do no	t agree within 10.0%	of the recalculated
	results.							

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LDC #: 19091A6 SDG #: \_\_\_\_\_

#### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: | of Reviewer: 2nd reviewer:

METHOD: Inorganics, Method

See con

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A". (Y N N/A Have results been reported and calculated correctly? <u>() N N/A</u>

Are results within the calibrated range of the instruments?

NN N/A Are all detection limits below the CRQL?

يك Compound (analyte) results for \_\_\_\_ reported with a positive detect were recalculated and verified using the following equation:

 $\mathcal{U} = \frac{0.15 \times \frac{0.041}{1000} \times 10^{-0.00}}{0.69 \times 45 \times 0,000196} = 158.99 \text{ mg/ng}$ Concentration =

#	Sample ID	Analyte	Reported Concentration () Wg/ Wg)	Calculated Concentration ( \mf/c)	Acceptable (Y/N)
	2	chlorate U Ur	4.8	4.8	Ý
		U	159	159	
		Cl.	317	318	
		F.,	3.5	3.5	
		NO2-N	3.4	3.4	
		SOY	524	5-5	<i>y</i>
		~ /	,		
				1	

Note:

RECALC.6

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

Project/Site Name:	BRC Tronox Parcel F
Collection Date:	June 10, 2008
LDC Report Date:	July 22, 2008
Matrix:	Soil
Parameters:	Gasoline Range Organics
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

#### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

#### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Gasoline Range Organics.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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.
- 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. Calibration

#### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) of calibration factors for compounds were less than 20.0%.

#### **b.** Calibration Verification

Calibration verification was performed at required frequencies. The percent differences (%D) of amounts in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No gasoline range organic contaminants were found in the method blanks.

No field blanks were identified in this SDG.

#### **IV. Accuracy and Precision Data**

#### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

Laboratory control samples were reviewed for each matrix as applicable. Although the LCS percent recovery (%R) was not within QC limits for one compound, the LCSD percent recoveries (%R) were within QC limits and no data were qualified.

#### V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### **VI. Compound Quantitation and CRQLs**

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### **VII. System Performance**

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### VIII. Overall Assessment of Data

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

#### **IX. Field Duplicates**

No field duplicates were identified in this SDG.

BRC Tronox Parcel F Gasoline Range Organics - Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Gasoline Range Organics - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Gasoline Range Organics - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

#### VALIDATION COMPLETENESS WORKSHEET

LDC #: 19091A7 SDG #: F8F110177 Laboratory: Test America

#### Level III/IV

Date: 7/19/08 Page: 7 of/ Reviewer: 2nd Reviewer

METHOD: GC Gasoline Range Organics (EPA SW846 Method 8015B)

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
١.	Technical holding times	A	Sampling dates: 6/10/09
lla.	Initial calibration	A	
llb.	Calibration verification/ICV	A	101 515
	Blanks	Δ	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	T58-FJ-06-02-10 LCS 10
IVc.	Laboratory control samples	sw	Les 10
V.	Target compound identification	Δ	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	Δ	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Х.	Field blanks	N	

D = Duplicate ND = No compounds detected A = Acceptable Note: TB = Trip blank N = Not provided/applicable R = Rinsate

FB = Field blank SW = See worksheet

EB = Equipment blank

\*\* Indicates sample underwent Level IV validation SOIL Validated Samples:

<b></b>							
1	TSB-FR-02-02-20'	11	F8F130000267	21	8165267	31	
2	TSB-FR-02-02-30'**	12		22		32	
37	TSB-FJ-02-02-10'**	13		23		33	
4	TSB-FJ-02-02-20'**	14		24		34	
5	TSB-FJ-02-02-30'	15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

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#### VALIDATION FINDINGS CHECKLIST

Page: \_/of \_2 Reviewer: \_\_\_\_7 2nd Reviewer: \_\_\_\_7

Method:GCHPLC					
Validation Area	Ye	s N		NA	Findings/Comments
Technical molonitarines		影響			
All technical holding times were met.	/	1			
Cooler temperature criteria was met.	~	1			÷
Did the laboratory perform a 5 point calibration prior to sample analysis?					
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?		-			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		+			
Did the initial calibration meet the curve fit acceptance criteria?		<u>ł_</u>			
Were the RT windows properly established?		1_			
IN/Southering Californian 22. / 2					
What type of continuing calibration calculation was performed?%D or%R	/	1			
Was a continuing calibration analyzed daily?	/				
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?	$\square$				
Were all the retention times within the acceptance windows?	$\square$				
Violantes and a second s					
Was a method blank associated with every sample in this SDG?	/-				
Was a method blank analyzed for each matrix and concentration?					
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	ľ	-	+		
Mit Slanog He Spakes and State Stat					
Were all surrogate %R within the QC limits?	17		Τ	Τ	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?				Ţ	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				Ŧ	
VIII Malux spike Malux spike duplicates / a	製作	國語	ję.		
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.	/				
Was a MS/MSD analyzed every 20 samples of each matrix?		-	1	T	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				T	
All settors top control samples . Sources the set of the set of the set					
Nas an LCS analyzed for this SDG?	1	4	Γ	L	
Nas an LCS analyzed per extraction batch?	$\square$		1	Τ	
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) vithin the QC limits?		/			

LDC #: 1969/4) SDG #: 40 Control

#### VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: <u>P1</u> 2nd Reviewer: <u>1</u>

		_		/
Validation Area	Yes	No	NA	Findings/Comments
OX. Regional Quality Assurance and Quality Control	796 S			
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?				
Were the retention times of reported detects within the RT windows?			7	· · ·
M Compound quantitation/CHCIS and Cheese M and a second state of the				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
System performance was found to be acceptable.		-		
Overall assessment of data was found to be acceptable.	/	-		
Field duplicate pairs were identified in this SDG.			-	
Target compounds were detected in the field duplicates.			t	-
Field blanks were identified in this SDG.		T	-	
Target compounds were detected in the field blanks.			7	

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LDC #:	SDG #:

## VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

ç Page: Reviewer: 2nd Reviewer:

\_\_\_\_ GC \_\_\_ HPLC METHOD: Please see qualifications below for all questions answered "N". Not applicable questions are identified as "NA". <u>V N N/A</u> Were a laboratory control samples (LCS) and laboratory control sample duplicate (LCSD) analyzed for each matrix in this SDG? <u>V N N/A</u> Were the LCS percent recoveries (%R) and relative percent differences (RPD) within the QC limits?

Level IV/D Only <u>Y M N/A</u> Was an LCS analyzed every 20 samples for each matrix or whenever a sample extraction was performed?

LUSICSDID         Compound Graphent         % Licston (List)         RPD (Limits) ( $-7$ )         Associated Samples           SJ ( $\mathcal{LS2L7}$ GA/O $12O$ $(-7)$ $(-1)$		K no out lesdin																				
Compound         %R (Limits)         LCSD         %R (Limits)         RPD (Limits)           GrACO         12-O         (7.3-/h3         (         )         (         )           Image: Second structure         Image: Second structu	Associated Sa	A// + B//																				
Compound ${}_{ARCD}$ ${}_{LCS}$ ${}_{ARLimits}$ ${}_{ARLimits}$ $G_{ARO}$ $12O$ $(73-1/2)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $($ $)$ $)$ $($ $)$ $)$ $)$ $)$ $)$ $)$ $)$ </th <th>RPD (Limits)</th> <td>( )</td> <td></td> <td>( )</td> <td>( )</td> <td>( )</td> <td>( )</td> <td>( )</td> <td></td>	RPD (Limits)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )		( )	( )	( )	( )	( )	
Compound         %R (Linits) $G_1 X_0$ $I_2 O$ $(7 3 - I/2)$ $G_1 X_0$ $I_2 O$ $(7 - 1)$ $(1 - 1)$			( )	( )	( )	( )		( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	
		(73-113	( )		( )			( )	( )	( )		( )				( )					( )	
8165267	Compound	64 KO																				
	rcs/rcsp id	8165267																				

292 19091 A7 3 SDG #: LDC #:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

Page: Reviewer. 2nd Reviewer.

HPLO METHOD: GC\_

The calibration Factor (CF), average CF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following

CF = A/C average CF = sum of the CF/number of standards %RSD = 100 \* (S/X)

A = Area of compound, C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs

Galibration         Calibration         Calibration         Bendication         Bendication									
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$				Reported	Recalculated	Reported	Receivitated		Recalculated
ALTION GRO AND MAD MAD MAD MAD MAD MAD MAD MAD	9	Date	Compound	CF (ノ・ジ std)	CF (ノ・ンstd)	Average CF (initial)	Average CF (initial)		
		20/12/08	GRO		1702649	171 82732	17/82732	3.915	3-915

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**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

5 Reviewer. Page: 2nd Reviewer:

HPLC METHOD: GC\_

The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the compounds identified below

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where: ave. CF = initial calibration average CF CF = continuing calibration CF A = Area of compound C = Concentration of compound

Recalculated ь О 0% 0.7 Reported å N N 9 4 **Recalculated** CF/Conc. CCV 7266.0 10195 56101 0.9976 CF/Conc. CCV Reported Average CF(Ical)/ CCV Conc. 0 ~ 1.0 Compound Q YB GRO Calibration Date 6/13/08 6/13/08 LC4 L377B LCA L3 88B Standard ID 2 ŧŧ ო

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

\_DC #:\_\_\_\_\_\_\_A09/ A7 3 SDG #: Jury

## VALIDATION FINDINGS WURKSHEEI Surrogate Results Verification

METHOD: \_\_\_\_\_GC \_\_\_ HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: 42

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Report <del>e</del> d	Recalculated	
ナチア	het she iled	0.04	E\$	83	٤۶	Q
			65550.0			

## Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

### Sample ID:

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

SURRCALCNew.wpd

LUU #. 1 909/ A 7 200 3 SDG #:

# <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

rd Page: / of Z 2nd Reviewer: Reviewer:

METHOD: GC HPLC The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

RPD =(({SSCMS - SSCMSD} \* 2) / (SSCMS + SSCMSD))\*100

MSD = Matrix spike duplicate SC = Sample concentration

> TSB - FJ - 06-02-10 MS/MSD samples:

	Spike	ex	Sample	Spike	Spike Sample	1111					
panoamoj.	PPY		Conc,	Concer	Concentration	matri	matrix spike	Matrix Spike Duplicate	e Duplicate	DSM/SM	ISD
Public Public	- W	IK3	ty Em)	Sur )	1/22	Percent	Percent Recovery	Percent Recovery	Recovery		
「「「「「「「「「」」」」	WS	MSD	, I	WS		Reported	Pacalo				
Gasoline (8015)	106	1.06	an	1.02	5100	76			Kecalc.	Керопеd	Recalc.
Diesel (8015)					011.0	0	د (	72	72	4.7	× /
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)		Ī									
HMX (8330)											
2,4,6-Trinitrotoluene (8330)		1	   .								
		Ī									
Comments: Refer to Matrix Snik	re/Matriv Si										
of the recalculated results.				worksheet fo	or list of gualifi	<u>cations and a</u>	ssociated sam	<u>ples when rep</u>	ported results	do not agree	within 10.0%

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LDC #: 190 91 A7 SDG #: 41 coner	aborator	y Contr	VA ol Sample	LIDATION	FINDINGS N Control	VALIDATION FINDINGS WORKSHEET ple/Laboratory Control Sample Duplic	IEET uplicates R	VALIDATION FINDINGS WORKSHEET Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification	fication	Page: <u>.</u> Reviewer:	le:eer:
METHOD: GC	HPLC									2n	2nd Reviewer:
The percent recoveries (%R) and relative percent differences (RPD) compounds identified below using the following calculation:	and relativusing the fo	e percent Ilowing ca	differences ( alculation:	(RPD) of the	laboratory co	introl sample	and laborator	of the laboratory control sample and laboratory control sample duplicate were recalculated for the	ple duplicate	were recalcu	lated for the
%Recovery = 100 * (SSC - SC)/SA		Where S S	SSC = Spiked concentration SA = Spike added	icentration		SC = Sample concentration	ncentration				
KPD =(((ssclcs - ssclcsD) * 2) / (ssclcs + ssclcsD))*100	/ (sscrcs + s	scrcsp).	100	LCS = Labora	ttory Control Sam	LCS = Laboratory Control Sample percent recovery		LCSD = Laboratory Control Sample duplicate percent recovery	Control Sample c	Juplicate percent	recovery
LCS/LCSD samples: 8 / 6 S	5267-	2	1								
	Spike	e ]	Sample	Spike	Spike Sample		LCS	LCSD	Q	rcs/rcsd	csD
Compound	PARA )	14N	Conci My	Conce (mrs)	Concentration	Percent	Percent Recovery	Percent Recovery	ecovery	RPD	
		LCSD	0 1			Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	0.1		1. 20	0.786	od	120	66	66	61	61
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Comments: Refer to Laboratory Control Sample/Laboratory Control	Cory Control	Sample/L	aboratory Co		Duplicate fir	<u>idings worksh</u>	eet for list of c	Sample Duplicate findings worksheet for list of qualifications and associated samples when reported	and associate	ed samples w	nen reported
			ion leaning.								

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LDC #: SDG #:	# 19091A7	VALIDATI Sampl	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET 2 <u>0</u>	Page: Reviewer:
MET	МЕТНОD: GC НРLС	· · ·		· · ·	ZNG Kevlewer:
マロ アン	NIA Were all reported revealed recalculate	Were all reported results recalculated and verified for all level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	or all level IV samples? Ipounds within 10% of the rep	orted results?	
Conc	Concentration <del>s</del> (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)	1			
A = 7 = 1 = 1 = 1 = 1 = 1 = 1 = 1 = 1 = 1 = 1	Area or height of the compound to be measured Final Volume of extract Dilution Factor	sasured Sample ID.		Compound Name	
	Average response factor of the compound In the initial calibration	concentration =	11		
Vs# 1 #8% 8 %	Vs= Initial volume of the sample Ws= Initial volume of the sample %S= Percent Solid		МN		
<b>#</b> ±	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
		-			
Comments:	ients:				

SAMPCALew.wpd

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

Project/Site Name:	BRC Tronox Parcel F
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Collection Date: June 10, 2008

LDC Report Date: July 22, 2008

Matrix:

Parameters: Diesel Range Organics

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

#### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

#### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8015B for Diesel Range Organics.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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.
- 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. Calibration

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#### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) of calibration factors for compounds were less than 20.0%.

#### b. Calibration Verification

Calibration verification was performed at required frequencies. The percent differences (%D) of amounts in continuing standard mixtures were within the 15.0% QC limits.

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds.

#### III. Blanks

Method blanks were reviewed for each matrix as applicable. No diesel range organic contaminants were found in the method blanks.

No field duplicates were identified in this SDG.

#### **IV. Accuracy and Precision Data**

#### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

#### b. Matrix Spike/Matrix Spike Duplicates

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

#### c. Laboratory Control Samples

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

#### V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### **VI. Compound Quantitation and CRQLs**

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

#### **VIII. Overall Assessment of Data**

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

#### IX. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel F Diesel Range Organics - Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Diesel Range Organics - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Diesel Range Organics - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: <u>19091A8</u> SDG #: <u>F8F110177</u> Laboratory: <u>Test America</u>

#### Level III/IV

Date: 7/19/08 Page: \_\_\_\_\_\_\_ viewer: Reviewer: D 2nd Reviewer:

METHOD: GC Diesel Range Organics (EPA SW846 Method 8015B)

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		Sampling dates: 6/10/08
lla.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	1  cm = 15
111.	Blanks	Δ	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	TSB-FJ-02-02-30'+ TSB-CJ-09-0'
IVc.	Laboratory control samples	A	105
V.	Target compound identification	A	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Х.	Field blanks	N	

 Note:
 A = Acceptable
 ND = No compounds detected
 D = Duplicate

 N = Not provided/applicable
 R = Rinsate
 TB = Trip blank

 SW = See worksheet
 FB = Field blank
 EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Level IV validation

11	TSB-FR-02-02-20'	11 <b>f</b>	8F130000-29/	21	8165291	31	
21	TSB-FR-02-02-30'**	12	F8F180000-312	22	8170312	32	
31	TSB-FJ-02-02-10'**	13		23		33	
42	TSB-FJ-02-02-20'**	14		24		34	
52	TSB-FJ-02-02-30'	15		25		35	
6		16		26		36	
7		17		27		37	
8		18		28		38	
9		19		29		39	
10		20		30		40	

Notes:

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#### **VALIDATION FINDINGS CHECKLIST**

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	/	
Method:	GC	HPLC
	Validatio	n Area
inical holding times		
nical holding times w	ere met.	
r temperature criteria w	as met.	
Sicelandione		
e laboratory perform a	5 point calibrati	on prior to sample
linear fit used for eval	unation? If yes	wara all pareant rai

	1	T	T	
Validation Area	Yes	No	NA	Findings/Comments
L ledimesticology messages and some and some set of the set				
All technical holding times were met.	/	1		
Cooler temperature criteria was met.				÷.
Did the laboratory perform a 5 point calibration prior to sample analysis?	T /	1		
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) $\leq$ 20%?	/	ł		
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	•	/		
Did the initial calibration meet the curve fit acceptance criteria?				
Were the RT windows properly established?	17	ſ		
De Contration - Linearen - La contration - La cont				
What type of continuing calibration calculation was performed?%D or%R				
Was a continuing calibration analyzed daily?				
Were all percent differences (%D) < 15%.0 or percent recoveries 85-115%?				
Were all the retention times within the acceptance windows?				
Was a method blank associated with every sample in this SDG?	ГЛ		T	
Was a method blank analyzed for each matrix and concentration?	17			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			_	
vi simogae spices				
Were all surrogate %R within the QC limits?	ГJ			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was				
a reanalysis performed to confirm %R?	m		4	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	124		1	
VIII Maiox spike Matox spike aduplicates of the set in the set in the set of				
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.				
Nas a MS/MSD analyzed every 20 samples of each matrix?	1		-+	
Nere the MS/MSD percent recoveries (%R) and the relative percent differences				
RPD) within the QC limits?	1			
All el aboratory control samples 1000000000000000000000000000000000000				<b>前方的保持,</b> 在11月1日(11月1日)
Vas an LCS analyzed for this SDG?	/		T	
Vas an LCS analyzed per extraction batch?				
Vere the LCS percent recoveries (%R) and relative percent difference (RPD) ithin the QC limits?	7			

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#### VALIDATION FINDINGS CHECKLIST

Page: 20f 2 Reviewer: 7 2nd Reviewer: 4

Validation Area	Yes			
X. Regional Quality Assurance and Quality Control		No	<u>  NA</u>	Findings/Comments
Were performance evaluation (PE) samples performed?	T	28.28		
Were the performance evaluation (PE) samples within the acceptance limits?				
X Tarte Antibiotic Contraction of the Contract of the Contract of the				
Were the retention times of reported detects within the RT windows?				
A Compound maintain Of Oto				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
System performance was found to be acceptable.			Ī	
Overall assessment of data was found to be acceptable.	7	-	Ī	
Field duplicate pairs were identified in this SDG.		Ţ		
Farget compounds were detected in the field duplicates.			7	
ield blanks were identified in this SDG.		1		
arget compounds were detected in the field blanks.			7	

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GC\_HPLC-SW.wpd version 1.0

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# Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

` of 2nd Reviewer. Page: Reviewer.

HPLC METHOD: GC\_

The calibration Factor (CF), average CF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following

CF = A/C average CF = sum of the CF/number of standards %RSD = 100 \* (S/X)

A = Area of compound, C = Concentration of compound, S = Standard deviation of the CF X = Mean of the CFs

#     Calibration     Calibration     Renotinated     Renotinated     Renotinated     Renotinated     Renotinated       1 $UAL$ $S/h_c/oX$ $Date$ $CF$ $CF$ $CF$ $Average CF$										
$\kappa$ StandardID         Compound         CF $V$			:		Reported			Receivitated		Receivated
$ (CAL 5/h_{0}/b_{2} PRO (h1a) (h1a$	#	Standard ID	Calibration Date	Compound	CF	CF CF	Average CF	Average CF		
1623     1603     3.472       1001     1003     3.472       1001     1001     1001	-	1691	S/16/0X	CI d'a	1 / worwin	(DAD) Day	(initial)	(initial)	%RSD	KRSD
					16236	16234		16023	2. 00.	2 072
	Γ								201.2	027.0
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	1					A COINTINGS AND	ALIAL LADOLOGI LALIA	sults do not agree	<u>within 10.0%</u>	of the recalcu
addition of the recalculated satisfies when reported fesults do not agree within 10.0% of the recalculated										

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**Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

Ğ Page: 2nd Reviewer:\_\_ Reviewer.

HPLO METHOD: GC\_

The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the compounds identified below

% Dlfference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

Where: ave. CF ≈ initial calibration average CF CF ≈ continuing calibration CF

A = Area of compound C = Concentration of compound

					Reported	Receivelated	Reported	Receiciteted
*	Standard ID	Calibration Date	Сотроциd	Average CF(Ical)/ CCV Conc	CF/Conc.	CF/Conc.	۵%	۵%
-	1 FCAL525	17/08	Dieve /	00.000l	996.5312	Ŏ	2,2	N
						cciall		(
							-	
2	ECAL SY9	c/18/08	Ĺ	Ż	1039.4417	103.9.4417	3.9	3.9
		1						
ო	ECALS75 6/19/0X	6/19/0%	7		( 1			
-				000/	717 8723	8-19-8723	u Ú	<u>ь</u> О
1		4		<u></u>				
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Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the

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# 00	SDG #	

### VALIDATION FINDINGS WUKKSHEE I Surrogate Results Verification

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METHOD: \_\_\_\_\_GC\_\_\_ HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: # 2

Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
			Reported	Recalculated	
not specified	'x	20.9 xcg	84	24	D
1 1					

### Sample ID:

Surrogate	Column/Detector	Surrogate Spik <del>e</del> d	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

### Sample ID:

Percent Difference			
Percent Recovery	Recalculated		
Percent Recovery	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			

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Ľ	SDG

## <u>Matrix Spike/Matrix Spike Duplicates Results Verification</u> VALIDATION FINDINGS WORKSHEET

Page: / of Z Reviewer: 2nd Reviewer:

HPLC y Q **METHOD:** 

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

SSC = Spiked sample concentration SA ≈ Spike added MS = Matrix spike

MSD = Matrix spike duplicate SC = Sample concentration

> 130 02 -02 - 471 TSB MS/MSD samples:

ws 27-7	12 (X) (	Conc.	Concer							
× 12		1 ma / fa								190
		DIN .	22-	IFX	Percent	Percent Recovery	Percent Recovery	Recovery	RPD	p
	USW	1	WS	MSD	Reported	Recalc.	Reported	Recalc	Denoted	
									netindev	
(8021B)	9.88		74.5	78.5	L¥.	12	0 8	2	1	(
				0.7	3	c.2	87	87	2.2	5:2
(KSK-175)										
(8151)										
(8151)										
Naphthalene (8310)										
Anthracene (8310)										
(8330)										
2,4,6-Trinitrotoluene (8330)										
								==		
				Ī		Ī		T		

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LDC #: 1909/ 45	aborator	y Contr	VA rol Sample	VALIDATION ple/Laborato	FINDINGS N Control	ATION FINDINGS WORKSHEET	IEET uplicates R	VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Duplicates Results Verification	ification	Page:	le:
METHOD: GC	HPLC									2n	2nd Reviewer
The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:	and relativ	e percent llowing ca	differences ( alculation:	RPD) of the	laboratory co	ntrol sample	and laborator	y control sam	ple duplicate	were recalcu	lated for the
%Recovery = 100 * (SSC - SC)/SA		Where S	SSC = Spiked concentration SA = Spike added	centration		SC = Sample concentration	ncentration				
SCLC	(ssclcs + s	scLcsD).	100	LCS = Labora	tory Control Sam	LCS = Laboratory Control Sample percent recovery		LCSD = Laboratory Control Sample duplicate percent recovery	Control Sample o	duplicate percent	recovery
LCS/LCSD samples: <u> </u>	8165291-1	102	1								
	Spike	e l	Sample	Spike :	Spike Sample		LCS	LCSD	0		csD
Compound	Added (mg./Ke	Kar)	Conc.		concentration	Percent 1	Percent Recovery	Percent Recovery	ecovery	RPD	
	LCS	LCSD	) ,	rcs		Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)											
Diesel (8015)	E. 58	NA	С	68.9	N4	٤۶	83	NA -			
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)											
Anthracene (8310)											
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											
Comments: Refer to Laboratory Control Sample/Laboratory Control	ory Control	Sample/I	aboratory Co		Duplicate fin	dings worksh	eet for list of c	Sample Duplicate findings worksheet for list of gualifications and associated samples when reported	and associate	ed samples w	nen reported
results do not agree within 10.0% of the recalculated results	.0% of the	recalcula	ted results.								

LCSCLCNew.wpd

S L	LDC #: 190 % AS	VALIDATI Sampl	VALIDATION FINDINGS WORKSHEET Sample Calculation Verification	HEET on	Page: <u>of</u> Reviewer: <u></u>
Σ	METHOD:GC HPLC				2nd Reviewer:
ж	N N/A Were all reported rest N N/A Were all recalculated	Were all reported results recalculated and verified for ail level IV samples? Were all recalculated results for detected target compounds within 10% of the reported results?	rr all level IV samples? pounds within 10% of the rep	oorted results?	
Ũ	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100)	Example:			
₹ 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	<ul> <li>Area or height of the compound to be measured</li> <li>Final Volume of extract</li> <li>Dilution Factor</li> </ul>			Compound Name	
ž žš	RF≖ Average response factor of the compound In the initial calibration Vs≖ initial volume of the sample	Concentration =	8		
<b>\$</b> %	vvs≖ Initial weight of the sample %S≖ Percent Solid	. <b>.</b>		<i>QN</i>	
Ľ					
ļ	# Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications
<u> </u>					
<u></u>					
<u> </u>					
]					
Cor	Comments:				
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SAMPCALew.wpd

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

Project/Site Name:	BRC Tronox Parcel F
Collection Date:	June 10, 2008
LDC Report Date:	July 22, 2008
Matrix:	Soil
Parameters:	Polynuclear Aromatic Hydrocarbons
Validation Level:	EPA Level III & IV
Laboratory:	TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

### Introduction

This data review covers 5 soil samples listed on the cover sheet including dilutions and reanalysis as applicable. The analyses were per EPA SW 846 Method 8310 for Polynuclear Aromatic Hydrocarbons.

This review follows a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section III.

Field duplicates are summarized in Section IX.

Samples indicated by a double asterisk on the front cover underwent a EPA Level IV review. A EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by Level III 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.
- 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. Calibration

### a. Initial Calibration

Initial calibration of compounds was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all compounds.

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### b. Calibration Verification

Calibration verification was performed at required frequencies.

The percent differences (%D) of calibration factors in continuing standard mixtures were within the 15.0% QC limits with the following exceptions:

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/16/08	Not specified	Benzo(g,h,i)perylene	15.2	TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	J+ (all detects)	A

The percent differences (%D) of the second source calibration standard were less than or equal to 15.0% for all compounds with the following exceptions:

Date	Detector	Compound	%D	Associated Samples	Flag	A or P
6/4/08	Not specified	Benzo(k)fluoranthene	16.69	All samples in SDG F8F110177	J+ (all detects)	A

Retention time windows were evaluated and considered technically acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples on which a Level III review was performed.

### III. Blanks

Method blanks were reviewed for each matrix as applicable. No polynuclear aromatic hydrocarbon contaminants were found in the method blanks.

No field blanks were identified in this SDG.

### **IV. Accuracy and Precision Data**

### a. Surrogate Recovery

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within QC limits.

### b. Matrix Spike/Matrix Spike Duplicates

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

### c. Laboratory Control Samples

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

### V. Target Compound Identification

All target compound identifications were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### VI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### VII. System Performance

The system performance was acceptable for samples on which a EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by Level III criteria.

### VIII. Overall Assessment of Data

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

### IX. Field Duplicates

No field duplicates were identified in this SDG.

### BRC Tronox Parcel F Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG F8F110177

SDG	Sample	Compound	Flag	A or P	Reason
F8F110177	TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Benzo(g,h,i)perylene	J+ (all detects)	A	Continuing calibration (%D)
F8F110177	TSB-FR-02-02-20' TSB-FR-02-02-30'** TSB-FJ-02-02-10'** TSB-FJ-02-02-20'** TSB-FJ-02-02-30'	Benzo(k)fluoranthene	J+ (all detects)	A	Continuing calibration (ICV %D)

### BRC Tronox Parcel F

Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

### BRC Tronox Parcel F Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -SDG F8F110177

No Sample Data Qualified in this SDG

LDC #: 19091A9 SDG #: F8F110177 Laboratory: Test America

### Level III/IV

Deter	T/p/by
Date:	
Page:_	<u></u>
Reviewer:	17
2nd Reviewer:	P

METHOD: GC Polynuclear Aromatic Hydrocarbons (EPA SW 846 Method 8310)

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
Ι.	Technical holding times	A	Sampling dates: 6/10/08
lla.	Initial calibration	A	
IIb.	Calibration verification/ICV	SW	$ w \neq  s $
111.	Blanks	Δ	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	75B-GJ-08-1D
IVc.	Laboratory control samples	A	105
V.	Target compound identification	Δ	Not reviewed for Level III validation.
VI.	Compound Quantitation and CRQLs	A	Not reviewed for Level III validation.
VII.	System Performance	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
Х.	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

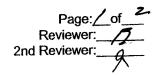
Validated Samples: \*\* Indicates sample underwent Level IV validation

1	TSB-FR-02-02-20'	11	FX F160000-158	21	8168158	31	
2	TSB-FR-02-02-30'**	12		22	•	32	
3	TSB-FJ-02-02-10'**	13		23		33	
4	TSB-FJ-02-02-20'**	14		24		34	
5	TSB-FJ-02-02-30'	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 #: 1909/A9 SDG #: <u>Le cone</u>

### VALIDATION FINDINGS CHECKLIST



Method: GC HPLC				
Validation Area	Yes	No	NA	Findings/Comments
f Technical holding-times				
All technical holding times were met.	$ \rightarrow $			
Cooler temperature criteria was met.	$\leq$	1997 A		
11. Trittal calibration	_			
Did the laboratory perform a 5 point calibration prior to sample analysis?	$\leq$			
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	_			
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?		_		
Did the initial calibration meet the curve fit acceptance criteria?			-	
Were the RT windows properly established?				
W: Continuing calibration			S C	
What type of continuing calibration calculation was performed?%D or %R	_			
Was a continuing calibration analyzed daily?	$\leq$		ļ	
Were all percent differences (%D) $\leq$ 15%.0 or percent recoveries 85-115%?	$\square$			
Were all the retention times within the acceptance windows?		-		
V Blanks	i si si T		<u> </u>	la de la constante de la const La constante de la constante de
Was a method blank associated with every sample in this SDG?				
Was a method blank analyzed for each matrix and concentration?				
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.			<u>t</u>	
VI. Sturrogale spikes			i I	T
Were all surrogate %R within the QC limits?	Ĺ			
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	V	e		
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?		Ľ		Ł
VII :Matrix spike/Matrix spike duplicates	<u> </u>	3	i T	1
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.	/ /			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII, Laboratory control samples				
Was an LCS analyzed for this SDG?	4	Į	<u> </u>	
Was an LCS analyzed per extraction batch?				<u> </u>

LDC #: 1909/A9 SDG #: \_\_\_\_\_\_\_

### VALIDATION FINDINGS CHECKLIST

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Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?			معربوه	
X. Regional Quality Assurance and Quality Goptiol				
Were performance evaluation (PE) samples performed?				
Were the performance evaluation (PE) samples within the acceptance limits?				·
X. Target compound identification				
Were the retention times of reported detects within the RT windows?				<u> </u>
XI. Compound quantifation/CRCLS	Ī			
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.		CARDIN CANTO		
XIII_Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV: Field duplicates				
Were field duplicate pairs identified in this SDG?		/	<u>t</u>	
Were target compounds idetected in the field duplicates?			-	
XV Fieldblanks				
Were field blanks identified in this SDG?		/	T	
Were target compounds detected in the field blanks?			/	ſ

CC HPLC
METHOD:

# VALIDATION FINDINGS WORKSHEET

8310	8330	8151	8141	8141(con't)	8021B
A Arenanhthana	A HMY	0.15	A Dichlorica	V Examiliation	
		0-+'* '		V. Fensuliotnion	v. Benzene
B. Acenaphthylene	B. RDX	B. 2,4-DB	B. Mevinphos	W. Bolstar	CC. Toluene
C. Anthracene	C. 1,3,5-Trinitrobenzene	C. 2,4,5-T	C. Demeton-O	X. EPN	EE. Ethyl Benzene
D. Benzo(a)anthracene	D. 1,3-Dinitrobenzene	D. 2,4,5-TP	D. Demeton-S	Y. Azinphos-methyl	SSS. O-Xylene
E. Benzo(a)pyrene	E. Tetryl	E. Dinoseb	E. Ethoprop	Z. Coumaphos	RRR. MP-Xylene
F. Benzo(b)fluoranthene	F. Nitrobenzene	F. Dichlorprop	F. Nated	AA. Parathion	GG. Total Xylene
G. Benzo(g,h,i)perylene	G. 2.4.6-Trinitrotoluene	G. Dicamba	G. Sulfotep	BB. Trichloronate	
H. Benzo(k)fluoranthene	H. 4-Amino-2,6-dinitrotoluene	H. Dalapon	H. Phorate	CC. Trichlorinate	
l. Chrysene	I. 2-Amino-4,6-dinitrotoluene	I. MCPP	1. Dimethoate	DD. Trifluralin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotolune	J. MCPA	J. Diazinon	EE. Def	
K. Fluoranthene	K. 2,6-Dinitrotoluene	K. Pentachlorophenol	K. Disulfoton	FF. Prowl	
L. Fluorene	L. 2-Nitrotoluene	L 2,4,5-TP (silvex)	L. Parathion-methyi	GG. Ethion	
M. Indeno(1,2,3-cd)pyrene	M. 3-Nitrotoluene	M. Silvex	M. Ronnel		
N. Naphthalene	N. 4-Nitrotoluene		N. Malathion		
O. Phenanthrene	O		O. Chlorpyrifos		
P. Pyrene	Ġ.		P. Fenthion		
ö	σ		Q. Parathion-ethyl		
e.			R. Trichlornate		
ċ.			S. Merphos		
			T. Stirofos		
			U. Tokuthion		

Notes:

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Ge/ HPLC

**METHOD:** 

### VALIDATION FINDINGS WORKSHEET **Continuing Calibration**

Reviewer:

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Page: /

2nd Reviewer:

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

What type of continuing calibration calculation was performed? %D or RPD  $\sqrt{N}$  Were continuing calibration standards analyzed at the required frequencies?  $\frac{1}{N}$  Did the continuing calibration standards meet the %D / RPD validation criteria of  $\leq 15.0\%$ ?

xer IV Only

Were the retention times for all calibrated compounds within their respective acceptance windows? N N/A

Qualifications	Jt/Act					1º/A dut																
Associated Samples	AII + BIK					4, 5																
RT (limit)	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )	( )
%D / RPD (Limit ≤ 15.0)	16.6					12.51																
Compound						<b>9</b>																
Detector/ Column	inot on cill	J J				1																
Standard ID	9 KU768					1600 QCA1373																
Date	80/1/9	•			, ,	6	. /															
#	+					4										L						

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cover 941906 SDG #:

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

5 Page: 2nd Reviewer: Reviewer:

HPLC METHOD: GC

The calibration Factor (CF), average CF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following

average CF = sum of the CF/number of standards %RSD = 100  $^{\star}$  (S/X) CF = A/C

- A = Area of compound,
- C = Concentration of compound,
- S = Standard deviation of the CF X = Mean of the CFs

Recalculated 3.240 128.1 %RSD 3.240 Reported %RSD 128-1 **Recalculated** Average CF (initial) 806710 2387 Average CF (initial) 012908 Reported 2 3507 S CF O. S std) Recalculated 815734 24206 KELS18 CF O. Std) Reported 21206 6 Compound Naphthalene Anthracene Calibration Date 80/4/2 Standard ID 1cAL # 2 ო 4

INICI C 1SB

results.

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

gro 909129 LDC #: SDG#

## **Continuing Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

5 Reviewer: 2nd Reviewer: Page:

HPLC METHOD: GC\_

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

% Difference = 100 \* (ave. CF - CF)/ave. CF CF = A/C

CF = continuing calibration CF A = Area of compound C = Concentration of compound Where: ave. CF = initial calibration average CF

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	CF/Conc. CCV	CF/Conc. CCV	Q%	AD%
	1 & CAL862 4/12 208	Ja/ 91/ 2	Naphtha leve	5:0	5-3624	5-3624	7.2	7.2
			Anthracene	0.10	0.534Y	P.5344	6.9	6.3
2	2 & CA1873 6/16/08	6/16/08			s.3778	5-3928	8.0	8:0
		<b>4</b>	1	Ą	0.5307	0.5307	<i>/</i> .9	6./
ო								
4								

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

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1147	recon	
120	2	
	SDG #	

## VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

Page: / of / Reviewer:\_\_\_\_\_\_\_2nd reviewer:\_\_\_\_\_\_

METHOD: \_\_\_\_\_Ge\_\_\_\_HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS \* 100

Where: SF = Surrogate Found SS = Surrogate Spiked

Sample ID: # 2⁄

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
-				Reported	Recalculated	
o- Terpheny	not zpecified	52	17.9118	72	72	C
0 1						

### Sample ID:

Percent Difference			
Percent Recovery	Recalculated		-
Percent Recovery	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			

### Sample ID:

Percent Difference			
Percent Recovery	Recalculated		-
Percent Recovery	Reported		
Surrogate Found			
Surrogate Spiked			
Column/Detector			
Surrogate			

909149	per could
1 :# 201	SDG #:

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

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

HPLC ပ္ထ **METHOD:** 

The percent recoveries (%R) and relative percent differences (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation: %Recovery = 100 \* (SSC - SC)/SA

Where

RPD =(((SSCMS - SSCMSD) \* 2) / (SSCMS + SSCMSD))\*100

0 - 20 -TSB - 91 MS/MSD samples:

SC = Sample concentration SSC = Spiked sample concentration SA = Spike added MS = Matrix spike

MSD = Matrix spike duplicate

Matrix Spike Duplicate MS/MSD	Percent Recovery RPD	lc. Renorted							73 73 2. O 2.0						
Matrix spike	Percent Recovery	Recalc.	 						22	76					
Ŵ	Perct	Reported							7 7	76					
Spike Sample	$(\mathcal{V})$	MSD							515	49.6					
Spike (		MS O							SOL	52.9					
Sample	Altr )	) i 0								÷.,				-	
ike Jed	IFU	MSD							709	70.7					
Spike Added	бя 	MS V							698	69-8					
	Compound		(8015)	(8015)	(8021B)	(RSK-175)	(8151)	(8151)	(8310)	(8310)	(8330)	oluene (8330)			
	Comp		Gasoline	Diesel	Benzene	Methane	2,4-D	Dinoseb	Naphthalene	Anthracene	НМХ	2,4,6-Trinitrotoluene (8330)			-

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the percent recoveries (%K) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:		:						•		•	
	K) and rels v using the	ttive percent following ca	t differences alculation:	(RPD) of the	laboratory cor	ntrol sample a	and laborator	v control samp	ole duplicate	were recalcul	ated for th
%Recovery = 100 * (SSC - SC)/SA RPD =(({SSCLCS - SSCLCSD} * 2) / (SSCLCS + SSCLCSD))*100 LCS/LCSD samples:	/sA }*2)/(ssclcs+sscl / 68/58-L05	+ ssclcsd))*	Where	iSC = Spiked sal SA = Spike adde CS = Laboratory	SSC = Spiked sample concentration SA = Spike added LCS = Laboratory Control Sample	E	SC = Sam LCSD = Laboi	SC = Sample concentration LCSD = Laboratory Control Sample duplicate	ple duplicate		
		Spike	Sample	Spike	Spike Sample		Ş	rcsD		TCS/TCSD	QS:
Compound		Added NG/KS	1 up //4	Conce	concentration	Percent Recovery	tecovery	Percent Recovery	icovery	RPD	~
		LCSD	ہ ۱	rcs	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	-										
Diesel (8015)											
Berizene (8021B)							-				
Methane (Rsk-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)	667	20		484	72	73	73				
Anthracene (8310)	66.7	7 1		51.2	1	77	77				
HMX (8330)											
2,4,6-Trinitrotoluene (8330)			-								

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Page: Reviewer: 2nd Reviewer:		Qualifications		
SHEET ion sported results?	Compound Name	Recalculated Results Concentrations		
VALIDATION FINDINGS WORKSHEET Sample Calculation Verification ad verified for all level IV samples?		Reported Concentrations		
9/A9       VALIDATION FINDINGS WORKSHEET         Control       Sample Calculation Verification         6C       HPLC         Were all reported results recalculated and verified for all level IV samples?         Were all recalculated results for detected target compounds within 10% of the reported results?	Example: Sample ID. Concentration =	Compound		
LDC #: 190 9/ A-9 SDG #: 200 9/ A-9 METHOD: GC HPLC Y N N/A Were all reported res Were all recalculated	Concentration= (A)(Fv)(Df) (RF)(Vs or Ws)(%S/100) A= Area or height of the compound to be measured Fv= Final Volume of extract Df= Dilution Factor RF= Average response factor of the compound in the initial calibration Vs= Initial volume of the sample Ws= Percent Solid	# Sample ID		Comments:

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

<b>Project/Site Nam</b>	BRC Tronox Parcel F

Collection Date: June 10, 2008

LDC Report Date: July 23, 2008

Matrix:

Parameters: Dioxins/Dibenzofurans

Soil

Validation Level: EPA Level III & IV

Laboratory: TestAmerica, Inc.

Sample Delivery Group (SDG): F8F110177

### Sample Identification

TSB-FR-02-02-20' TSB-FR-02-02-30'\*\* TSB-FJ-02-02-10'\*\* TSB-FJ-02-02-20'\*\* TSB-FJ-02-02-30'

\*\*Indicates sample underwent EPA Level IV review

### Introduction

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

This review follows USEPA Contract Laboratory Program National Functional Guidelines for Polychlorinated Dioxins/Dibenzofurans Data Review (September 2005) as there are no current guidelines for the method stated above.

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

Blank results are summarized in Section V.

Field duplicates are summarized in Section XIV.

Samples indicated by a double asterisk on the front cover underwent EPA Level IV review. EPA Level III review was performed on all of the other samples. Raw data were not evaluated for the samples reviewed by EPA Level III 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.
- 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.
- 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 EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### III. Initial Calibration

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

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

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

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

No field blanks were identified in this SDG.

### VI. Matrix Spike/Matrix Spike Duplicates

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

### VII. Laboratory Control Samples (LCS)

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

### VIII. Regional Quality Assurance and Quality Control

Not applicable.

### IX. Internal Standards

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

Sample	Internal Standards	%R (Limits)	Compound	Flag	A or P
8169351MB	<sup>13</sup> C-1,2,3,4,7,8-HxCDF	38 (40-135)	1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF	J (all detects) UJ (all non-detects)	Ρ

### X. Target Compound Identifications

All target compound identifications were within validation criteria for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### XII. System Performance

The system performance was acceptable for samples on which EPA Level IV review was performed. Raw data were not evaluated for the samples reviewed by EPA Level III criteria.

### XIII. Overall Assessment of Data

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

### XIV. Field Duplicates

No field duplicates were identified in this SDG.

BRC Tronox Parcel F Dioxins/Dibenzofurans - Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Dioxins/Dibenzofurans - Laboratory Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

BRC Tronox Parcel F Dioxins/Dibenzofurans - Field Blank Data Qualification Summary - SDG F8F110177

No Sample Data Qualified in this SDG

### VALIDATION COMPLETENESS WORKSHEET

LDC #: 19091A21 SDG #: F8F110177 Laboratory: Test America

### Level III/IV

Date: <u>7/19/o</u> s
Page: <u>1</u> of
Reviewer: K
2nd Reviewer:
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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
Ι.	Technical holding times	Å	Sampling dates: 6/10/08
П.	GC/MS Instrument performance check	4	
.	Initial calibration	4	
IV.	Routine calibration// <del>CV</del>	A	
V.	Blanks	4	
VI.	Matrix spike/Matrix spike duplicates	N	dient specified
VII.	Laboratory control samples	Å	Les
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	5N	
<b>X</b> .	Target compound identifications	4	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	<u></u>	Not reviewed for Level III validation.
XII.	System performance	Å	Not reviewed for Level III validation.
XIII.	Overall assessment of data	4	
XIV.	Field duplicates	N	
XV.	Field blanks	И	

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 Level IV validation

1	TSB-FR-02-02-20'	11	8169351UB	21	31	
2	TSB-FR-02-02-30'**	12		22	32	
3	TSB-FJ-02-02-10'**	13		23	33	
4	TSB-FJ-02-02-20'**	14		24	34	
5	TSB-FJ-02-02-30'	15		25	35	
6		16		26	36	
7		17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

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### VALIDATION FINDINGS CHECKLIST

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Page:<u></u>of<u></u> Reviewer:<u></u> 2nd Reviewer:

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

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

### LDC #: 190914>1 VA SDG #: FSF110177

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### VALIDATION FINDINGS CHECKLIST

Page:_	2-of3
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Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	/		_	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
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 $\geq$ 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 $\geq$ 2.5?				
Does the maximum intensity of each specified characteristic ion coincide within $\pm$ 2 seconds (includes labeled standards)?				-
For PCDF identification, was any signal (S/N $\geq$ 2.5, at $\pm$ 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?			1	·
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	~			
XII. System performance	ı			
System performance was found to be acceptable.	/			
XIII. Overall assessment of data	 1	 T		
Overall assessment of data was found to be acceptable.	$\square$			
XIV. Field duplicates	T			
Field duplicate pairs were identified in this SDG.		/		

### VALIDATION FINDINGS CHECKLIST

Page: <u>3</u>of <u>3</u> Reviewer: <u>J</u> 2nd Reviewer: <u>A</u>

Validation Area	Yes	No	NA	Findings/Comments
Target compounds were detected in the field duplicates,				
XV. Field blanks				
Field blanks were identified in this SDG.			F	
Target compounds were detected in the field blanks.			/	

DXN-SW90.IV version 1.0

LDC #: 19091A21 SDG #: F8F110177

# 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	Н. 2,3,7,8-ТСDF	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	0.1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

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## VALIDATION FINDINGS WORKSHEET Internal Standards

Page: 1 of 1 2nd Reviewer:\_\_\_ 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". <u>Y (N) N/A</u> Are all internal standard recoveries were within the 40-135% criteria? W N/A Was the S/N ratio all internal standard neaks > 10?

H						
*	Date	Lab ID/Reference	Internal Standard	% Recove	% Recovery (Limit: 40-135%)	Qualifications
		8169351MB	E	25	( <u>1</u> 2-ch)	T/47 /P (K-N Z)
					)	
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		Internal Standards	Check Standard Used		Recovery Standards	Check Standard Used
×	<sup>13</sup> C-2,3,7,8-TCDF	SDF		K. <sup>13</sup> C-1.2.3.4-TCDD	-TCDD	
ß	+	DD		—	<sup>13</sup> C-1,2,3,7,8,9-HxCDD	
Ö	+	PeCDF		M.		
ġ	+	PeCDD		Ž		
וש		3-HxCDF		Ō		
цÏ	+	3-HxCDD		P.		
יס :	┿	7,8-HpCDF		ď		
Ŧ.	+	7,8-HpCDD		H		

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LDC #: 19071271 SDG #: P8 F110177

Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

( of / Page: 2nd Reviewer: Reviewer:

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

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

 $\label{eq:RF} RRF = (A_{*})(C_{*})/(A_{*})(C_{*})$  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,

 $\label{eq:associated} \begin{array}{l} A_{e} = Area \mbox{ of associated internal standard } \\ C_{e} = Concentration \mbox{ of internal standard } \\ X = Mean \mbox{ of the } RRFs \end{array}$ 

Recalculated N . 1 5.0 ľ, a フォ à %RSD Reported 2 0 %RSD n U 5.0 ہ م 10.2 Recalculated ( ( S 2, std) 0.87 D. 82 0.93 RRF ( CS⇒ std) Reported 0.87 0.93 0.82 78: Recalculated Average RRF 297.0 0.820 415.0 0.844 441. (initial) Average RRF (Initial) Reported 0.798 0,844 0.821 516.0 127. Compound (Reference Internal Standard) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDF (1°C-2,3,7,8-TCDF) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2.3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) 2,3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) OCDF (13C-OCDD) OCDF ("C-OCDD) OCDF ("C-OCDD) Calibration 50/91/7 Date Standard ID 104L \* ~

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.

SDG #: F8F110177 LDC #: 19091421

## **Routine Calibration Results Verification** VALIDATION FINDINGS WORKSHEET

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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\_)(C\_)/(A\_)(C\_)

ave. RRF = initial calibration average RRF RRF = continuing calibration RRF Where:

 $A_x = Area of compound, C_x = Concentration of compound,$ 

 $A_{\mathbf{k}} = Area$  of associated internal standard  $C_{\mathbf{k}} = Concentration of internal standard$ = Concentration of internal standard

Recalculated λ 8 7 200 6 7.3 8.4 1 Г 0% 'n N Ø Reported 4 エ 0.Y 7.4 Ŋ \$ T 0% Recalculated 0.82 0.82 0.89 0.78 0.00 0.8 RRF (CC) 84 10,0 .68 5 0.82 5 Reported 0.80 0.89 0. <u></u> . 68 0.78 0.82 0.91 RRF (CC) 0.8 Average RRF (initial) 616.0 0.913 861.0 0.844 0.844 0.798 124 4 0.821 28.0 Compound (Reference Internal Standard) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1,2,3,4,6,7,8-HpCDD (<sup>13</sup>C-1,2,4,6,7,8,-HpCDD) 1.2.3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>13</sup>C-1,2,3,6,7,8-HxCDD) 1,2,3,6,7,8-HxCDD (<sup>3</sup>C-1,2,3,6,7,8-HxCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDF (\*C-2,3,7,8-TCDF) 2,3,7,8-TCDD (<sup>13</sup>C-2,3,7,8-TCDD) 2,3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) 2.3,7,8-TCDF (<sup>13</sup>C-2,3,7,8-TCDF) OCDF (13C-OCDD) OCDF (13C-OCDD) OCDF ("C-OCDD) 80/08/9 Calibration 80/22/9 Date Standard ID Aredore 570630B \* 2 ო

C:\WPDOCS\WRK\DIOXIN90\CONCLC90.21

recalculated results.

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

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LDC #:	SDG #:_

## VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Page: <u>lof l</u> Reviewer: <u>\*</u> 2nd Reviewer: <u>\*</u>

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

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

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

RPD = I LCS - LCSD I \* 2/(LCS + LCSD)

LCS = Laboraotry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

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LCS ID:

Spil	ke	Spiked S	sample	ICS	s	1 CSD			CSD
Added	ed な)	Concentration	tration در )	Percent Recovery	tecovery	Percent Recovery	ecovery	RPD	Ο
I CS	ر ۱ دیر		0 1 CSD	Renorted	Recalc	Renorted	Recalc	Denorted	Decalculated
8		16.7		83	84				
3		701		101	101				
		95.4		So	22				
Ŷ		89.2		63	kel				
<b>Je</b> 0		(83		45	do				
	-								
					-		-		

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.

	╟								
Descriptor	ptor Accurate mass <sup>(a)</sup>	lon ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(a)</sup>	lon ID	Elemental Composition	Analyte
	303.9016	Σ	C, H, "CILO	TCDF	4	407.7818	0⊤W		11-05-1
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C10	TCDF		409.7788	4+M		
	315.9419	Σ	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>ss</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	Σ		
	317.9389	M+2	"C <sub>12</sub> H, "Cl <sub>3</sub> "ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C, H <sup>35</sup> Cl, TClO	HDCDF (0)
	013,0000	Σ	C <sub>12</sub> H <sup>*</sup> <sup>sc</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2		HeCDD
	321.8936	M+2	C12H3*CI3*C102	TCDD		425.7737	M+4	C. H <sup>w</sup> Cl. 7Cl. 0	HUCDI
	331.9368	Σ	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> *Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169		13CH*CI.37CIO	
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>3</sub> 7ClO <sub>2</sub>	TCDD (S)		437.8140			
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>ac</sup> Cl <sub>5</sub> <sup>ar</sup> ClO	HXCDPE	_	479.7165	M+4		
	[354.9792]	LOCK	C <sub>6</sub> F <sub>13</sub>	PFK		[430.9728]	TOCK		
~	339,8597	M+2	C.,H.ªCI.WCIO	PaCDE	Ľ	444 7400			
	341.8567				>	441.7420		C12 ~ CI7 ~ CIO	OCDF
	351,9000		13C H 35C 1 37C 10			440./089			OCDF
	353.8970					45/./377		C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	ocpp
	355 8546					459.7348			ocbb
	357 8516			rectuu		469.7780		<sup>113</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)
	01.00.100		C12H3*C13*C12O2	PecDD	-	471.7750	M+4	<sup>13</sup> C, <sup>35</sup> Cl, <sup>37</sup> Cl, O,	ocdd (S)
	30/.8949		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>2</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)		513.6775	M+4	C. SCI. SCLO	
	369.8919		<sup>13</sup> C <sub>12</sub> H <sub>3</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	PeCDD (S)		[422.9278]	LOCK	C.F.	PEK I
	409.7974		C12H3*CI87CIO	HPCDPE		•			-
	[354.9792]	LOCK	C F 3	PFK					
e	373.8208	M+2	C., H. accl. arClO	HACDE					
	375.8178	-	C.,H.*CI,vCI,O	HXCDF			_		
	383.8639	Σ	<sup>13</sup> C.H. <sup>35</sup> CLO	HYCDF (S)					
	385.8610		<sup>13</sup> Ci <sub>2</sub> H <sup>5</sup> *Cl <sup>3</sup> 7ClO	HXCDF (S)					
	389.8156		C.,H.ªCI, vCIO,	HxCDD					
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>ac</sup> Cl <sub>2</sub> <sup>37</sup> Cl <sub>3</sub> O <sub>5</sub>	HXCDD					
	401,8559		<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)					
	403.8529		<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>36</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HxCDD (S)					
	445.7555		C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDPE					
	[430.9728]	Lock	C,F1,	PFK			<u> </u>		
(B)	The following nuclidic masses were used:	es were used:							

H = 1.007825C = 12.0000000  $^{13}$ C = 13.003355 F = 18.9984

O = 15.994915 <sup>35</sup>Cl = 34.968853 <sup>37</sup>Cl = 36.965903

S = internal/recovery standard

C:\WPDOCS\WRK\DIOXIN90\TCI90.21

lons Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

LDC #: 19091421 SDG #: F8F110177

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: <u>l</u>of <u>l</u> Reviewer: <u>A</u> 2nd reviewer: <u>A</u>

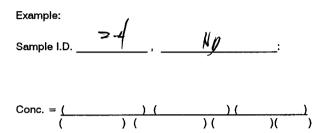
### METHOD: HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)



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

Conce	ntratior	$p = (A_{*})(I_{*})(DF) (A_{*})(RRF)(V_{o})(%S)$
A <sub>x</sub>	=	Area of the characteristic ion (EICP) for the compound to be measured
A <sub>is</sub>	=	Area of the characteristic ion (EICP) for the specific internal standard
l <sub>s</sub>	=	Amount of internal standard added in nanograms (ng)
V,	=	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.



#Reported<br/>ConcentrationCalculated<br/>ConcentrationQualificationImage: Sample IDCompoundImage: Sample IDQualificationImage: Sample IDImage: Sample ID