



**LABORATORY DATA CONSULTANTS, INC.**

7750 El Camino Real, Suite 2L Carlsbad, CA 92009 Phone: 760/634-0437 Fax: 760/634-0439

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

August 6, 2008

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:**

<b><u>SDG #</u></b>	<b><u>Fraction</u></b>
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



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

**Project/Site Name:** BRC Tronox Parcel F  
**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.
- 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/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 ( $\geq 0.05$ )	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)	Iodomethane	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)	Iodomethane	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 TIC (RT in minutes)	Reported Concentration	Modified Final 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)	P

## 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-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3,5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene 4-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)	P



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-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3,5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene 4-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)	P
TSB-FJ-02-02-20**	1,4-Dichlorobenzene-d4	171259 (187131-748522)	1,1,2,2-Tetrachloroethene 1,2,3-Trichlorobenzene 1,2,3-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3,5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene 4-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)	P

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-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3,5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene 4-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)	P

### **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	Iodomethane	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-Trichloropropane 1,2,4-Trichlorobenzene 1,2,4-Trimethylbenzene 1,2-Dichlorobenzene 1,2-Dibromo-3-chloropropane 1,3,5-Trimethylbenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2-Chlorotoluene 4-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)	P	Internal standards (area)

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

<b>SDG</b>	<b>Sample</b>	<b>Compound TIC (RT in minutes)</b>	<b>Modified Final Concentration</b>	<b>A or P</b>
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 #: 19091A1

## VALIDATION COMPLETENESS WORKSHEET

SDG #: F8F110177

Level III/IV

Laboratory: Test America

Date: 7/19/08

Page: 1 of 1

Reviewer: *fl*2nd Reviewer: *fl*

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
I.	Technical holding times	$\Delta$	Sampling dates: 6/10/08
II.	GC/MS Instrument performance check	$\Delta$	
III.	Initial calibration	$\Delta$	% RSD, 12 20.990
IV.	Continuing calibration/ICV	SW	ICV $\leq$ 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	
X.	Internal standards	SW	
XI.	Target compound identification	$\Delta$	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	$\Delta$	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	$\Delta$	Not reviewed for Level III validation.
XIV.	System performance	$\Delta$	Not reviewed for Level III validation.
XV.	Overall assessment of data	$\Delta$	
XVI.	Field duplicates	N	
XVII.	Field blanks	SW	TB = 6

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

*SOIL + water*

1	TSB-FR-02-02-20'	11	F8F120000-446	21	816446	31
2	TSB-FR-02-02-30'	12	F8F200000-125	22	8172125	32
3	TSB-FJ-02-02-10'	13	F8F200000-361	23	8172361 nona	33
4	TSB-FJ-02-02-20'	14		24		34
5	TSB-FJ-02-02-30'	15		25		35
6	3 = Nonana TB-2 6/10/08	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 #: 19091A1  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: F7  
 2nd Reviewer: [Signature]

Method: Volatiles (EPA SW 846 Method 8260B)

Validation Area	Yes	No	NA	Findings/Comments
<b>Technical Holding Times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>BFB Instrument Performance</b>				
Were the BFB performance results reviewed and found to be within the specified criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all samples analyzed within the 12 hour clock criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria of > 0.990?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) > 0.05?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Standardization</b>				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) and relative response factors (RRF) within method criteria for all CCCs and SPCCs?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) ≤ 25% and relative response factors (RRF) ≥ 0.05?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed at least once every 12 hours for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Surrogate Recovery</b>				
Were all surrogate %R within QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
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?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>Matrix Spike/Duplicate</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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>Control Samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 19091A1  
 SDG #: all cover

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X Internal Standards</b>				
Were internal standard area counts within -50% or +100% of the associated calibration standard?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI Sample Compound Identification</b>				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII Sample Quantification (CQLs)</b>				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIII Library Reference Spectra (LRS)</b>				
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were relative intensities of the major ions within $\pm 20\%$ between the sample and the reference spectra?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>System Performance</b>				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>Overall Assessment</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XIV Field Duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field duplicates.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>XV Field Blanks</b>				
Field blanks were identified in this SDG.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	



# TARGET COMPOUND WORKSHEET

METHOD: VOA (EPA SW 846 Method 8260B)

A. Chloromethane*	U. 1,1,2-Trichloroethane	OO. 2,2-Dichloropropane	III. n-Butylbenzene	CCCC.1-Chlorohexane
B. Bromomethane	V. Benzene	PP. Bromochloromethane	JJ. 1,2-Dichlorobenzene	DDDD. Isopropyl alcohol
C. Vinyl chloride**	W. trans-1,3-Dichloropropene	QQ. 1,1-Dichloropropene	KKK. 1,2,4-Trichlorobenzene	EEEE. Acetonitrile
D. Chloroethane	X. Bromoform*	RR. Dibromomethane	LLL. Hexachlorobutadiene	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-Trichlorobenzene	HHHH. 1,4-Dioxane
G. Carbon disulfide	AA. Tetrachloroethene	UU. 1,1,1,2-Tetrachloroethane	OOO. 1,3,5-Trichlorobenzene	IIII. Isobutyl alcohol
H. 1,1-Dichloroethene**	BB. 1,1,2,2-Tetrachloroethane*	VV. Isopropylbenzene	PPP. trans-1,2-Dichloroethene	JJJJ. Methacrylonitrile
I. 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-Trimethylbenzene	UUU. 1,2-Dichlorotetrafluoroethane	Oooo.
N. 1,1,1-Trichloroethane	HH. Vinyl acetate	BBB. 4-Chlorotoluene	VVV. 4-Ethyltoluene	PPPP.
O. Carbon tetrachloride	II. 2-Chloroethylvinyl ether	CCC. tert-Butylbenzene	WWW. Ethanol	QQQQ.
P. Bromodichloromethane	JJ. Dichlorodifluoromethane	DDD. 1,2,4-Trimethylbenzene	XXX. Di-isopropyl ether	RRRR.
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	TTTT.
S. Trichloroethene	MM. 1,2-Dibromo-3-chloropropane	GGG. p-Isopropyltoluene	AAA. Ethyl tert-butyl ether	UUUU.
T. Dibromochloromethane	NN. Methyl ethyl ketone	HHH. 1,4-Dichlorobenzene	BBB. tert-Amyl methyl ether	VVVV.

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



















**Volatile Internal Standards**

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane ✓
1,1,2-Trichloroethane	1,2-Dibromoethane	1,2,3-Trichlorobenzene ✓
1,1-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichloropropane ✓
1,1-Dichloroethene	1-Chlorohexane	1,2,4-Trichlorobenzene ✓
1,1-Dichloropropene	<del>Bromoforn</del>	1,2,4-Trimethylbenzene ✓
1,2-Dichloroethane	Chlorobenzene	1,2-Dichlorobenzene ✓
1,2-Dichloropropane	Dibromochloromethane	1,2-Dibromo-3-chloropropane ✓
2,2-Dichloropropane	Ethylbenzene	1,3,5-Trimethylbenzene ✓
Acetone	m,p-Xylene	1,3-Dichlorobenzene ✓
Benzene	o-Xylene	1,4-Dichlorobenzene ✓
Bromochloromethane	Styrene	2-Chlorotoluene ✓
Bromodichloromethane	Tetrachloroethene	4-Chlorotoluene ✓
Bromomethane	1,1,2-Trichloroethane	Bromobenzene ✓
Carbon tetrachloride	<del>Toluene</del>	<del>Hexachlorobutadiene</del>
Chloroethane	<del>trans-1,3-Dichloropropene</del>	Isopropylbenzene ✓
Chloroform	2-Nitropropane	<del>Methyl isobutyl ketone</del>
Chloromethane	4-Methyl-2-pentanone	n-Butylbenzene ✓
cis-1,2-Dichloroethene	2-Hexanone	n-Propylbenzene ✓
cis-1,3-Dichloropropene	Dimethyl disulfide	Naphthalene
Dibromomethane	Xylenes (total)	<del>p-Isopropyltoluene, p-cymene</del>
Dichlorodifluoromethane		sec-Butylbenzene ✓
Methylene chloride		tert-Butylbenzene ✓
Methyl-tert-butyl ether		1,3,5-Trichlorobenzene
2-Butanone		Nonanal
Trichloroethene		Bromoforn ✓
<del>Toluene</del>		
trans-1,2-Dichloroethene		
trans-1,3-Dichloropropene		
Trichlorofluoromethane		
Vinyl chloride		
Carbon disulfide		

- Iodomethane
- Acetonitrile
- Vinyl Acetate
- 1,1,2-Trichloro-1,1,2-Trifluoroethane
- Ethanol
- 3,3-Dimethylpentane
- 2,3-
- 2,2-
- 2,4-
- 2,2,3-Trimethylbutane
- 3-Ethylpentane
- 2-Methylhexane
- 3- ↓
- Heptane
- 1,2-Dichloroethene (total)

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

**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:

$RRF = (A_s)(C_w)/(A_w)(C_s)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$

$A_s$  = Area of compound,  
 $C_s$  = Concentration of compound,  
 $S$  = Standard deviation of the RRFs  
 $X$  = Mean of the RRFs

$A_w$  = Area of associated internal standard  
 $C_w$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (SD std)	RRF (SD std)	RRF (SD std)	Average RRF (Initial)	%RSD	Average RRF (Initial)	%RSD	
1	ICAL-X	6/9/08	Vinyl chloride (1st internal standard)	0.34552	0.34552	0.33147	0.33147	5.136	5.136		
			Ethyl Benzene (2nd internal standard)	2.30191	2.30191	2.19908	2.19908	6.139	6.139		
			1,2-Dichlorobenzene (3rd internal standard)	1.29993	1.29993	1.28078	1.28078	5.32652	5.33		
2	ICAL-X BR	6/12/08	2,2-Dimethyl Pentane (1st internal standard)	0.52673	0.52673	0.53039	0.53039	4.99617	4.99617		
			(2nd internal standard)								
			(3rd internal standard)								
3			(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 results.

LDC #: 19091A)  
 SDG #: per cover

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

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

**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 =  $100 \cdot (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$   
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF  
 RRF = continuing calibration RRF  
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $A_{is}$  = Area of associated internal standard  
 $C_{is}$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	XCAL2316	6/12/08	Vinyl Chloride (1st internal standard)	0.33747	0.30844	8.60370	0.30844	8.6037
			Ethyl Benzene (2nd internal standard)	2.1908	2.37076	7.80675	2.37076	7.80675
			1,2-DCB (3rd internal standard)	1.26078	1.38777	8.35372	1.38777	8.3537
2			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					
3			(1st internal standard)					
			(2nd internal standard)					
			(3rd internal standard)					
4			(1st 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 #: 19091A1  
 SDG #: per cover

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

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

**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: # 2

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Toluene-d8	50.0	53.0191	106	106	0
Bromofluorobenzene	↓	55.9184	112	112	↓
1,2-Dichloroethane-d4	↓	59.4200	119	119	↓
Dibromofluoromethane	↓	55.0604	110	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  
 SDG #: per cover

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

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

**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:

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

RPD =  $100 * MSC - MSDC / (MSC + MSDC)$       MSC = Matrix spike percent recovery      MSDC = Matrix spike duplicate percent recovery

MS/MSD sample: 728

Compound	Spike Added		Sample Concentration	Spiked Sample Concentration		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery	Recalc.	Percent Recovery	Recalc.	Reported	Recalculated
1,1-Dichloroethene	65.0	64.9	ND	67.3	71.4	106	106	110	110	3.0	3.0
Trichloroethene				73.4	72.0	113	113	111	111	2.0	2.0
Benzene				66.2	65.2	102	102	100	100	1.6	1.6
Toluene				68.7	67.7	106	106	104	104	1.6	1.6
Chlorobenzene				66.0	65.4	101	101	101	101	0.83	0.83

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 #: 19091 A1  
 SDG #: per contract

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Sample Results Verification**

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

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

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

% Recovery =  $100 \cdot \text{SSC}/\text{SA}$  Where: SSC = Spiked sample concentration  
 SA = Spike added

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

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 816 4446 - LCS

Compound	Spike Added (ug/kg)		Spiked Sample Concentration (ug/kg)		LCS Percent Recovery		LCSD Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
1,1-Dichloroethene	50.10	NA	48.10	NA	96	96				
Trichloroethene			53.0		106	106				
Benzene			51.2		102	102				
Toluene			52.2		104	104				
Chlorobenzene			50.5		101	101	NA			

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





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

**Project/Site Name:** BRC Tronox Parcel F  
**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

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.
- 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/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 ( $\geq 0.05$ ) 0.04408 ( $\geq 0.05$ )	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 ( $\geq 0.05$ )	All samples in SDG F8F110177	J (all detects)	A
	n-(Hydroxymethyl)phthalimide	0.04331 ( $\geq 0.05$ )		UJ (all non-detects)	
				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)

**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 #: 19091A2  
 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/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
I.	Technical holding times	A	Sampling dates: 6/10/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	SW	% RSD, $r^2 \geq 0.990$
IV.	Continuing calibration/ICV	SW	ICV $\leq 25$
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A	TSB - GJ-08-10
VIII.	Laboratory control samples	SW	LCS
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	A	
XI.	Target compound identification	A	Not reviewed for Level III validation.
XII.	Compound quantitation/CRQLs	A	Not reviewed for Level III validation.
XIII.	Tentatively identified compounds (TICs)	A	Not reviewed for Level III validation.
XIV.	System performance	A	Not reviewed for Level III validation.
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	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

TSB	Sample ID	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	
1	TSB-FR-02-02-20'						8168439																									
2	TSB-FR-02-02-30**																															
3	TSB-FJ-02-02-10**																															
4	TSB-FJ-02-02-20**																															
5	TSB-FJ-02-02-30'																															
6	F8F160000-439						8168439																									
7																																
8																																
9																																
10																																



LDC #: 19091A2  
 SDG #: no cover

VALIDATION FINDINGS CHECKLIST

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

Method: Semivolatiles (EPA SW 846 Method 8270C)

Validation Area	Yes	No	NA	Findings/Comments
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
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?	/			
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 > 0.990?	/			
Were all percent relative standard deviations (%RSD) ≤ 30% and relative response factors (RRF) > 0.05?	/		/	
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) ≤ 25% and relative response factors (RRF) ≥ 0.05?	/		/	
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.			/	
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?			/	
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?	/			
Was an LCS analyzed for this SDG?				

LDC #: 19091A2  
 SDG #: su cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: B  
 2nd Reviewer: g

Validation Area	Yes	No	NA	Findings/Comments
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were internal standard area counts within -50% or +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds from the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the major ions (> 10 percent relative intensity) in the reference spectrum evaluated in sample spectrum?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were relative intensities of the major ions within $\pm$ 20% between the sample and the reference spectra?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the raw data indicate that the laboratory performed a library search for all required peaks in the chromatograms (samples and blanks)?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
System performance was found to be acceptable.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Field duplicate pairs were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field duplicates.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target compounds were detected in the field blanks.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

# VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	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	II. 4-Nitrophenol*	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-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT.
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	VVV.
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.







VALIDATION FINDINGS WORKSHEET  
 Initial Calibration Calculation Verification

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:

$RRF = (A_x)(C_s) / (A_s)(C_x)$   
 average RRF = sum of the RRFs / number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_x$  = Area of compound,  
 $C_x$  = Concentration of compound,  
 $S$  = Standard deviation of the RRFs,  
 $A_s$  = Area of associated internal standard  
 $C_s$  = Concentration of internal standard  
 $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				RRF (SD std)	RRF (SD std)	RRF (SD std)	RRF (SD std)	Average RRF (Initial)	Average RRF (Initial)	%RSD	%RSD
1	ICAL-J	6/2/08	Phenol (1st internal standard)	1.87853	1.87853	1.85537	1.85537	1.070	1.070	1.070	1.070
			Naphthalene (2nd internal standard)	1.09438	1.09438	1.10901	1.10901	1.328	1.328	1.328	1.328
			Fluorene (3rd internal standard)	1.41778	1.41778	1.41229	1.41229	0.573	0.573	0.573	0.573
			Pentachlorophenol (4th internal standard)	0.20260	0.20260	0.19634	0.19634	10.255	10.255	10.255	10.255
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.90763	0.90763	0.86343	0.86343	9.524	9.524	9.524	9.524
			Benzo(a)pyrene (6th internal standard)	1.13808	1.13808	1.11182	1.11182	6.486	6.486	6.486	6.486
2	ICAL BRX J	6/18	Acetophenone (1st internal standard)	0.51976	0.51976	0.51274	0.51274	0.7151	0.7151	0.7151	0.7151
			Naphthalene (2nd internal standard)								
			<del>Fluorene (3rd internal standard)</del> phthalimide								
			Pentachlorophenol (4th internal standard)								
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.04162	0.04162	0.04408	0.04408	8.41339	8.41339	8.41339	8.41339
			Benzo(a)pyrene (6th internal standard)								
3			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 qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 19091A2  
 SDG #: per cover

VALIDATION FINDINGS WORKSHEET  
 Continuing Calibration Results Verification

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

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 =  $100 \cdot (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$   
 $\text{RRF} = (A_s) / (C_s) \cdot (A_m) / (C_m)$

Where: ave RRF = initial calibration average RRF  
 RRF = continuing calibration RRF  
 $A_s$  = Area of compound,  
 $C_s$  = Concentration of compound,  
 $A_m$  = Area of associated internal standard  
 $C_m$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	JCAL5195	6/18/08	Phenol (1st internal standard)	1.85537	1.87174	0.88210	0.88210	0.88210
			Naphthalene (2nd internal standard)	1.10901	1.10135	0.69070	0.69070	0.69070
			Fluorene (3rd internal standard)	1.41229	1.39801	1.01058	1.01058	1.01058
			Pentachlorophenol (4th internal standard)	0.19634	0.20370	3.74980	3.74980	3.74980
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.86343	0.87088	0.86222	0.86222	0.86222
			Benzo(a)pyrene (6th internal standard)	1.1182	1.1507	0.27280	0.27280	0.27280
2	JCAL5196	6/18/08	Acetophenone	0.51274	0.52185	1.77632	1.77632	1.77632
			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)	0.04408	0.04331	1.73819	1.73819	1.73819
			Pentachlorophenol (4th internal standard)					
3	JCAL5197	6/18/08	Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th 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 #: 19091A2

SDG #: *see cover*

### VALIDATION FINDINGS WORKSHEET

### Surrogate Results Verification

Page: 1 of 1

Reviewer: *B*

2nd reviewer: *f*

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: 2

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5	50	35.2132	70	70	0
2-Fluorobiphenyl	↓	37.0385	74	74	↓
Terphenyl-d14	↓	36.1641	72	72	
Phenol-d5	75	52.8544	70	70	↓
2-Fluorophenol	↓	52.0442	69	69	
2,4,6-Tribromophenol	↓	55.2829	74	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					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

LDC #: 19091A2  
 SDG #: per cover

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

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

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

Where: SSC = Spiked sample concentration  
 SA = Spike added

SC = Sample concentration

RPD =  $|MS - MSD| * 2 / (MS + MSD)$

MS = Matrix spike percent recovery

MSD = Matrix spike duplicate percent recovery

MS/MSD samples: TSB-GJ-08-10

Compound	Spike Added (ug/kg)		Sample Concentration (ug/kg)	Spiked Sample Concentration (ug/kg)		Matrix Spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD		MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
Phenol	3570	3570	ND	2490	2450	70	70	69	69	1.7	1.7
N-Nitroso-di-n-propylamine				2730	2670	77	77	75	75	2.2	2.2
4-Chloro-3-methylphenol				2760	2690	77	77	75	75	2.8	2.8
Acenaphthene				2640	2620	74	74	73	73	1.0	1.0
Pentachlorophenol				2300	2230	64	64	62	62	3.0	3.0
Pyrene				2460	2390	69	69	67	67	2.7	2.7

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 #: 19091A2

SDG #: per cover

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Page: 1 of 1

Reviewer: A

2nd Reviewer: R

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

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

LCS/LCSD samples: 810 8439

Compound	Spike Added (ug/kg)		Spike Concentration (ug/kg)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Phenol	3330	NA	2260	NA	71	71				
N-Nitroso-di-n-propylamine			2570		77	77				
4-Chloro-3-methylphenol			2560		77	77				
Acenaphthene			2510		75	75				
Pentachlorophenol			2240		67	67				
Pyrene			2350		70	70	NA			

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.



**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:** 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 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**

<b>SDG</b>	<b>Sample</b>	<b>Compound</b>	<b>Flag</b>	<b>A or P</b>	<b>Reason</b>
F8F110177	TSB-FJ-02-02-20' <sup>**</sup> 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 #: 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	SW	ICV ≤ 15
V.	Blanks	Δ	
VI.	Surrogate spikes	A	
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.	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      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

5014

1	TSB-FR-02-02-20'	11	F8F/60000-164	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 cones

VALIDATION FINDINGS CHECKLIST

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

**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 #: 19091A35  
 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 #: all com

# VALIDATION FINDINGS WORKSHEET

## Initial Calibration Calculation Verification

Page: 1 of 1

Reviewer: JAF

2nd Reviewer: L

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 I ch A	530216040	530216040	510995140	510995140	3.14887	3.14887	3.14887	3.14887
			methoxychlor ↓	161496680	161496680	152272620	152272620	6.2575	6.2575	6.2575	6.2575
2			↓ ch B	245001720	245001720	273533412	273533412	2.96584	2.96584	2.96584	2.96584
				44217640	44217640	42222360	42222360	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.

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

LDC #: 1909/A3a  
 SDG #: per conch

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

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

#	Standard ID	Calibration Date	Compound	Average CF (cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	KCAL064	6/18/08	endosulfan / methoxychlor	0.0252 ↓	3.7 0.8	0.0259 0.0252	3.7 0.8	
2	KCAL080	6/18/08	↓	↓	1.0 2.7	0.0252 0.0257	1.0 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	chA	0.02	0.01839	92	92	0
<del>Tetrachloro-m-xylene</del> 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 #: 19091A3a  
 SDG #: per cont

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Sample/Laboratory Control Sample Duplicate Results Verification**

Page: 6 of 7  
 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 =  $100 * (LCS - LCSD) / ((LCS + LCSD) / 2)$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS/LCSD samples: 8168/64 LCS

Compound	Spike Added <i>(149/48)</i>		Spiked Sample Concentration <i>(149/48)</i>		LCS		LCSD		Percent Recovery		Percent Recovery		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	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 #: per cover

### VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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

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: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**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.
- 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 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  
 SDG #: F8F110177  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 7/18/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**METHOD:** GC Polychlorinated Biphenyls (EPA SW 846 Method 8082)

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	ND	
III.	Initial calibration	Δ	
IV.	Continuing calibration/ICV	A	ICV ≤ 15
V.	Blanks	A	
VI.	Surrogate spikes	A	
VII.	Matrix spike/Matrix spike duplicates	A N	Client specified TSB-GJ-08-10
VIII.	Laboratory control samples	A	LCS
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	Δ	
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

1	TSB-FR-02-02-20'	11	F8F160000-162	21	8168162	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 #: 19091A3b  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ____ %D or %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) < 15% or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Blanks</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>V. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VI. 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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

LDC #: 19091A3b  
 SDG #: PL cover

VALIDATION FINDINGS CHECKLIST

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

Validation Area	Yes	No	NA	Findings/Comments
<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. Retention Time 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 and dry weight factors 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.			/	



LDC #: 19091A3b  
 SDG #: su comb

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 =  $100 \cdot (S/X)$   
 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 (100Data)	CF (100Std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	5/21/08	Areolol 1260 cHA ↓ cHB	27377 38550	27377 38550	27977 39164	27977 39164	12.0 9.582	12.0 9.582		
2											
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 #: 19091A3b  
 SDG #: see cover

VALIDATION FINDINGS WORKSHEET  
Continuing Calibration Results Verification

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

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

#	Standard ID	Calibration Date	Compound	Average CF(cal)/ CCV Conc.	Reported		Recalculated	
					CF/Conc. CCV	%D	CF/Conc. CCV	%D
1	PCA1089 13:04	6/18/08	Arroclor 1260 eHA	1000	952.1902	4.8	952.1902	4.8
2	PCA1100 16:03	6/18/08	Arroclor 1260 eHA	1000	937.3342	6.3	937.3342	6.3
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.

DC #: 1904/1 A 30  
SDG #: per com

VALIDATION FINDINGS WORKSHEET I  
Surrogate Results Verification

Page: 01  
Reviewer: [Signature]  
2nd reviewer: [Signature]

METHOD: GC HPLC

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

Where: SF = Surrogate Found  
SS = Surrogate Spiked

% Recovery: SF/SS \* 100

Sample ID: # |

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
DCB	c.w.A	20	16.25SD	81	81	0

Sample ID:

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

Sample ID:

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

LUU #: 17041 ASD  
SDG #: fu cover

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

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

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:

$\%R_{\text{recovery}} = 100 \cdot (SSC - SC) / SA$  Where SSC = Spiked sample concentration, SA = Spike added, MS = Matrix spike

$RPD = \frac{((SSCMS - SSCMSD) \cdot 2) / ((SSCMS + SSCMSD)) \cdot 100}{MS}$  Where SSC = Sample concentration, MSD = Matrix spike duplicate

MS/MSD samples: TSB-GJ-08-10

Compound	Spike Added		Sample Conc.		Spike Sample Concentration		Matrix spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	177	178	ND	ND	181	194	102	102	109	109	7.4	7.4
Diesel (8015)												
Benzene (8021B)												
Methane (RSK-175)												
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)												
Anthracene (8310)												
HMX (8330)												
2,4,6-Trinitrotoluene (8330)												
Aroclor 1260	177	178	ND	ND	181	194	102	102	109	109	7.4	7.4

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 #: 19091A3b

VALIDATION FINDINGS WORKSHEET

Page: 1 of 1

SDG #: fu cover

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer: [Signature]

METHOD: GC HPLC

2nd Reviewer: [Signature]

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

RPD =  $((SSCLCS - SSCLCSD) * 2) / ((SSCLCS + SSCLCSD)) * 100$  LCS = Laboratory Control Sample percent recovery LCS/D = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: LCS

Compound	Spike Added (ug/kg)		Sample Conc. (ug/kg)	Spike Sample Concentration (ug/kg)		LCS Percent Recovery		LCS/D Percent Recovery		RPD	
	LCS	LCS/D		LCS	LCS/D	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)											
Aroclor 1260	167	NA		171	NA	103	103	NA	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 #: 19091A3b

SDG #: *for copy*

# VALIDATION FINDINGS WORKSHEET

## Sample Calculation Verification

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

METHOD: GC HPLC

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 within 10% of the reported results?

Concentration =  $\frac{A(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$

- 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 = Initial weight of the sample
- %S = Percent Solid

Example: \_\_\_\_\_  
Sample ID: \_\_\_\_\_ Compound Name: MP

Concentration = \_\_\_\_\_

#	Sample ID	Compound	Reported Concentrations	Recalculated Results Concentrations	Qualifications

Comments: \_\_\_\_\_

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

**Project/Site Name:** BRC Tronox Parcel F

**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.
- 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. 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)	Iron	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	50.0 (75-125)	50.0 (75-125)	-	J- (all detects) UJ (all non-detects)	A
	Barium	61.1 (75-125)	61.0 (75-125)	-		
	Copper	73.2 (75-125)	-	-		
	Magnesium	43.4 (75-125)	34.8 (75-125)	-		
	Niobium	38.8 (75-125)	39.3 (75-125)	-		
	Phosphorus	43.6 (75-125)	63.8 (75-125)	-		
	Tungsten	71.5 (75-125)	71.0 (75-125)	-		
Zinc	-	74.8 (75-125)	-			

## 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)	A

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	13.8 ( $\leq 10$ )	All samples in SDG F8F110177	J (all detects)	A
	Phosphorus	15.6 ( $\leq 10$ )		J (all detects)	
	Titanium	19.2 ( $\leq 10$ )		J (all detects)	

## **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  
 SDG #: F8F110177  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 7/21-8  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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
I.	Technical holding times	A	Sampling dates: 6/10/08
II.	Calibration	A	
III.	Blanks	SW	
IV.	ICP Interference Check Sample (ICS) Analysis	A	
V.	Matrix Spike Analysis	SW	3 MS/MSD TSB-FJ-06-2-101
VI.	Duplicate Sample Analysis	N	
VII.	Laboratory Control Samples (LCS)	A	LCs
VIII.	Internal Standard (ICP-MS)	SW	Not reviewed for Level III
IX.	Furnace Atomic Absorption QC	N	Not utilized
X.	ICP Serial Dilution	SW	
XI.	Sample Result Verification	A	Not reviewed for Level III validation.
XII.	Overall Assessment of Data	A	
XIII.	Field Duplicates	N	
XIV.	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: 5 or 1      \*\* 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: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

LDC #: 19091A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

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

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

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. Calibration</b>				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury and 85-115% for cyanide) QC limits?	/			
Were all initial calibration correlation coefficients > 0.995? (Level IV only)	/			
<b>III. Blanks</b>				
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.	/			
<b>IV. ICP Interference Check Sample</b>				
Were ICP interference check samples performed daily?	/			
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?	/			
<b>IV. Matrix spike/Matrix spike duplicates</b>				
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) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of +/- RL (+/-2X RL for soil) was used for samples that were ≤ 5X the RL, including when only one of the duplicate sample values were < 5X the RL.	/			
<b>V. 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 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			
<b>VI. Furnace Atomic Absorption QC</b>				
If MSA was performed, was the correlation coefficients > 0.995?			/	
Do all applicable analyses have duplicate injections? (Level IV only)			/	
For sample concentrations > RL, are applicable duplicate injection RSD values < 20%? (Level IV only)			/	
Were analytical spike recoveries within the 85-115% QC limits?			/	

LDC #: 19091A4  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: MM  
 2nd Reviewer: J

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. ICP Serial Dilution</b>				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the IDL?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<u>&gt; 100X MWL for 20 P/mf</u>
Were all percent differences (%Ds) < 10%?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>VIII. Internal Standards (EPA SW 846 Method 6020)</b>				
Were all the percent recoveries (%R) within the 30-120% of the intensity of the internal standard in the associated initial calibration?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
If the %Rs were outside the criteria, was a reanalysis performed?	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>IX. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>X. Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XI. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>XII. Field duplicates</b>				
Field duplicate pairs were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field duplicates.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>XIII. Field blanks</b>				
Field blanks were identified in this SDG.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Target analytes were detected in the field blanks.	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	



LDC #: 1909/AY  
 SDG #: see above

**VALIDATION FINDINGS WORKSHEET**  
**Sample Specific Element Reference**

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

All circled elements are applicable to each sample.

Sample ID	Matrix	Target Analyte List (TAL)
<u>1-5</u>	<u>Soil</u>	<u>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,</u>
		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,
<u>1-5</u>	<u>Soil</u>	<u>Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Li, S, Zr,</u>
		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,
<b>Analysis Method</b>		
ICP		<u>Li, S,</u>
ICP-MS		<u>Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn, Mo, B, Si,</u>
ICP-MS		<u>Nb, Pd, P, Pt, Sn, Sr, Ti, W, U, Zr,</u>
GFAA		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, CN:

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







VALIDATION FINDINGS WORKSHEET  
 ICP Serial Dilution

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

LEVEL IV ONLY:

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

#	Date	Diluted Sample ID	Matrix	Analyte	%D (Limits)	Associated Samples	Qualifications
1		T5B-FJ-06-02-10	soil	Ca	13.8	A1	JLT/A
				P	15.6	J	J
				Ti	19.2		

Comments: Ni, V < 100 XMDL

LDC #: 1909144  
 SDG #: See cover

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

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

**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:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

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

Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	Recalculated		Reported		Acceptable (Y/N)
					%R	%R	%R	%R	
ICV	ICP (Initial calibration)	Li	4037	4000	100.9	100.9	100.9	100.9	Y
	GFAA (Initial calibration)								
ICV	CVAA (Initial calibration)	Hg	2.5	2.5	100.4	100.4	100.4	100.4	Y
ICV	ICP (Continuing calibration)	S	52680	50000	105.4	105.4	105.4	105.4	Y
	GFAA (Continuing calibration)								
ICV	CVAA (Continuing calibration)	Hg	4.9	5.0	98.0	98.0	98.0	98.0	Y
ICV	ICPMS (Initial calibration)	Pb	1019.4	1000	101.9	101.9	101.9	101.9	Y
ICV	ICPMS (Continuing calibration)	P	3881.6	4000	97.0	97.0	97.0	97.0	Y

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.

LDC #: 19091A4  
 SDG #: See cover

**VALIDATION FINDINGS WORKSHEET**  
**Level IV Recalculation Worksheet**

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

**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:

$$\%R = \frac{\text{Found} \times 100}{\text{True}}$$

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.

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

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

Where, S = Original sample concentration  
 D = Duplicate sample concentration

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

$$\%D = \frac{|I-SDR|}{I} \times 100$$

Where, I = Initial Sample Result (mg/L)  
 SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	Recalculated		Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D	
ICSA11	ICP interference check	Zn	104.2	102	104	104	Y
LCS	Laboratory control sample	Cu	149	142	104.9	104.8	Y
TSB-FJ06-0210	Matrix spike	Ba	(SSR-SR) 2.3723	2.6551	89.3	89.4	Y
	Duplicate	Al	9153	910	6.8	6.8	Y
	ICP serial dilution	Ba	555.65 <del>548.87</del>	561.42	100	100	Y

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.

LDC #: 1909/AY  
 SDG #: See work

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

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

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 2 were recalculated and verified using the following equation:

$$\text{Concentration} = \frac{(\text{RD})(\text{FV})(\text{Dil})}{(\text{In. Vol.})(\%S)}$$

Recalculation:

- RD = Raw data concentration
- FV = Final volume (ml)
- In. Vol. = Initial volume (ml) or weight (G)
- Dil = Dilution factor
- %S = Decimal percent solids

$$S = \frac{9.314 \text{ mg/L} \times 0.1 \text{ L} \times 1000 \text{ g/mg}}{0.5 \text{ g} \times 0.6931} = 2688 \text{ mg/kg}$$

Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
2	Li	133	133	Y
	S	2690	2690	Y
	Al	18200	18200	Y
	As	35.5	35.5	Y
	Ba	56.2	56.2	Y
	Be	0.97	0.97	Y
	B	27.8	27.8	Y
	Ca	23400	23400	Y
	Cr	26.4	26.4	Y
	Co	8.8	8.8	Y
	Cu	28.8	28.8	Y
	Fe	19900	19900	Y
	Pb	10.6	10.6	Y
	Mg	45100	45100	Y
	Mn	310	310	Y
	Mo	2.9	2.8	Y
	Ni	20.7	20.7	Y
	Pd	0.48	0.48	Y
	P	812	812	Y
	K	3780	3780	Y
	Si	1200	1200	Y
	Ag	0.19	0.19	Y



LDC #: 19091A4  
SDG #: See work

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

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

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 2 were recalculated and verified using the following equation:

$$\text{Concentration} = \frac{(\text{RD})(\text{FV})(\text{Dil})}{(\text{In. Vol.})(\%S)}$$

Recalculation:

- RD = Raw data concentration
- FV = Final volume (ml)
- In. Vol. = Initial volume (ml) or weight (G)
- Dil = Dilution factor
- %S = Decimal percent solids

$$\text{Na} = \frac{668.431 \text{ ug/L} \times 0.1 \text{ L} \times 5}{0.58 \text{ L} \times 0.6931} = 964.4 \text{ ug/L}$$

Sample ID	Analyte	Reported Concentration (ug/L)	Calculated Concentration (ug/L)	Acceptable (Y/N)
<u>2</u>	<u>Na</u>	<u>964</u>	<u>964</u>	<u>Y</u>
	<u>Sr</u>	<u>219</u>	<u>219</u>	<u>Y</u>
	<u>Ti</u>	<u>866</u>	<u>866</u>	<u>Y</u>
	<u>Y</u>	<u>6.7</u>	<u>6.7</u>	<u>Y</u>
	<u>V</u>	<u>60.9</u>	<u>60.9</u>	<u>Y</u>
	<u>Zn</u>	<u>650</u>	<u>650</u>	<u>Y</u>
	<u>Zr</u>	<u>44.4</u>	<u>44.4</u>	<u>Y</u>

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

**Project/Site Name:** BRC Tronox Parcel F  
**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, Chlorine, 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.
- 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. 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
MB	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  
 SDG #: F8F110177  
 Laboratory: Test America

**VALIDATION COMPLETENESS WORKSHEET**

Level III/IV

Date: 7/27/08

Page: 1 of 1

Reviewer: MW

2nd Reviewer: J

**METHOD: (Analyte)** Bromide, Bromine, Chlorate, Chloride, Chlorine, 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
I.	Technical holding times	A	Sampling dates: 6/10/08
IIa.	Initial calibration	A	
IIb.	Calibration verification	A	
III.	Blanks	SW	
IV	Matrix Spike/Matrix Spike Duplicates	A	3 MS/Rep TSB-FJ-06-02-10'
V	Duplicates	A	
VI.	Laboratory control samples	A	LCs
VII.	Sample result verification	A	Not reviewed for Level III validation.
VIII.	Overall assessment of data	A	
IX.	Field duplicates	N	
X	Field blanks	N	

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

Validated Samples: soil \*\* 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	MB	16		26		36
7		17		27		37
8		18		28		38
9		19		29		39
10		20		30		40

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



LDC #: 19091 Ab  
 SDG #: See cover

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2  
 Reviewer: mm  
 2nd Reviewer: g

Method: Inorganics (EPA Method See cover)

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. Calibration</b>				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial calibration correlation coefficients > 0.995?	/			
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)	/			
<b>III. Blanks</b>				
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.	/			
<b>IV. Matrix spike/Matrix spike duplicates and Duplicates</b>				
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) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of ≤ CRDL (≤ 2X CRDL for soil) was used for samples that were ≤ 5X the CRDL, including when only one of the duplicate sample values were ≤ 5X the CRDL.	/			
<b>V. 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 80-120% (85-115% for Method 300.0) QC limits?	/			
<b>VI. Regional Quality Assurance and Quality Control</b>				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	

LDC #: 19091 Ab  
 SDG #: see cov

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: MM  
 2nd Reviewer: A

Validation Area	Yes	No	NA	Findings/Comments
<b>VII. Sample Result Verification</b>				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
Were detection limits < RL?	/			
<b>VIII. Overall assessment of data</b>				
Overall assessment of data was found to be acceptable.	/			
Field duplicate pairs were identified in this SDG.		✓		
Target analytes were detected in the field duplicates.			✓	
Field blanks were identified in this SDG.		✓		
Target analytes were detected in the field blanks.			/	





LDC #: 1991A6  
 SDG #: See cover

**Validatin Findings Worksheet**  
**Initial and Continuing Calibration Calculation Verification**

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

**Method:** Inorganics, Method See cover  
 The correlation coefficient (r) for the calibration of cl was recalculated. Calibration date: 6/18/08

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

%R =  $\frac{\text{Found} \times 100}{\text{True}}$  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	Standard	Conc. (ug/L)	Area	Recalculated		Reported		Acceptable (Y/N)
					r or r <sup>2</sup>	r or r <sup>2</sup>			
Initial calibration	Cl	s1	200	0.04	0.99984	0.99991			Y
		s2	500	0.091					
		s3	1000	0.191					
		s4	2500	0.474					
		s5	5000	0.989					
ccv Calibration verification	abstract	4000	4081		102	NK		Y	
ccv Calibration verification	F	1000	1035.4		103.5	103.5		Y	
ccv Calibration verification	Bx	2000	2016.6		100.8	100.8		Y	

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.

LDC #: 1929 | A6  
 SDG #: See cover

VALIDATION FINDINGS WORKSHEET  
 Level IV Recalculation Worksheet

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

METHOD: Inorganics, Method See cover

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

$\%R = \frac{\text{Found}}{\text{True}} \times 100$  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.

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

$RPD = \frac{S-D}{(S+D)/2} \times 100$  Where, S = Original sample concentration  
 D = Duplicate sample concentration

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated		Reported	Acceptable (Y/N)
					%R / RPD	%R / RPD		
L07	Laboratory control sample	019	1173	1330	88	88	88	Y
TSB-EJ -06-02-1-	Matrix spike sample	element	43.6 (SSR-SR)	42.5	103	103	103	Y
↓	Duplicate sample	SO4	242	245	1.2	1.2	1.2	Y

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.

LDC #: 19091A6  
 SDG #: see cover

**VALIDATION FINDINGS WORKSHEET**  
 Sample Calculation Verification

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

METHOD: Inorganics, Method see cover

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?
- Y N N/A Are results within the calibrated range of the instruments?
- Y N N/A Are all detection limits below the CRQL?

Compound (analyte) results for 2 reported with a positive detect were recalculated and verified using the following equation:

Concentration = 
$$C = \frac{0.215 \times \frac{0.04l}{\cancel{1ml}} \times 10}{0.69 \times \mu g \times 0.000196} = 158.97 \text{ mg/kg}$$

#	Sample ID	Analyte	Reported Concentration (mg/kg)	Calculated Concentration (mg/kg)	Acceptable (Y/N)
1	2	chloride	4.8	4.8	Y
		Cl	159	159	↓
		Cl <sub>2</sub>	317	318	
		F	3.5	3.5	
		NO <sub>3</sub> -N	3.4	3.4	
		SO <sub>4</sub>	524	525	

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:** 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

LDC #: 19091A7  
 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 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
I.	Technical holding times	A	Sampling dates: <u>6/10/08</u>
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	A	<u>ICV ≤ 15</u>
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	<u>TSB-FJ-06-02-10</u>
IVc.	Laboratory control samples	<u>SW</u>	<u>LCS 10</u>
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	
X.	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 SOIL

<u>1</u>	TSB-FR-02-02-20'	11	<u>F8F130000-267</u>	21	<u>8165267</u>	31	
<u>2</u>	TSB-FR-02-02-30'	12		22		32	
<u>3</u>	TSB-FJ-02-02-10'	13		23		33	
<u>4</u>	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	

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

LDC #: 19091 A 7  
 SDG #: you cover

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC

Validation Area	Yes	No	NA	Findings/Comments
<b>I. Technical holding times</b>				
All technical holding times were met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>II. Initial calibration</b>				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a linear fit used for evaluation? If yes, were all percent relative standard deviations (%RSD) < 20%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a curve fit used for evaluation? If Yes, what was the acceptance criteria used?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did the initial calibration meet the curve fit acceptance criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the RT windows properly established?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>III. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ____ %D or %R	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a continuing calibration analyzed daily?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all percent differences (%D) < 15% or percent recoveries 85-115%?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were all the retention times within the acceptance windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>IV. Blank samples</b>				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a method blank analyzed for each matrix and concentration?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
<b>V. Surrogate spikes</b>				
Were all surrogate %R within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
If the percent recovery (%R) of one or more surrogates was outside QC limits, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
If any %R was less than 10 percent, was a reanalysis performed to confirm %R?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
<b>VI. 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.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

LDC #: 1909147  
 SDG #: fu cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: PI  
 2nd Reviewer: J

Validation Area	Yes	No	NA	Findings/Comments
<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. Retention Time 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 and dry weight factors 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.			/	





LDC #: 19091A7  
 SDG #: for cover

**VALIDATION FINDINGS WORKSHEET**  
Initial Calibration Calculation Verification

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

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 (1-σ std)	CF (1-σ std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	1CAL	5/27/08	GRO	17025649	17025649	17182732	17182732	3.915	3.915	3.915	3.915
2											
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.



VALIDATION FINDINGS WORKSHEET I  
 Surrogate Results Verification

DC #: 19091A7  
 SDG #: full concn  
 METHOD: GC HPLC

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

Where: SF = Surrogate Found  
 SS = Surrogate Spiked

% Recovery: SF/SS \* 100

Sample ID: #2

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
TFT	not specified	0.04	83 0.03339	83	83	0

Sample ID:

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

Sample ID:

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



METHOD: GC HPLC

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 =  $100 \cdot (SSC \cdot SC) / SA$  Where SSC = Spiked concentration SC = Sample concentration SA = Spike added

RPD =  $\frac{((SSCLCS - SSC) / SSC) + ((SSCLCS - SSC) / SSC)}{2} \cdot 100$  LCS = Laboratory Control Sample percent recovery LCS/D = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8/65 267 - LC >

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)	Spike Sample Concentration (mg/kg)		LCS Percent Recovery		LCS/D Percent Recovery		LCS/D RPD	
	LCS	LCSD		LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)	1.0	1.0	---	1.20	0.986	120	120	99	97	19	19
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 Sample Duplicate findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.



**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:** Diesel 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 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**

### **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 #: 19091A8  
 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 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	$\Delta$	Sampling dates: <u>6/10/08</u>
IIa.	Initial calibration	$\Delta$	
IIb.	Calibration verification/ICV	A	<u>ICV = 15</u>
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	<u>TSB-FJ-02-02-30' + TSB-CJ-09-0'</u>
IVc.	Laboratory control samples	A	<u>LCS</u>
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	
X.	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	<u>F8F130000-291</u>	21	<u>8165291</u>	31	
21	TSB-FR-02-02-30'***	12	<u>F8F180000-312</u>	22	<u>8170312</u>	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: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

LDC #: 1909/AB  
 SDG #: pu cover

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC

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. 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) < 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?	/			
<b>III. Continuing calibration</b>				
What type of continuing calibration calculation was performed? ___%D or %R	/			
Was a continuing calibration analyzed daily?	/			
Were all percent differences (%D) < 15% or percent recoveries 85-115%?	/			
Were all the retention times within the acceptance windows?	/			
<b>IV. Blanks</b>				
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.		/		
<b>V. 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?	/		/	
<b>VI. 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>VII. 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?	/			

LDC #: 19091A8  
 SDG #: 44 cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: PT  
 2nd Reviewer: g

Validation Area	Yes	No	NA	Findings/Comments
<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. Retention Time 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 and dry weight factors 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.			/	

LDC #: 19021 AX  
 SDG #: for count

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

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

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 =  $100 \cdot (S/X)$
- 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 (100/Std)	CF (100/Std)	Average CF (Initial)	Average CF (Initial)	%RSD	%RSD		
1	ICAL	5/16/08	PRO	16236	16236	16023	16023	3.456	3.456	3.456	3.456
2											
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 #: 19021A8  
 SDG #: fu cover

**VALIDATION FINDINGS WORKSHEET**  
Continuing Calibration Results Verification

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

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 =  $100 \cdot (\text{ave. CF} - \text{CF}) / \text{ave. CF}$  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	ECAL525	6/17/08	Diesel	1000.00	996.5312	0.3	996.53	0.3
2	ECAL549	6/18/08	↓	↓	1039.4417	3.9	1039.4417	3.9
3	ECAL575	6/19/08	↓	1000	979.8723	2.0	979.8723	2.0
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.



**VALIDATION FINDINGS WORKSHEET I**  
**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: # 2

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
O-Terphenyl	not specified	25	20.989	84	84	0

Sample ID:

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

Sample ID:

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

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

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      SSC = Spiked sample concentration  
 SA = Spike added  
 MS = Matrix spike  
 RPD =  $(((SSCMS - SSCMSD) * 2) / ((SSCMS + SSCMSD)) * 100)$       SC = Sample concentration  
 MSD = Matrix spike duplicate

MS/MSD samples: TSB - FJ - 02 - 02 - 30'

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)		Spike Sample Concentration (mg/kg)		Matrix spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)	87.2	88.6	74.5	74.5	74.5	74.5	85	85	89	89	5.2	5.2
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 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.

**VALIDATION FINDINGS WORKSHEET**  
Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC HPLC

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 =  $100 \cdot (SSC - SC) / SA$  Where SSC = Spiked concentration SA = Spike added SC = Sample concentration

RPD =  $\frac{((SSCLCS - SSCLCD) \cdot 2) / ((SSCLCS + SSCLCD)) \cdot 100}{LCS}$  LCS = Laboratory Control Sample percent recovery LCS/D = Laboratory Control Sample duplicate percent recovery

LCS/LCSD samples: 8165291- LCS

Compound	Spike Added (mg/kg)		Sample Conc. (mg/kg)	Spike Sample Concentration (mg/kg)		LCS		LCS/D	
	LCS	LCSD		LCS	LCSD	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)									
Diesel (8015)	83.3	NA	0	68.9	NA	83	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 Sample Duplicate findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.



**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

**VALIDATION COMPLETENESS WORKSHEET**  
 Level III/IV

Date: 7/17/08  
 Page: 1 of 1  
 Reviewer: [Signature]  
 2nd Reviewer: [Signature]

**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
I.	Technical holding times	A	Sampling dates: <u>6/10/08</u>
IIa.	Initial calibration	A	
IIb.	Calibration verification/ICV	SW	<u>14 ≤ 15</u>
III.	Blanks	A	
IVa.	Surrogate recovery	A	
IVb.	Matrix spike/Matrix spike duplicates	A	<u>TSB-GJ-08-10</u>
IVc.	Laboratory control samples	A	<u>LCS</u>
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	
X.	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

Sample #	Sample ID	11	21	31
<u>1</u>	<u>TSB-FR-02-02-20'</u>	<u>11</u>	<u>F8 F160000-158</u>	<u>31</u>
<u>2</u>	<u>TSB-FR-02-02-30**</u>	<u>12</u>		<u>32</u>
<u>3</u>	<u>TSB-FJ-02-02-10**</u>	<u>13</u>		<u>33</u>
<u>4</u>	<u>TSB-FJ-02-02-20**</u>	<u>14</u>		<u>34</u>
<u>5</u>	<u>TSB-FJ-02-02-30'</u>	<u>15</u>		<u>35</u>
<u>6</u>		<u>16</u>		<u>36</u>
<u>7</u>		<u>17</u>		<u>37</u>
<u>8</u>		<u>18</u>		<u>38</u>
<u>9</u>		<u>19</u>		<u>39</u>
<u>10</u>		<u>20</u>		<u>40</u>

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

LDC #: 19091A9  
 SDG #: fire cover

VALIDATION FINDINGS CHECKLIST

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

Method: GC HPLC

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. 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) < 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?	/			
<b>III. Continuing calibration</b>				
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?	/			
<b>IV. Blanks</b>				
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.		/		
<b>V. 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?	/		/	
<b>VI. 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>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			

LDC #: 1909/A9  
 SDG #: per cover

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2  
 Reviewer: F7  
 2nd Reviewer: g

Validation Area	Yes	No	NA	Findings/Comments
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
IX: Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
X: Target compound identification				
Were the retention times of reported detects within the RT windows?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
XI: Compound quantitation/CRQLs				
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XII: System performance				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIII: Overall assessment of data				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIV: Field duplicates				
Were field duplicate pairs identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds idetected in the field duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
XV: Field blanks				
Were field blanks identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field blanks?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	

VALIDATION FINDINGS WORKSHEET

METHOD: GC HPLC

8310	8330	8151	8141	8141(cont)	8021B
A. Acenaphthene	A. HMX	A. 2,4-D	A. Dichlorvos	V. Fensulfothion	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-Q	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. Naled	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	
I. Chrysene	I. 2-Amino-4,6-dinitrotoluene	I. MCPP	I. Dimethoate	DD. Trifluralin	
J. Dibenz(a,h)anthracene	J. 2,4-Dinitrotoluene	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-methyl	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	P.		P. Fenthion		
Q.	Q		Q. Parathion-ethyl		
R.			R. Trichloronate		
S.			S. Merphos		
			T. Stirofos		
			U. Tokuthion		

Notes:









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

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
p-Terphenyl	not specified	25	17.9118	72	72	0

Sample ID:

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

Sample ID:

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

LDC #: 19091A9  
 SDG #: see cover

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

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

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:

$$\% \text{Recovery} = 100 * ((\text{SSC} - \text{SC}) / \text{SA})$$

SSC = Spiked sample concentration  
 SA = Spike added

SC = Sample concentration

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

MS = Matrix spike

MSD = Matrix spike duplicate

MS/MSD samples: TSB-9J-08-10

Compound	Spike Added (ug/kg)		Sample Conc. (ug/kg)		Spike Sample Concentration (ug/kg)		Matrix spike Percent Recovery		Matrix Spike Duplicate Percent Recovery		MS/MSD RPD	
	MS	MSD	MS	MSD	MS	MSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)												
Diesel (8015)												
Benzene (8021B)												
Methane (RSK-175)												
2,4-D (8151)												
Dinoseb (8151)												
Naphthalene (8310)	69.8	70.9			51.5		72	72	73	73	2.0	2.0
Anthracene (8310)	69.8	70.9			49.6		76	76	70	70	6.5	6.5
HMX (8330)												
2,4,6-Trinitrotoluene (8330)												

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 #: 19091A9

VALIDATION FINDINGS WORKSHEET

SDG #: Full copy Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

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

METHOD: GC HPLC

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:

$\% \text{Recovery} = 100 * (\text{SSC} - \text{SC}) / \text{SA}$       Where       $\text{SSC} = \text{Spiked sample concentration}$        $\text{SC} = \text{Sample concentration}$   
 $\text{RPD} = \left( \frac{|\text{SSCLCS} - \text{SSCLCSD}| * 2}{\text{SSCLCS} + \text{SSCLCSD}} \right) * 100$        $\text{SA} = \text{Spike added}$   
 $\text{LCS/LCSD samples: } 816815B - LCS$        $\text{LCS} = \text{Laboratory Control Sample}$        $\text{LCSD} = \text{Laboratory Control Sample duplicate}$

Compound	Spike Added		Sample Conc. (ug/kg)	Spike Sample Concentration		LCS		LCSD		LCS/LCSD	
	LCS	LCSD		LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)			---								
Diesel (8015)											
Benzene (8021B)											
Methane (RSK-175)											
2,4-D (8151)											
Dinoseb (8151)											
Naphthalene (8310)	667	na		484	na	73	73				
Anthracene (8310)	66.7	↓		51.2	↓	77	77				
HMX (8330)											
2,4,6-Trinitrotoluene (8330)											

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.



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

**Project/Site Name:** BRC Tronox Parcel F  
**Collection Date:** June 10, 2008  
**LDC Report Date:** July 23, 2008  
**Matrix:** Soil  
**Parameters:** Dioxins/Dibenzofurans  
**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)	P

## 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

LDC #: 19091A21  
 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:** HRGC/HRMS Dioxins/Dibenzofurans (EPA SW 846 Method 8290)

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

	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 6/10/08
II.	GC/MS Instrument performance check	A	
III.	Initial calibration	A	
IV.	Routine calibration/ICV <i>ICV</i>	A	
V.	Blanks	A	
VI.	Matrix spike/Matrix spike duplicates	N	<i>limit specified</i>
VII.	Laboratory control samples	A	<i>LOS</i>
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	<i>SW</i>	
X.	Target compound identifications	A	Not reviewed for Level III validation.
XI.	Compound quantitation and CRQLs	A	Not reviewed for Level III validation.
XII.	System performance	A	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

*Soil*

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	

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

LDC #: 19091A21  
 SDG #: F8F110177

VALIDATION FINDINGS CHECKLIST

Page: 1 of 3  
 Reviewer: R  
 2nd Reviewer: A

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

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/MS Instrument performance check</b>				
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)?	/			
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?	/			
<b>III. Initial calibration</b>				
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 $\geq 10$ ?	/			
<b>IV. Continuing calibration</b>				
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?	/			
<b>V. Blanks</b>				
Was a method blank associated with every sample in this SDG?	/			
Was a method blank performed for each matrix and concentration?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet?		/		
<b>VI. 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.		/		
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			/	
<b>VII. Laboratory control samples</b>				
Was an LCS analyzed for this SDG?	/			

LDC #: 19091A21  
 SDG #: F8F110177

VALIDATION FINDINGS CHECKLIST

Page: 2 of 3  
 Reviewer: LC  
 2nd Reviewer: h

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 PCDF channel?			/	
Was an acceptable lock mass recorded and monitored?			/	
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?			/	
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XII. System performance				
System performance was found to be acceptable.	/			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		

LDC #: 19091A21  
SDG #: F8F110177

VALIDATION FINDINGS CHECKLIST

Page: 3 of 3  
Reviewer: JL  
2nd Reviewer: g

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.		/		
Target compounds were detected in the field blanks.			/	



# VALIDATION FINDINGS WORKSHEET

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

A. 2,3,7,8-TCDD	F. 1,2,3,4,6,7,8-HpCDD	K. 1,2,3,4,7,8-HxCDF	P. 1,2,3,4,7,8,9-HpCDF	U. Total HpCDD
B. 1,2,3,7,8-PeCDD	G. OCDD	L. 1,2,3,6,7,8-HxCDF	Q. OCDF	V. Total TCDF
C. 1,2,3,4,7,8-HxCDD	H. 2,3,7,8-TCDF	M. 2,3,4,6,7,8-HxCDF	R. Total TCDD	W. Total PeCDF
D. 1,2,3,6,7,8-HxCDD	I. 1,2,3,7,8-PeCDF	N. 1,2,3,7,8,9-HxCDF	S. Total PeCDD	X. Total HxCDF
E. 1,2,3,7,8,9-HxCDD	J. 2,3,4,7,8-PeCDF	O. 1,2,3,4,6,7,8-HpCDF	T. Total HxCDD	Y. Total HpCDF

Notes:

---



---



---



**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:

$RRF = (A_x)(C_s)/(A_s)(C_x)$   
 average RRF = sum of the RRFs/number of standards  
 $\%RSD = 100 * (S/X)$   
 $A_x$  = Area of compound,  $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,  $C_s$  = Concentration of internal standard  
 $S$  = Standard deviation of the RRFs,  $X$  = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported		Recalculated		Reported		Recalculated	
				Average RRF (Initial)	Average RRF (Initial)	RRF (CS3 std)	RRF (CS3 std)	%RSD	%RSD		
1	CAL	6/16/09	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.798	0.82	0.82	12.5	12.5	12.7	12.7
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.912	0.93	0.93	10.2	10.2	10.3	10.3
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.821	0.820	0.87	0.87	13.9	13.9	14.1	14.1
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.844	0.844	0.88	0.88	12.8	12.8	12.7	12.7
			OCDF ( <sup>13</sup> C-OCDF)	1.721	1.722	1.86	1.86	16.2	16.2	16.3	16.3
2			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)								
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)								
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)								
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)								
			OCDF ( <sup>13</sup> C-OCDF)								

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.

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

**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 =  $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$       Where:      ave. RRF = initial calibration average RRF  
 RRF =  $(A_x)(C_s) / (A_s)(C_x)$       RRF = continuing calibration RRF  
 $A_x$  = Area of compound,       $A_s$  = Area of associated internal standard  
 $C_x$  = Concentration of compound,       $C_s$  = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Reported		Recalculated	
					RRF (CC)	%D	RRF (CC)	%D
1	STD627A	6/27/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.81	1.5	0.81	1.5
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.78	14.4	0.78	14.4
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.82	0.82	0.2	0.82	0.3
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.844	0.82	2.4	0.82	2.5
			OCDF ( <sup>13</sup> C-OCDD)	1.721	1.53	11.2	1.53	11.2
2	STD630B	6/30/08	2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)	0.798	0.84	4.9	0.84	4.9
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)	0.913	0.80	12.8	0.80	12.8
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)	0.82	0.89	8.4	0.89	8.4
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)	0.844	0.91	7.3	0.91	7.3
			OCDF ( <sup>13</sup> C-OCDD)	1.721	1.68	2.6	1.68	2.7
3			2,3,7,8-TCDF ( <sup>13</sup> C-2,3,7,8-TCDF)					
			2,3,7,8-TCDD ( <sup>13</sup> C-2,3,7,8-TCDD)					
			1,2,3,6,7,8-HxCDD ( <sup>13</sup> C-1,2,3,6,7,8-HxCDD)					
			1,2,3,4,6,7,8-HpCDD ( <sup>13</sup> C-1,2,4,6,7,8-HpCDD)					
			OCDF ( <sup>13</sup> C-OCDD)					

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

**VALIDATION FINDINGS WORKSHEET**  
**Laboratory Control Sample Results Verification**

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

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

% Recovery =  $100 * \frac{SSC}{SA}$  Where: SSC = Spiked sample concentration  
 SA = Spike added

RPD =  $100 * \frac{LCS - LCSD}{LCS + LCSD}$

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: 816935/LCS

Compound	Spike Added ( <u>20/100</u> )		Spiked Sample Concentration ( <u>16.7/106</u> )		LCS		LCSD		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
2,3,7,8-TCDD	<u>20</u>		<u>16.7</u>		<u>83</u>	<u>84</u>				
1,2,3,7,8-PeCDD	<u>100</u>		<u>106</u>		<u>106</u>	<u>106</u>				
1,2,3,4,7,8-HxCDD	<u>200</u>		<u>95.4</u>		<u>95</u>	<u>95</u>				
1,2,3,4,7,8,9-HpCDF			<u>89.2</u>		<u>89</u>	<u>89</u>				
OCDF			<u>183</u>		<u>92</u>	<u>92</u>				

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

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate mass <sup>(a)</sup>	Ion ID	Elemental Composition	Analyte	Descriptor	Accurate Mass <sup>(b)</sup>	Ion ID	Elemental Composition	Analyte		
1	303.9016	M	C <sub>12</sub> H <sub>35</sub> Cl <sub>9</sub> O	TCDF	4	407.7818	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	305.8987	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> C <sub>10</sub>	TCDF		409.7788	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O	HpCDF		
	315.9419	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O	TCDF (S)		417.8250	M	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>4</sub> O	HpCDF (S)		
	317.9389	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	TCDF (S)		419.8220	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDF		
	319.8965	M	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD		423.7767	M+2	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO	HpCDD		
	321.8936	M+2	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> C <sub>10</sub> <sub>2</sub>	TCDD		425.7737	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD		
	331.9368	M	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>4</sub> O <sub>2</sub>	TCDD (S)		435.8169	M+2	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	333.9338	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO <sub>2</sub>	TCDD (S)		437.8140	M+4	<sup>13</sup> C <sub>12</sub> H <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> Cl <sub>2</sub> O <sub>2</sub>	HpCDD (S)		
	375.8364	M+2	C <sub>12</sub> H <sub>4</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDFPE		479.7165	M+4	C <sub>12</sub> H <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> Cl <sub>2</sub> O	NCDPE		
	[354.9792]	LOCK	C <sub>9</sub> F <sub>13</sub>	PFK		[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK		
	2	339.8597	M+2	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO		PeCDF	5	441.7428	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO	OCDF
		341.8567	M+4	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> Cl <sub>2</sub> O		PeCDF		443.7399	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDF
		351.9000	M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> C <sub>10</sub>		PeCDF (S)		457.7377	M+2	C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
		353.8970	M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> C <sub>10</sub>		PeCDF (S)		459.7348	M+4	C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD
355.8546		M+2	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	469.7780	M+2		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>7</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
357.8516		M+4	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD	471.7750	M+4		<sup>13</sup> C <sub>12</sub> <sup>35</sup> Cl <sub>6</sub> <sup>37</sup> ClO <sub>2</sub>	OCDD (S)		
367.8949		M+2	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	513.6775	M+4		C <sub>12</sub> <sup>35</sup> Cl <sub>8</sub> <sup>37</sup> Cl <sub>2</sub> O	DCDPE		
369.8919		M+4	<sup>13</sup> C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>3</sub> <sup>37</sup> ClO <sub>2</sub>	PeCDD (S)	[422.9278]	LOCK		C <sub>10</sub> F <sub>17</sub>	PFK		
409.7974		M+2	C <sub>12</sub> H <sub>35</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> ClO	HpCDDPE							
[354.9792]		LOCK	C <sub>9</sub> F <sub>13</sub>	PFK							
3		373.8208	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF						
		375.8178	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> Cl <sub>2</sub> O	HxCDF						
		383.8639	M	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> O	HxCDF (S)						
		385.8610	M+2	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO	HxCDF (S)						
	389.8156	M+2	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>5</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	391.8127	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD							
	401.8559	M+2	<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	M+4	<sup>13</sup> C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>4</sub> <sup>37</sup> ClO <sub>2</sub>	HxCDD (S)							
	445.7555	M+4	C <sub>12</sub> H <sub>2</sub> <sup>35</sup> Cl <sub>9</sub> <sup>37</sup> Cl <sub>2</sub> O	OCDFPE							
	[430.9728]	LOCK	C <sub>9</sub> F <sub>17</sub>	PFK							

(a) The following nuclidic masses were used:

- H = 1.007825
- C = 12.000000
- <sup>13</sup>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

