LDC Report# 19091A3a

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name:	BRC Tronox Parcel F

Collection Date: June 10, 2008

LDC Report Date: October 20, 2008

Matrix:

Soil

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.

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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.

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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. 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.

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.

*Removed above Continuing calibration (%D) finding.

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.

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

No Sample Data Qualified in this SDG

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

VALIDATION COMPLETENESS WORKSHEET

Level III/IV

LDC #: <u>19091A3a</u> SDG #: <u>F8F110177</u> Laboratory: <u>Test America</u> Date: 7/19/08 Page: ______ Reviewer: ______ 2nd Reviewer: ______

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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
11.	GC/ECD Instrument Performance Check	Δ	· · · · · · · · · · · · · · · · · · ·
111.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	Sat	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	Δ	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 $< 0 1 l_{-}$

				_		_	·····
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
. Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.	Ĺ	ł		
II. GC/ECD Instrument performance check				
Was the instrument performance found to be acceptable?		<u>t</u>		
III. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?		ł		
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?	/	Ł		
Were the required standard concentrations analyzed in the initial calibration?		\mathbf{F}		
IV. Continuing calibration				
What type of continuing calibration calculation was performed?%D or%R	/			
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%?	Via	AT JA		
Were all the retention times within the acceptance windows?				
V. Blanks				
Was a method blank associated with every sample in this SDG?	\leq		[
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.				
YL Surrogate spikes				
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?			1	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?				

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VALIDATION FINDINGS CHECKLIST

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

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?	/	Í		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?				
VIII, Laboratory control samples		r		P
Was an LCS analyzed for this SDG?		<u> </u>		
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			<u> </u>	
Were the retention times of reported detects within the RT windows?				
XI. Compound quantitation/CROLs				
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.				
XIII: Overall assessment of data			;	
Overall assessment of data was found to be acceptable.	\land			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.			\nearrow	
KV: Field:blanks				
Field blanks were identified in this SDG.		7	T	
Farget compounds were detected in the field blanks.			\nearrow	

VALIDATION FINDINGS WORKSHEET

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

	<u> </u>	Q. Endrin ketone	Y. Arocior-1242	GG.
B. beta-BHC J. 4,4'-D	ŪE	R. Endrin aldehyde	Z. Aroclor-1248	HH.
C. delta-BHC K. Endri	<u> </u>	S. alpha-Chlordane	AA. Araclor-1254	
D. gamma-BHC L. Endo	suffan II	T. gamma-Chlordane	BB. Arociot-1260	
E. Heptachlor M. 4,4-D	00	U. Toxaphene	CC. DB 608	KK.
F. Aldrin N. Endo:	isulfan sulfate	V. Aroclor-1016	DD. DB 1701	It.
G. Heptachlor epoxide O. 4,4-D	DDT	W. Arocior-1221	E.	MM.
H. Endosulfan I P. Metho	oxychlor	X. Aroclor-1232	FF.	NN.

Notes:

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Initial Calibration Calculation Verification VALIDATION FINDINGS WORKSHEET

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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:

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

CF = A/C

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

				Reported	Recalculated	Reported	Recalculated	Reported	Recatculated
#	Standard ID	Calibration Date	Compound	CF D. D.25 std)	CF DIOM std)	Average CF (initial)	Average CF (initial)	%RSD	%RSD
-	1042	80/91/9	endowhan I chA	530216040	53021604C	04/566015	04126015	3.148	3-148
			me thosey chiel &	161496680	16149640	029262251	orgelees1	6.2075	12577
\square		ŗ	δ						
ы			1 chB	28500 1720	allanstr	17553334	z/AEESEL® 2	a. 96 58	2596-2 1
		<u>.</u>	~	44217640	CH9LIZAA	ax ere h	UNE 22.244	6.0188	8/0.2 8
. 67							-		
4									

results.

LDC #: 19091430 SDG#: ALL CONT

Continuing Calibration Results Verification VALIDATION FINDINGS WORKSHEET

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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 ۵% 0 3.1 0.0 . н Reported 0 5 N N 0.8 0% **Recalculated** CF/Conc. CCV 0.022 0.0057 0.0252 0.039 CF/Conc. CCV 0.0X2 Reported 63000 0.0252 - JXO 'O Average CF(Ical)/ CCV Conc. USEO O chA metho ity chlor Compound endosu/pan 6/18/08 c/18/08 Calibration Date £081080 k cdLoby Standard ID 4 ო 2 *

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. Comments:

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VALIDATION FINDINGS WORKSHEET Surrogate Results Verification



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:

Percent Percent Percent Surrogate Surrogate Recovery Column Spiked Found Recovery Difference Surrogate Reported Recalculated ChA Tetrachioro-m-xylene 0.01839 0.02 0 92 92 Tetrachioro-IT-xylene DCB ¥ 84 J 0.01682 О 84 Decachlorobiphenyl Decachlorobiphenyl

Sample ID:

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachioro-m-xylene	:					
Tetrachioro-m-xyiene						
Decachiorobiphenyl						
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		<u> </u>				
Tetrachloro-m-xylene						
Decachlorobiphenyl						
Decachlorobiphenyl						

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

Page: / of 2nd Reviewer: Reviewer:

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

Where: SSC = Spiked sample concentration SA = Spike added

SC = Concentration

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

MS = Matrix spike percent recovery 01-80-15 l 75 B MS/MSD samples:__

MSD = Matrix spike duplicate percent recovery

	• ¥	pike Jded	Sample Concentration	Spiked	Sample	Matrix	t Spike	Matrix Spik	e Duplicate	W	/WSD
Compound	~		())		Percent	Recovery	Percent	Recovery		Co
	WS	MSD		SW	USM	Danottod					
							Vecalc.	керопеа	Kecalc.	Reported	Recalculated
gamma-bhC	17.7	レント		15:6	15.3	ž	88	22	87	2.17	2.0
4,4'-DDT	1	>		15:6	16.3	ž	Z	32	6		1.1
							<i>,</i>	2	2	7:7	1-7
	7.0/										
			·								

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% of the recalculated results.

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SDG # pur com	1 A34	aboratory C	control San	VALIDATIC <u>nple/Labor</u> z	ON FINDINGS	WORKSHEE1 <u>Sample Dupli</u>	r cate Results	Verification	Page: 6f Reviewer: 2nd Reviewer: 2	No.
METHOD: GC Pes	sticides/PCBs	s (EPA SW 84	6 Method 808	1/8082)						
The percent recovic compounds identifi	eries (%R) ar ed below usir	nd Relative P€ ng the followin	srcent differen ig calculation:	ice (RPD) of t	he laboratory cont	rol sample and l	aboratory control	sample duplicat	e were recalculated for th	Q
% Recovery = 100* (SS	sc-sc)/sA		Where: SS SA	SC = Spiked samp	ile concentration	õ	C = Concentration			
RPD = I LCS - LCSD I * LCS/LCSD sample	. 2/(LCS + LCSD) <u>8164 2e</u>		:S = Laboratory α	ontrol sample percent i	recovery LC	SSD = Laboratory cor	itrol sample duplicat	e percent recovery	
	S A	spike ddad	Spiked	d Sample		cs	۲ L	SD SS	LCS/LCSD	
Compound	2))	14×		se //Y	Percent	Recovery	Percent	Recovery	RPD	
	rcs	LCSD	rcs	rcsp	Reported	Recalc.	Reported	Recalc.	Reported Recalc	
gamma-BHC	16.7	NA NA	15:0	NA NA	90	90				
4,4'-DDT	~	1	16.8	1	101	/0/	WF			
Comments: Refer	<u>to Laboratory</u> <u>e within 10.0%</u>	/ Control Sam	<u>ple/Laboratory</u> vulated results.	Control Same	ole Duplicate findir	Ids worksheet for	r list of qualificati	ons and associat	ted samples when reporte	
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LDC #: 19091A30 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		;	
Conc. = <u>(</u> (
=			
	ND		

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

Note: