

## 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.

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.
- UU 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.

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.

**\*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

LDC #: 19091A3a  
 SDG #: F8F110177  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 7/19/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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
I.	Technical holding times	Δ	Sampling dates: 6/10/08
II.	GC/ECD Instrument Performance Check	Δ	
III.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	A SW	ICV = 15
V.	Blanks	Δ	
VI.	Surrogate spikes	Δ	
VII.	Matrix spike/Matrix spike duplicates	A	TSB-GJ-08-10
VIII.	Laboratory control samples	A	LCS
IX.	Regional quality assurance and quality control	N	
Xa.	Floril 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      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

SOLK

1	TSB-FR-02-02-20'	11	F8F/160000-164	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	



LDC #: 1909/A3a  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: F7  
 2nd Reviewer: 9

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

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
<b>II. GC/ECD Instrument performance check</b>				
Was the instrument performance found to be acceptable?	/			
<b>III. Initial calibration</b>				
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?			/	
Were the RT windows properly established?	/			
Were the required standard concentrations analyzed in the initial calibration?	/			
<b>IV. Continuing calibration</b>				
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 recoveries 85-115%?	/			
Were all the retention times within the acceptance windows?	/			
<b>V. Blanks</b>				
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.			/	
<b>VI. Surrogate spikes</b>				
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?			/	

LDC #: 19091A3C  
 SDG #: All cones

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. Matrix spike/Matrix spike duplicates</b>				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
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?	/			
<b>VIII. Laboratory control samples</b>				
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?	/			
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	
<b>X. Target compound identification</b>				
Were the retention times of reported detects within the RT windows?			/	
<b>XI. Compound quantitation/CRQLs</b>				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions, dry weight factors, and clean-up activities applicable to level IV validation?	/			
<b>XII. System performance</b>				
System performance was found to be acceptable.	/			
<b>XIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
<b>XIV. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.		/		
Target compounds were detected in the field duplicates.			/	
<b>XV. Field blanks</b>				
Field blanks were identified in this SDG.		/		
Target compounds were detected in the field blanks.			/	

# VALIDATION FINDINGS WORKSHEET

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

A. alpha-BHC	I. 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	JJ.
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. DB 608	KK.
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. DB 1701	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE.	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF.	NN.

Notes: \_\_\_\_\_

LDC #: 19091A3a  
 SDG #: 19091A3a

**VALIDATION FINDINGS WORKSHEET**  
**Initial Calibration Calculation Verification**

Page: 1 of 1  
 Reviewer: YFP  
 2nd Reviewer: SC

METHOD: GC HPLC

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 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

#	Standard ID	Calibration Date	Compound	Reported		Recalculated		Reported		Recalculated	
				CF 0.025 std)	CF 0.025 std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	ICAL	6/16/08	endosulfan / chA methyloxychlor ↓	530216040	530216040	510995140	510995140	3.14887	3.14887	3.14887	3.14887
				161496680	161496680	152272620	152272620	6.2575	6.2575	6.2575	6.2575
2			chB ↓	285001720	285001720	273533412	273533412	8.96584	8.96584	8.96584	8.96584
				44217640	44217640	42223360	42223360	6.01863	6.01863	6.01863	6.01863
3											
4											

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

LDC #: 1909/A3a  
 SDG #: pc cmt

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

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

METHOD: GC /          HPLC         

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 =  $100 \cdot \frac{\text{ave. CF} - \text{CF}}{\text{ave. CF}}$   
 CF = A/C  
 Where: ave. CF = initial calibration average CF  
 CF = continuing calibration CF  
 A = Area of compound  
 C = Concentration of compound

#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	KCAL064	6/18/08	endosulfan / chA	0.0259	3.7	0.0259	3.7	
			methoxychlor	0.0252	0.8	0.0252	0.8	
2	KCAL080	6/18/08	↓	0.0252	1.0	0.0252	1.0	
			↓	0.0257	2.7	0.0257	2.7	
3								
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 #: 19091A3a  
 SDG #: pu cover

**VALIDATION FINDINGS WORKSHEET**  
**Surrogate Results Verification**

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

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

Sample ID: # 2

Surrogate	Column	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
Tetrachloro-m-xylene	ch A	0.02	0.01839	92	92	0
Tetrachloro-m-xylene	DCB ↓	↓	0.01682	84	84	0
Decachlorobiphenyl						
Decachlorobiphenyl						

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

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

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

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

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

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

Notes: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

LDC #: 19091 ABC  
 SDG #: per card

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

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

**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 the following calculation:

% Recovery =  $100 \times (SSC-SC)/SA$       Where: SSC = Spiked sample concentration      SC = Concentration  
 SA = Spike added  
 RPD =  $100 \times |MS - MSD| / (MS + MSD)$       MS = Matrix spike percent recovery      MSD = Matrix spike duplicate percent recovery  
 MS/MSD samples: TSB-9J-08-10

Compound	Spike Added ( )		Sample Concentration ( )	Spiked Sample Concentration ( )		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
gamma-BHC	17.7	17.5	-	15.6	15.3	88	88	87	87	2.0	2.0
4,4'-DDT	↓	↓	-	15.6	16.3	88	88	93	93	4.4	4.4

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.

LDC #: 19091A34

SDG #: per count

**VALIDATION FINDINGS WORKSHEET**

**Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification**

Page: 6 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

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 =  $100 * (SSC-SC)/SA$

Where: SSC = Spiked sample concentration  
SA = Spike added

SC = Concentration

RPD =  $1 LCS - LCSD \cdot 2 / (LCS + LCSD)$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8168164-LCS

Compound	Spike Added (ug/kg)		Spiked Sample Concentration (ug/kg)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
gamma-BHC	16.7	NA	15.0	NA	90	90				
4,4'-DDT	↓	↓	16.8	↓	101	101	NA			

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 #: 19091A3a  
 SDG #: su cover

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

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

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

Y N N/A  
 Y N N/A

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. = ( \_\_\_\_\_ )  
 ( \_\_\_\_\_ )

=

ND

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

Note: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_